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In vino veritas. An assessment of current research of amphorae contents from Conimbriga (Portugal)

In vino veritas. Uma avaliação da investigação em curso sobre o conteúdo de ânforas de Conimbriga (Portugal)

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Abstract
This paper proposes an evaluation of current projects for identifying the contents of assumed wine transport amphorae recovered in the archaeological excavations in Conimbriga (prov. Lusitania, Portugal). The sampling strategy of known typologies of wine amphorae of Italic, Gaullish and Iberian origin, as well as other, locally produced typologies of uncertain use is described, as well as the methodologies of analysis, both by GC-MS and by LC-MS/MS.
The results of those analyses are presented in conjunction with the typological classification, as means to assess the weight of the production of wine destined to export in the context of the local agricultural activity.
Methodological issues concerning sample retrieval and analytical procedures are discussed in the context of planned future research.

Keywords:
Amphorae, Wine, Roman economy, Gas Chromatography, Liquid Chromatography

Resumo
Este artigo propõe uma avaliação dos projetos em curso para caracterizar o conteúdo das ânforas supostamente destinadas ao transporte de vinho recuperadas nas escavações arqueológicas de Conimbriga

(província da Lusitânia, Portugal). É descrita a estratégia de amostragem de tipologias conhecidas de ânforas de vinho de origem itálica, gaulesa e ibérica, e de outras tipologias de produção local e de utilização incerta, bem como as metodologias de análise, tanto por GC-MS como por LC-MS/MS. Os resultados destas análises são apresentados em conjunto com a classificação tipológica, como forma de avaliar o peso da produção de vinho destinada à exportação no contexto da atividade agrícola local.
Questões metodológicas relativas à recuperação de amostras e procedimentos analíticos são discutidas no contexto de futuras linhas de pesquisa.

Palavras-chave:
Ânforas, vinho, economia romana, cromatografia gasosa, cromatografia líquida

Introduction

Wine was an important part of ancient diet and food-related culture. It was also an important element of the ancient economy, both at local level and in long distance commerce. One difficulty is finding adequate means to assess that importance in those distinct levels, since various archaeological indicators are seldom equally relevant to different contexts: comparisons are not straightforward.

That is precisely the difficulty with amphorae, ceramic vessels typically for export in bulk over long distances, coexisting with small-scale, proximity distribution, which presumably occurred using other containers, like casks or wineskins.

This is an essential problem for the study of consumption centers, because it creates a discontinuity in the archaeological record, as shown in Conimbriga.

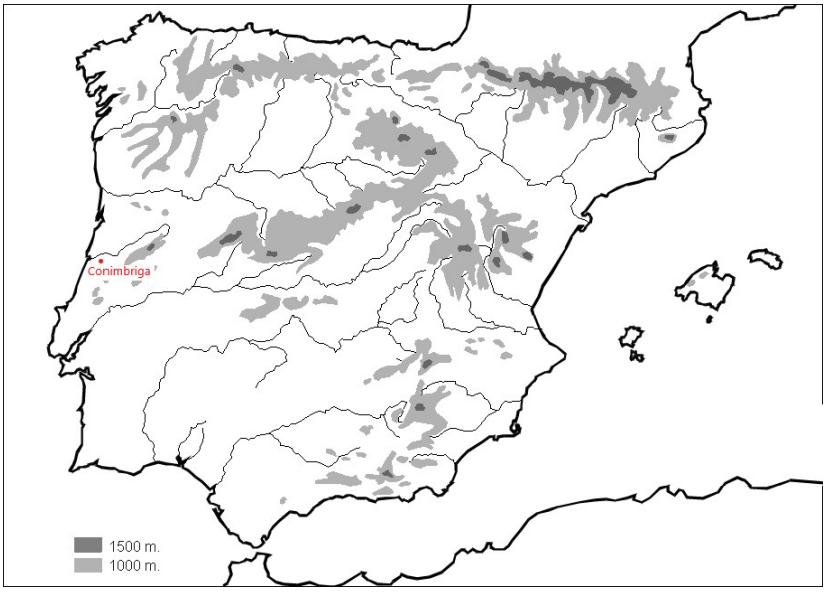

Figure 1 – Location of Conimbriga in the Iberian Peninsula.

