NEW METHODS, NEW POSSIBILITIES

AN EVALUATION OF ORGANIC RESIDUE ANALYSIS EXTRACTION METHODS FOR THE ARCHAEOLOGY OF THE IBERIAN PENINSULA

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RESUMEN Hasta hoy, el campo de los análisis orgánicos de residuos lipídicos ha ayudado a entender la función de cerámica así como detectar productos que de otra forma serían invisibles. En la Península Ibérica, el método ha sido aplicado de forma escasa pero continua. Después de más de 20 años de análisis, una de las principales limitaciones para el completo desarrollo de esta técnica es el bajo nivel de preservación que presentan los residuos en el sur de Europa. Sin embargo, nuevas técnicas de extracción están consiguiendo mejores resultados con menor cantidad de muestra. En consecuencia, existe la necesidad de evaluar como distintas técnicas de extracción de lípidos podrían afectar los resultados. Pero aún más importante, cuales serian las implicaciones y las nuevas preguntas arqueológicas que podrían ser respuestas con una nueva ola de análisis más agresivos y menos invasivos? Las diferentes alterativas que hoy existen para la preparación de muestras serán evaluadas en términos de sus puntos fuertes y flacos para la arqueología de la Península Ibérica.

PALABRAS CLAVE Análisis orgánicos de residuos, Prehistoria, extracción de metanol Acidificado, δ13C, tasas de preservación

ABSTRACT The field of lipid organic residue analysis on pottery has so far helped understand pottery functionality as well as to detect products otherwise invisible. In the Iberian Peninsula, the method has been scarcely but continually applied. After more than 20 years of analyses, one of the main issues for the full development of this technique has been the poor level of preservation that residues present in southern Europe. Nevertheless, new extraction methodologies are nowadays achieving better yields with less sample quantity. Therefore, there is a need to evaluate how different extraction methods can affect results, but, even more important, what would be the implications and the new archaeological questions that could be answered with a new wave of less invasive yet more aggressive extraction methods? The different alternatives which nowadays exist in sample preparation will be evaluated regarding its strengths and weaknesses in the archaeology of the Iberian Peninsula.

KEYWORDS Organic residue analysis, Prehistory, acidified methanol extraction, δ13C, preservation rates

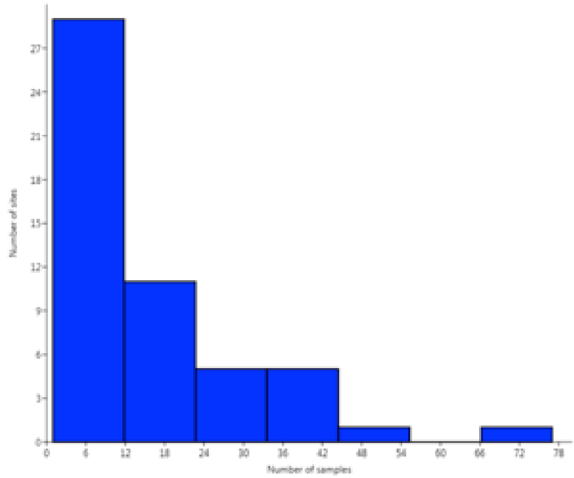
ORGANIC RESIDUE ANALYSIS IN THE IBERIAN PENINSULA

It is not the aim of this article to present an extended literature review on the state of organic residue analysis in the Iberian Peninsula. The recent works by Guerra (2006) and Molina (2015) present a wide coverage of the studies published both internationally and locally. Nowadays, the archaeological value of lipid organic residue analysis is the acquisition of extra data on the possible contents the vessels had. Sometimes interpretations utilize the data to assign different uses to the spaces where the pottery was found (Sánchez *et al.*, 1998). Residue characterisation has been also used to discuss the symbolic and ritual roles bell beaker pottery could have had (Guerra, 2006). Nevertheless, the general lack of a clear archaeological question solvable through these analyses has relegated reports on pottery residues to annexes (Clausell *et al.*, 2000), footnotes (Castro *et al.*, 2012) and purely descriptive reports (Tresserras, 2009), a place where they hardly help make an impact into the current knowledge and understanding of prehistoric societies.

Far from this last idea, organic residue analysis offers the chance to answer for the active role of pottery in society. Usually research has tried to find a correlation between vessel shape and content (Sánchez *et al.*, 2011, p. 224), while another option is the detection of certain products and thus prove the exploitation of specific natural resources and its production processes. As an example, previous research has answered whether deep dietetic changes in the Mesolithic-Neolithic transition in northern Europe existed (Cramp *et al.*, 2014), whether consumption of dairy products was widespread despite of lactose intolerance (Evershed *et al.*, 2008) or whether a close relationship existed between

fish products and pottery in hunter-gatherer societies (Craig *et al.*, 2013). Nonetheless, although answers to these questions are available for many other European regions, organic residue analysis in the Iberian Peninsula has not been able to provide enough quality data to clearly assess these questions.
Therefore, there is a need to understand the way organic residue analysis is performed in the Iberian Peninsula in order to find the best strategies capable of yielding the best quality data with the most possibilities for archaeological interpretation. A wide (but not complete) literature review of 38 reports (all of them cited in this article) of Organic Residue Analysis performed in the Iberian Peninsula revealed that, up to date, more than 700 vessels dating between the first Neolithic and the Roman conquest have been analysed from 52 sites (figure 1). Nevertheless, when critically assessing these reports three main tendencies seem to appear:

1. Approximate location of the archaeological sites reported in the literature whith pothserds analysed by GC-MS in order to detect embedded lipid residues. 1 Devesa do Rei (Prieto *et al.*, 2009), 2 Forno dos Mouros (Prieto *et al.*, 2005), 3 Mirás (Prieto *et al.*, 2005), 4 Agronovo (Prieto *et al.*, 2009), 5 Cova de l'Or (Martí *et al.*, 2009), 6 Fuente Álamo (Tresserras, 2004), 7 La Almoloya (Molina, 2015), 8 Gatas (Oltra, 2010), 9 Fossa de Prats (Yáñez *et al.*, 2002a), 10 Tomba de Segudet (Yáñez *et al.*, 2002b), 11 Can Roqueta (Tresserras, 1997), 12 Ca l'Oliaire (Martín *et al.*, 2005), 13 Gavà (Tresserras, 2009), 14 Torrelló del Boverot (Clausell *et al.*, 2000), 15 Mas Castellar (Tresserras, 2000), 16 Castellon Alto (Parras et al., 2011), 17 Peñalosa (Manzano *et al.*, 2015), 18 Remojadero de Pescado (Sánchez and Cañabate, 1998), 19 Polideportivo de Martos (Sánchez *et al.*, 1998), 20 Marroquíes Bajos (Cañabate and Sánchez, 1997), 21 Cerro el Pajarillo (Parras *et al.*, 2015), 22 Puente Tablas (Parras *et al.*, 2015), 23 Las Atalayuelas (Sánchez *et al.*, 2011), 24 Puente la Olla (Cañabate and Sánchez, 1995), 25 Genó (Maya *et al.*, 1999), 26 Cova del Sardo (Tarifa, 2015), 27 Humanejos (Rios *et al.*, 2011), 28 Camino de las Yeseras (Rios *et al.*, 2011) , 29 Tira del Lienzo (Molina, 2015), 30 La Bastida (Molina, 2015), 31 Murviedro (Oltra, 2010), 32 A Froxa (Prieto *et al.*, 2005), 33 Caballeira do Espíritu Santo (Prieto *et al.*, 2009), 34 San Cosme 3 (Prieto *et al.*, 2005), 35 Monte Buxel (Prieto *et al.*, 2005), 36 Monte de Os Escurros (Prieto *et al.*, 2009), 37 Castro dos Ratinhos (García, 2010), 38 Bela Vista (Bastos, 2013), 39 Perdigões (Bastos, 2013), 40 Garvão (Rosada *et al.*, 2014), 41 Valdepernales (Rojo-Guerra *et al.*, 2008), 42 Abrigo de Carlos Alvarez (Rojo-Guerra *et al.*, 2008), 43 Los Dolientes (Rojo-Guerra *et al.*, 2008), 44 Puig Roig del Roget (Genera *et al.*, 2014), 45 Can Sadurní (Spiteri, 2012), 46 Tosal Montañés (Tresserras, 2006), 47 Valle de las Higueras (Bueno *et al.*, 2005), 48 Dolmen de Azután (Tresserras and Matamala, 2005), 49 San Bernardo (Guerra *et al.*, 2012), 50 El Nogalillo (Guerra *et al.*, 2012), 51 Almenara de Adaja (Guerra, 2006), 52 Valada do Mato (Salque *et al.*, 2015), 53 Mendandia (Salque *et al.*, 2015), 54 Los Cascajos (Salque *et al.*, 2015), 55 Kobaederra (Salque *et al.*, 2015), 56 Atxoste (Salque *et al.*, 2015).