The oldest Roman wine amphorae registered in Conimbriga are the Italic, along with the Greco-Italic form, reaching their peak with the Dressel 1 amphora. Despite having a weight/quantity ratio, the import of the Dressel 2-4 amphora does not keep up with the import levels of its predecessor, appearing in Conimbriga in smaller numbers. In the middle of the 1st century AD, there seems to have been a decline in Italic products imports in favor of Hispanic amphorae, particularly from Baetica, with the Haltern 70, Dressel 28 and *Urceus* amphorae. In fact, the most notable wine imports in the town are the Baetic amphorae, with the Haltern 70 amphora standing out for its significant number, and present on the site in all its variants, from the earliest to the latest productions. It should be noted, however, that several researchers defend the multipurpose use of this type. There is also the import of Gallic and Rhodian wine amphorae, but in small numbers, from the 1st century AD onwards.

Apparently, from the Flavian period onwards there was a drop in wine imports in Conimbriga, with the production of amphorae of a local/regional nature with the imitation of the Dressel 2-4, Gauloise 4 and 5 and other typologies and the Conimbriga 45/46 local amphora as explained below.

The Lusitanian production of Lusitanian 3 and later the Lusitanian 9 have been present since the 2nd century AD. Some researchers have associated their content with wine,[1] while others have linked it with fish,[2] but the issue is far from clear.

Only a few simple fragments of Late Roman Amphorae, such as LRA3, LRA4, and LRA5, were exhumed in the late antique period. This panorama of declining imports and presumably emerging local production poses a series of issues.

Lusitanian wine is a 21st century dilemma, if one considers that the two articles that set the scientific problems the research currently faces, date from final years of the last century.[3] Some issues still have not been resolved despite the time that has passed, and we'll mention just two that have a marginal incidence in the research carried out at Conimbriga:

– the first of these is the problem of casks, which, to some research is the single most important evidence for the whole issue.[4] Even if the item is not looked at that way, casks are a major argument for the alleged late disappearance of wine amphorae (the second problem).
– the similarity of the amphorae currently classified as Lusitanian 3 with the Gauloise 4 (a demonstrably wine dedicated amphora) has led to the same purpose identification being applied to the supposed Lusitanian copy. However, that assumption has been criticised on the grounds that Lusitanian 3 amphorae are clearly the precursors of Almagro 51C (a fish-product container) and were mainly produced in workshops otherwise dedicated to fish-products amphorae.

The other side of the problem is the identification of production facilities, the archaeological sites they are associated with, and their relation to broader occupation patterns in regions adequate for wine production. A general appraisal of these issues was presented by P. A. Pereira[5] as an overview for

1 Fabião 1998.
2 Diogo 1987; Mayet and Silva 1998.
3 Brun 1997; Fabião 1998.
4 Etienne and Mayet 2000; cf. Marliére 2002.
5 Pereira 2017.

the current issues, but research advancement is, of course, heavily dependent on new findings and systematic surveys, which are long in coming.

A parallel line to these concerns has been proposed, from an ethno--archaeological perspective, by I. V. Pinto:[6] the production of wine in the Roman period in *dolia* by the process of fermentation nowadays known in Southern Portugal as "curtimenta" (all the fermentation occurs in the *dolium* proper). This would explain an occupation pattern archaeologically characterised by a multiplicity of small sites producing exclusively *dolia* fragments and construction materials known in the region,[7] which would be the remains of small rural installations dedicated to cash crops in the context of larger agricultural explorations, as one of the current authors has argued.[8] *Dolia* are becoming a matter of interest for archaeologists,[9] but the fact they were probably a multi-purpose container (namely to the storage of olive oil too), doesn't allow for direct answers to the questions of wine production and transport.

This brief survey frames the problems wine amphorae research faces in a consumption site such as the Roman town of Conimbriga (or, for the case of Lisbon, as exemplified by V. Filipe[10]). There is the need to clearly identify import containers and local production ones, assess the relation of the latter with the occupation pattern of the surrounding landscape and explain the gaps and inconsistencies in the record (on the assumption that agricultural production patterns, although subject to innovation and adaptations, change ever so slowly, and that they never became abstemious).

1. The case of Conimbriga

Research in Conimbriga is focused on the study of the amphorae retrieved in archaeological excavation in the town, from 1873 to 2020.

6 Pereira 1997.

7 Mantas and Silliéres 1990.

8 Correia 2018; Correia and De Man 2010.

9 Quaresma et al. (in print).

10 Filipe 2023.

Figure 2 – Plan of Conimbriga in the late 1st century AD. Buildings where sample amphorae were excavated are indicated: 1 – Forum; 2 – South baths; 3 – Insula West of the baths; 4 - House of the fountains; 5 – Commercial building; 6 – House of the swastika; 7 – Amphitheatre.

The study of amphorae in Conimbriga emerged with the Franco-Portuguese excavations and the publication in volume VI of the *Fouilles de Conimbriga* of 58 amphorae (fragments or groups of fragments representing distinct individual vases) [11] The research project one of the co-authors of this manuscript (IB) is currently carrying out includes the study of all excavations conducted in the Roman city until the year 2020, recording more than 800 individuals, to date.

The import of wine amphorae in Conimbriga is second in the table of amphorae imports in the city, behind the imports of fish products, which are first.

The bulk of wine imports occurred from the Republic until the Flavian era. It was within the scope of institutional supply to the army that the first Roman wine amphorae arrived during the Republic,[12] taking advantage of the Atlantic route, which would significantly intensify to reach Britannia

11 Alarcão et al. 1976.

12 Imperial 2020.

and the *limes* in later periods. The flow increases with the intensification of relations between Romans and natives. As we have seen above, Italian wines domination ceases when Hispanic wines take over, since they had a better location for access to the Atlantic coast. From this period onwards, the import of wine amphorae decreased significantly, which seems to be related to the creation and consequent development of skills that lead to local wine production, as demonstrated by the production of local amphorae. An early conclusion of this research, then still in its preliminary stages, was precisely the identification of a hitherto unknown class of locally produced amphorae, of *sui generis* shape, destined to the transport of wine, named Class Conimbriga 45-46, in accordance with the numbering of fragments first published unclassified in 1976.[13] The morphological identity of these amphorae was manifest; the near location of the production was evidenced by the appurtenance of the ceramic paste to the same chemical groups of ceramic construction materials; and the transport of wine was confirmed by content analysis of the two best exemplars using GC-MS.

The existence of a local type of wine amphora attests to the fact that wine as a revenue production was important in the territory of the town. The research on this topic offers important insights, although it is insufficient for a clear explanation of production structures.

The Roman occupation of the city's territory evidences a villa-based agricultural exploration landscape, but it is certainly of a later date than the urban structuring of the territory. When late aulic architecture is removed from the chronological palimpsest, the pattern of rural occupation testifies to the ancient, pre-Roman roots of an occupation related to small land exploration structures, some of them of gentile origin, and to economic intensification, which found a particularly fertile field here.[14]

In the absence of significant survey projects in the territory, especially for the earliest period of territorial structuring under Roman rule, a broad picture is all that can be drawn. But a perfectly implemented structure is clear: the territory of Conimbriga was essentially articulated by the Olisippo-Bracara road, particularly in the segment joining Sellium to Aeminium, and the vast space surrounding the Mondego estuary.