2. Histogram presenting sample sizes in terms of the number of sites.

Small sample sizes: From the 52 sites with studies that report the number of analysed samples, barely 20% present sample sizes higher than the minimum accepted for statistical significance (30) (figure 2). In fact, only 50% of the studies report more than 10 samples. The extent of this problem has to be understood in a research field, archaeology, in which the access to remains from the past and the funds are neither easy nor unlimited. However, in the case of pottery and potsherds, which are usually the most common archaeological remains, these sampling problems could be easily solved.
***Preservation rates*:** It is common practice amongst international publications to report the amount of samples that yielded above the µ -> 5 µg/g of lipid threshold for significant presence of fats. The rate between all the analysed samples and those with a positive result is called the preservation rate and aims to provide an estimate for the number of samples that might yield significant residues. This problem rises from the actual impossibility to detect whether lipids survived in the potsherd without performing destructive analyses. Although when assessing the extent of post-depositional degradation processes, the possibility that the vessels never interacted with any fatty product must also be taken into account, low preservation rates might be explained by two possibilities: a) the environment in which pots were buried highly damaged the lipids embedded in the clay matrix, b) only a reduced amount of vessels ever contained fats. In the case of the Iberian Peninsula, its geography creates significant environmental differences between the seashore and inland. In order to take these possible variations into account (figure 1) sites have been grouped in regional clusters based on broad climatic conditions. Two regions (the North West and the Basque County) could not be evaluated due to lack of published data. Also, preservation rates were not obtained in sites with less than three samples. The other regions are presented in table 1. In order to account for possible wide general trends in the consumption of more or less fatty products, preservation rates for the four main prehistoric periods have also been reported.

TABLE 1. Preservation rates for each region and chronology in the Iberian Peninsula. Sites with just one sample analysed are included to show their little effect in the overall preservation rate.

		Number of shreds reported	Reported positive results	Mean Preservation rate	Number of sites
Regions	North East	175	51	**0.29**	10
	South East	190	79	**0.41**	7
	Centre	136	29	**0.21**	10
	Atlantic	47	15	**0.32**	2
Chronology	Neolithic	58	26	**0.45**	4
	Chalcolithic	87	18	**0.20**	7
	Bronze Age	267	105	**0.39**	12
	Iron Age	119	20	**0.17**	6
Sites with more than 3 samples		531	169	**0.32**	29
Sites with at least 1 sample		564	187	**0.33**	42

As it can be seen, regardless of prehistoric period or place in the Iberian Peninsula, preservation rates hardly exceed 50%. Moreover, only in a minority of sites (10%) (Martí *et al*., 2009; Molina, 2015; Manzano *et al*., 2015) the number of positive results is higher than the number of potsherds that yielded virtually no lipids. A comparison between these values and reports from other European regions (table 2) clearly suggests that the climate plays a key role in explaining why fewer percentages of pots present interpretable lipids.

Nevertheless, a third factor might be into play in order to explain preservation rates, the process of lipid extraction itself. Assuming no chemical procedure will perfectly extract all lipids, a choice must be made between strong base extractions (Regert *et al*., 1998; Evershed, 2008; Correa-Ascencio and Evershed, 2014), designed to fully recover all organic matter and the lipids more tightly bound to the ceramic matrix (Correa-Ascencio and Evershed, 2014) but partially degrading its chemical structures; or "soft" extractions (Evershed *et al*., 1990), which will preserve the integrity of the molecules but create false negatives. Therefore, where climatic conditions have allowed good lipid preservation, strong base extractions would reduce the interpretation potential while producing the same amount of false negatives. However, in highly hydrolysing and oxidising environments where fat would have already degraded, strong extractions would not deteriorate organic matter further (thus interpretation potential would remain the same) but significantly reduce the amount of false negatives (Correa-Ascencio and Evershed, 2014).

When reported in the literature, two main extraction procedures have been generally used in the Iberian Peninsula. The first is the classical Chloroform/methanol "soft" extraction (Evershed *et al*., 1990). This is the most internationally widespread procedure (Copley *et al*., 2005; Evershed *et al*., 2008) and, as a result, it has also been widely used in the Iberian Peninsula (Oltra, 2010; Parras *et al*., 2011; Bastos, 2013; Genera *et al*., 2014). The second more extensively used procedure is the use of a Soxhlet apparatus and a range organic solvents (Tresserras, 1997; 2004; 2006). Nonetheless, this last extraction is hardly acknowledged by their users (Clausell *et al*., 2000; Tresserras *et al*., 1999), making it difficult to evaluate the extractive capacity of their technique, although it would be safe to assume that only "soft" extractions were performed since the appearance of specific extractions for Mediterranean regions were just recently reported for the first time (Correa-Ascencio and Evershed, 2014; Gregg and Slater, 2010). In consequence, it might seem that a technique designed for environments with good organic matter preservation and weak lipid bonding has been repeatedly used for the Iberian Peninsula. In light of this fact, it should be evaluated whether the decrease of preservation rates for Mediterranean environments (table 2)

TABLE 2. Comparison of the Iberian preservation rate with other European regions.

Region	Preservation rate range	References
British Isles	50 to 60%	(Mukherjee *et al*., 2005; Evershed *et al*., 1997)
Eastern Mediterranean	20 to 30%	(Thissen *et al*., 2010; Evershed *et al*., 2008)
Italic Peninsula	12 to 24%	(Salque *et al*., 2012; Spiteri, 2012)
Iberian Peninsula	17 to 45%	Table 1

is partially caused by reported false negatives.

***Residue interpretation*:** Once lipids have been extracted and analysed, three types of residues have been commonly detected in the Iberian Peninsula: Beeswax (Heron *et al.*, 1994), degraded animal fat (Regert *et al.*, 1998) and well preserved fats. Plant oils and other highly interesting indicators such as thermal degradation (Raven *et al.*, 1997; Evershed *et al.*, 1995; Hansel *et al.*, 2004) are very scarcely reported.

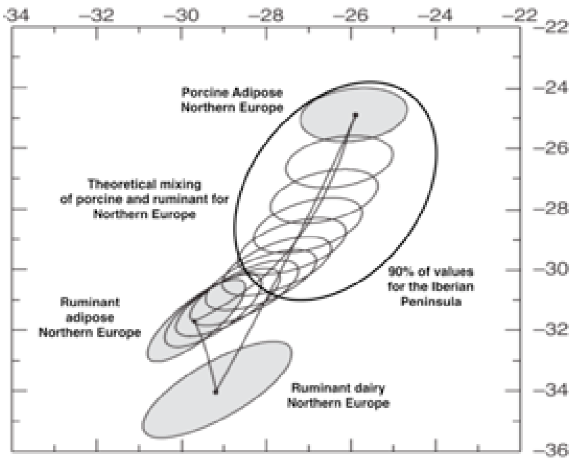
The interpretation of lipid profiles is not straightforward. Because analytical results yield data that is not directly usable by the archaeologist, lipid profiles must be translated into specific products such as animal fat, milk or honey. The method used for this translation is the biomarker concept. Following this principle, organic products can be defined by a group and quantity of molecules which, when detected in the chromatograms, will prove presence of that product (Evershed, 2008). Archaeological biomarkers for a wide range of products have been developed taking degradation effects such as hydrolysis and oxidation into account (Dunne *et al.*, 2012, p. 342). A key biomarker for the detection of milk, and the differenctiation between different types of animal fat and marine or terrestrial products is the $\delta^{13}C$ value for Plamitic ($C_{16:0}$) and Stearic ($C_{18:0}$) fatty acids. These values are obtained through GC-C-IRMS (gas chromatography coupled to combustion and isotope ratio mass spectrometry), which is complementary to the common GC-MS analysis and only performed when molecules in the sample have been correctly identified and separated. In the Iberian Peninsula, reports of GC-C-IRMS analysis of stearic and palmitic acids is scarce and only reported for some Neolithic and Bronze Age sites (Molina, 2015; Martí *et al.*, 2009; Oltra, 2010; Manzano *et al.*, 2015; Spiteri, 2012) But the interpretation of its values, although it holds a lot of potential for the understanding of prehistoric societies, presents some problems. Figure 3 depicts the area where results from the Iberian Peninsula tend to plot. This area is compared with the reference values reported by Copley *et al.* (2003), which have been commonly used as biomarkers. As it can be seen, the Iberian samples present a clear offset towards more enriched isotopic values. Experimental analyses performed at the island of Malta (Spiteri, 2012) provided values for animal and milk fatty acids that support this enrichment, which had already been detected for other world regions (Mukherjee *et al.*, 2005).