13 Alarcão 1976: 87.

14 Correia and De Man 2010.

The area is dominated by a network of secondary roads, alternatives to the main one, which articulated the dispersion of small agricultural sites and larger ones, possible *vici*. In fact, part of a site is known to be included in this category (although certainly not a *vicus* as traditionally and legally characterized), significantly located in a road *trivium*.

The Eira Velha site,[15] occupied an open area, mid-slope, with an extension of around 0.5 hectare., c. 10km from Conimbriga, and corresponds to the evolution of a first isolated building, dating from the 1st century AD, which was added (and, at a certain point, partially replaced, implying its demolition), by another, which was perhaps preceded by a portico next to the road and which, through *fauces*, gave access to a central atrium. At the end of the 3rd century or the beginning of the 4th century AD, this building also underwent some renovations. In this second phase, another building is built on the opposite side of the *trivium*, which includes a wine press and a possible commercial establishment, a *caupona*. The building subsequently underwent some renovations, until its progressive abandonment.

One can resort to archaeo-ethnography and modern toponomastics and think that the site was a "Venda", a commercial site near a road, which sells local products to travellers and plays an important role in the provision of local community with imported goods. The role wine plays in this economy gets a very interesting example.

Although anecdotal, in the sense of being a single example among the plurality of sites that certainly existed, and therefore its representativeness is not capable of being adequately estimated, Eira Velha suggests that the evolution of the occupation of the territory, in its concrete aspect, does not happen through large phases motivated by Roman surveying interventions, but through the specific, gradual evolution of property and settlement structures, some of them most likely with pre-Roman roots, which reach their full conformation in the imperial era.[16]

The crucial point is then the clear determination that wine was central in the economics that one can guess behind the pattern detected in rural occupation and in consumption rhythms in the town.

15 Ramos and Simão 2012; Simão and Ramos 2014; Rasteiro 2023.

16 Correia 2024: 140-142.

The advancement of the amphorae study has offered new types of vases, which brings us to the current situation, where our research problems are:

– Is it possible to determine the variety of amphorae types that were locally produced?
– Is it possible to determine their uses?
– What is the position of those types in the general evolution of wine consumption habits in the town?
– How does that relate to wine production structures in the territory?

The answer to the first question is yes, and it is being addressed with comparative physical and chemical analyses of amphorae and common pottery of local origin.

The answer to the last question falls out of the scope of this paper; it will concentrate especially on the second question and touch marginally on the third.

2. Experimental

Organic residue analysis from selected amphorae is clearly a major tool in the present research for detecting ancient wine molecules preserved in archaeological material, especially pottery. Despite the success of the current analytical methods,[17] recent work has pointed out that due to the ambiguity of the selected biomarkers, they do not provide indisputable evidence for the detection of ancient wine residues in pottery without an explicit archaeological context.[18] In addition, other difficulties in identifying wine residues are associated with the high solubility of its components, resulting in poor preservation. These issues justify the evaluation of different and complementary analytical methods. Therefore, two different analytical techniques were used in this study: Gas Chromatography-Mass Spectrometry (GC-MS) and Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS). The GC-MS analysis followed a well-established technique

17 Barnard et al. 2011; Garnier and Valamoti 2016; McGovern 2017.

18 Drieu et al. 2020.

widely used in archaeology. The LC-MS/MS, on the other hand, adopted a novel analytical technique that targets new ancient wine biomarkers (Elezi et al. in preparation).

Typology **Method**	**Conimbriga 45-46 (see 4.2.6)**	**Gauloise 4, local (see 4.2.7)**	**Dressel 2/4, local (see 4.2.8)**	**Dressel 28, local (see 4.2.9)**
GC-MS (see 3.3.1-4)	Correia et al. 2015	Samples A, B, C	Sample D	
LC-MS/MS (see 3.3.5-7)	Sample 3495	Sample 3496		Sample 3498

Table 1 – Summary of conclusive analyses.

2.1 Gas Chromatography-Mass Spectrometry (GC-MS)

2.1.1 Sample preparation and analysis

The samples were collected with a clean scalpel and grounded to a fine powder with an agate mortar and pestle, and 7 mL of a mixture of chloroform:methanol (2:1 v/v) was added to each sample. The mixtures were vortexed, extracted in a sonic bath for 20 min and centrifuged at 2500 rpm for 15 min. The supernatants (solvent) were transferred with Pasteur pipettes and the extraction procedures were repeated. Each sample supernatants were combined and dried at 40 °C using a gentle stream of nitrogen. The resulting dried extracts were re-suspended with *n*-hexane and derivatized with N,O-bis (trimethylsilyl) trifluoroacetamide with 1% of trimethylchlorosilane (BSTFA + 1% TMCS) in a microwave oven (700 W, 30 s). After removing the derivatizing agent in excess under a gentle stream of nitrogen, the extracts were redissolved in *n*-hexane and analyzed by GC-MS.

The chromatographic analyses were performed with a Shimadzu GC2010 gas chromatograph coupled to a GCMS-QP2010 Plus Mass Spectrometer device. The equipment was operated in full scan mode, with the following experimental conditions:

a) column Zebron ZB-5HT (15 m length, 0.25 mm I.D., 0.10 µm film thickness) using helium as carrier gas with a constant flux of 1.5 mL min^{-1};
b) injection volume of 1 µL;
c) injector temperature of 250 °C;
d) heating program: 50 °C for 2 min, 50 to 300 °C (10 °C min^{-1}), hold at 300 °C (5 min), 300 °C to 400 °C (10 °C min^{-1}), hold at 400 °C (5 min), totalling 47 min;
e) ionization mode, electronic impact at 70 eV;
f) ion source temperature of 240 °C and interface temperature of 280 °C;
g) scanned masses from *m/z* 50 to 1090.

The identification of compounds was based on the analysis of fragmentation patterns and the comparison of resulting spectra with spectra from the commercial libraries Wiley 8 and Nist17.

	Sample	Mass used in the analysis
Sample A	B6	0.4762 g
Sample B	72 B Fig.7	0.3830 g
Sample C	66-F-1 /67/12	0.3688 g
Sample D	64-G-IX.35 - Canalização/72/5	0.6742 g

Table 2 – Samples analysed by GC-MS.

2.2 Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS)

2.2.1 Sample preparation and analysis

The archaeological ceramic samples were pulverized using a pestle and mortar. 2 g of pottery powder was added to glass test tubes and trea-

ted with 5 mL of Methanol/DI Water/Formic Acid mixture 50:50:0.1 (v/v/v). The samples were vortexed and centrifuged at 2000 xg for 15 min and the supernatants were transferred into new glass test tubes. The extraction procedures were repeated adding 3 mL of the mixture, the supernatants were pulled into the new tubes, and 100 picomoles of Daidzin in Ethanol were added to all samples as an internal standard for potential quantification analysis. The samples were dried in a vacuum concentrator for 4 hours. The dried samples were reconstituted in 100 µl of Methanol/DI Water/Formic Acid (95:5:01) (v/v/v), vortexed thoroughly, and centrifuged at 2000 xg for 5 min. The supernatant was transferred to HPLC vials, and 25 µl was injected into an LTQ_XL mass spectrometer system for analysis.