Therefore, it seems clear that a shift due to climatic conditions exist in the carbon isotopic values of organic molecules. These changes are rooted in the way plants, the base of the terrestrial food chain, incorporate carbon and engage in diverse fractionation processes. Many factors might modify the $\delta^{13}C$ values of plant tissues. Light, nutrients, temperature and water availability would make the values increase. However, deciduous trees, (woody plants present higher $\delta^{13}C$ values than herbaceous plants) and altitude might suggest values could also descend. Finally, as climate varies with the territory new more adapted species tend to appear (Heaton, 1999). These species present lower $\delta^{13}C$ values than its "less adapted" counterparts. Hence, values seem to differ up to 2‰ depending on the region (van Klinken *et al.*, 2000). If isotopic values from organic residues are interpreted without taking this possible shift into account, there is the risk of performing incorrect product assignations, thus affecting its archaeological significance.

RETHINKING THE SAMPLE PREPARATION AND INTERPRETATION

Taking all the limitations of the method presented in the previous section into account it is clear that different preservation environments need different analytical approaches. Following this idea, two changes on the way lipid organic residue analysis is performed in the Iberian Peninsula could highly improve its applicability to archaeology:

- Switch to specific extractions. New extraction procedures could reduce false negatives (Correa-Ascencio and Evershed, 2014; Gregg and Slater, 2010) and also



3. $\delta^{13}C_{18:0} - \delta^{13}C_{16:0}$ plot presenting the common European reference values against reported results from sites at the Iberian Peninsula. Original: Copley et al., 2003. Sources: Molina, 2015; Martí *et al.*, 2009; Oltra, 2010; Manzano *et al.*, 2015; Spiteri, 2012.

TABLE 3. Advantages and limitations of the Acidified Methanol extraction.

Advantages	Limitations
When performing GC-C-IRMS the extraction is quicker	Complex molecules are lost
Achieves higher lipid yields	Uses more dangerous reagents
Allows reduction of sample quantities	Might be more sensible to contamination
Capable of fully derivatising the sample	Still in late experimental stage
Hydrolyses the sample	

lower the amount of sample needed for analysis (Papakosta *et al*., 2015). The different factors in favour and against the acidified methanol extraction are presented in table 3 and, given that this is still a young procedure, it is recommended to always perform a first screening with a "soft" technique as a way to evaluate the extent of degradation. The acidified methanol extraction has already been used in contexts from other world regions with highly promising results in terms of preservation rates (Taché and Craig, 2015).

- Although paleodietary studies on bone collagen will need specific reference samples for the correct interpretation of the isotopic values, in the case or organic residues, milk can still be distinguished from adipose fats because the different biochemical processes in the animals digestive system act on top of the already enriched plant values. Mukherjee *et al*. (2005) suggest the use of the $\Delta^{13}C$ values ($\delta^{13}C_{18:0} - \delta^{13}C_{16:0}$), which correct for almost all of the environmental effects and allow an accurate interpretation of the results. Indeed, although this approach resolves the climatic issues, it also removes other variation factors which could be of archaeological interest; one of these is the possible mixture between different species of animal fats. When mixture of different types of species is suspected, the only way to assess it is to revert to the specific molecular values and apply the model formula proposed in the literature (Mukherjee *et al.*, 2005). Nevertheless, this exercise will need new reference fat values for regions in Southern Europe.

CONCLUSION

Rethinking the way lipid organic residue analysis is performed in the Iberian Peninsula might open the possibility to ask new archaeological questions that so far might seem difficult to answer. Was dairying a widespread practice with the arrival of the Neolithic? What was the extent of fish consumption by social groups located in the seashore? What was the role of beeswax, a part of the diet (honey) or a coating? It is clear that, by implementing the most optimised methods, new possibilities are open for discussion.

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