The analyses were conducted at the UCLA Pasarow Mass Spectrometry Laboratory. A targeted LC-MS/MS assay was developed on a Thermo Scientific Linear Ion Trap LTQ-XL mass spectrometer for Oenin (OE), Vitisin A (VA), Vitisin B (VB), and Daidzin (DA) monitoring transitions OE 493-331 *m/z*, VA 561-399 *m/z*, VB 517-355 *m/z*, DA 417-255 *m/z*). The instrument was set to scan in positive ion mode and was coupled to a Dionex Ultimate 300 HPLC system (Thermo Scientific) through a reversed-phase GL Science analytical column (Inert Sustain 2 µm, Phenyl 150 x 2.1 mm). The HPLC method utilized a mixture of eluents A (99.9:0.1 v/v Water/Formic Acid) and B (99.9:0.1 v/v Acetonitrile/Formic Acid), and a gradient was used for the elution of the compounds (min/%B: 0/5, 5/5, 12/40, 26/75, 28/5, 40/5). A fragment ion originating from each compound was monitored at a specific LC retention time to ensure specificity and accurate identification in the complex archaeological ceramic sample.

2.3 Results

2.3.1 Sample A - B6 (GC-MS)

Sample B6 revealed substantial chemical markers of resins from the *Pinaceae* family, such as dehydroabietic acid and 7-oxo-dehydroabietic

acid, the two most abundant compounds in the chromatogram, as well as pimaric and isopimaric acids, retene, and phenanthrene (Fig. 3). Simonellite is an intermediate compound formed by the thermal degradation of dehydroabietic acid during the production of pitch from natural resin, under high-temperature conditions or through wood distillation in low-oxygen environments (Hjulström et al. 2006; Fujii et al. 2019), with retene, one of the most intense compounds in the chromatogram, as a final product. The detection of retene, methyl dehydroabietate, and the methyl ester of 7-oxo-dehydroabietic acid unequivocally support the presence of pitch produced by the distillation of woods from the *Pinaceae* family. Different wine biomarkers were detected, particularly benzoic, vanillic, and azelaic acids. The presence of these biomarkers, alongside with the detection of pitch, a sealing material commonly used as a waterproofing agent in ceramics, suggests that the vessel might be a wine amphora. However, the absence of tartaric and succinic acids introduces some uncertainty to this conclusion.

Lipid degradation products, including substantial quantities of monoacylglycerols such as 1-monopalmitin and 1-monostearin, along with lesser amounts of diacylglycerols such as 1,2-dipalmitin, 1,3-dipalmitin, 1,2-distearin, 1,3-distearin, 1,2-distearoylglycerol, and 1,3-distearoylglycerol, were also detected. The palmitic to stearic acid ratio (P/S) of 1.2 suggests a predominance of animal fats, which seems aligned with the presence of cholesterol, a biomarker for animal fats. However, small peaks of squalene seem to indicate a possible contamination of the ceramic materials, likely due to handling without gloves and the consequent transfer of fats from human skin. In result, it is unclear whether the fats are of archaeological origin or a result of recent contaminations. Additionally, we detected 2,3,4,6-tetrachlorophenol, a precursor to pentachlorophenol, an organo-chlorine compound used mainly as a sapstain control agent to protect newly cut wood surfaces against fungal attack, that would integrate the composition of wood preservation products. This compound points to a potentially contamination of ceramics due to their prolonged storage in a wood cabinet.

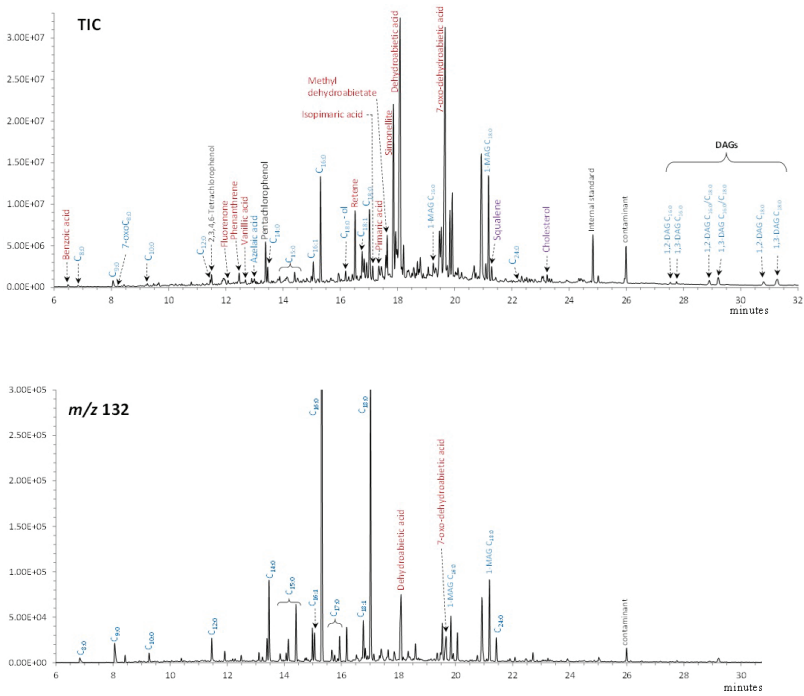

Figure 3 – Total Ion Chromatogram (TIC, Fig.3a) and Extracted Ion Chromatogram (*m/z* 132, Fig.3b) of sample B6. Cn:x, fatty acids with carbon length n and number of unsaturation x; MAG, Monoacylglycerols; DAG, diacylglycerols.

2.3.2 Sample B - 72_B_F1(7) (GC-MS)

Sample 72_B_F1(7) also shows clear evidence of waterproofing with pitch, namely by the presence of dehydroabietic acid, 7-oxo--dehydroabietic acid, and simonellite, characteristic of Pinaceae family resins (Fig.4). The detection of retene suggests that the resin was exposed to high temperatures, while the methylated derivatives of abietic acid (methyl dehydroabietate and methyl ester of dehydroabietic acid) indicate that it was distilled into pitch in the presence of wood. This sample shows no traces of wine biomarkers. Like the previous sample, 72_B_Fig7b exhibited significant peaks of degraded lipid compounds, with substantial amounts of monoacylglycerols such as

1-monopalmitin and 1-monostearin, the dominant peak, and smaller amounts of diacylglycerols including 1,2-distearin, 1,3-distearin, 1,2-distearoylglycerol, and 1,3-distearoylglycerol. Important peaks of triglycerides (non-degraded fats) such as $C_{57}H_{104}O_6$ (triolein), $C_{55}H_{102}O_6$, and $C_{53}H_{100}O_6$, $C_{53}H_{98}O_6$, $C_{51}H_{96}O_6$ were also detected. The palmitic to stearic acid ratio was 1.4, suggesting a predominance of animal fats. Again, the combined presence of cholesterol and squalene proposes the sample contamination from handling without gloves. Additionally, there are also indications of contamination with wood preservation products.

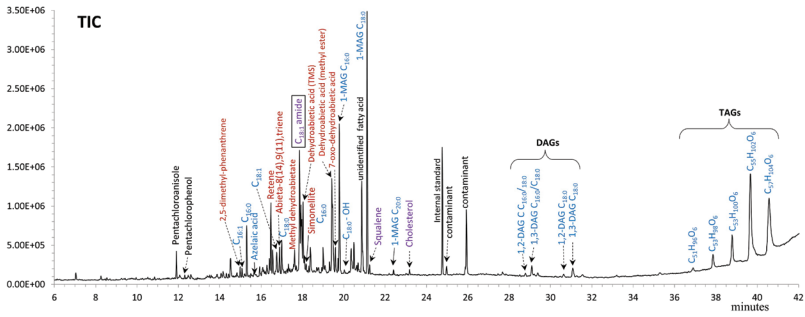

Figure 4 – Total Ion Chromatogram (TIC) of sample B - 72_B_F1(7). Cn:x, fatty acids with carbon length n and number of unsaturation x; MAG, Monoacylglycerols; DAG, diacylglycerols

2.3.3 Sample C - 66_F1 (GC-MS)

Unlike the remaining samples, 66_F1 showed no evidence of waterproofing with resins or the presence of wine tracers. Its primary signature indicates fat compounds (Fig. 5).

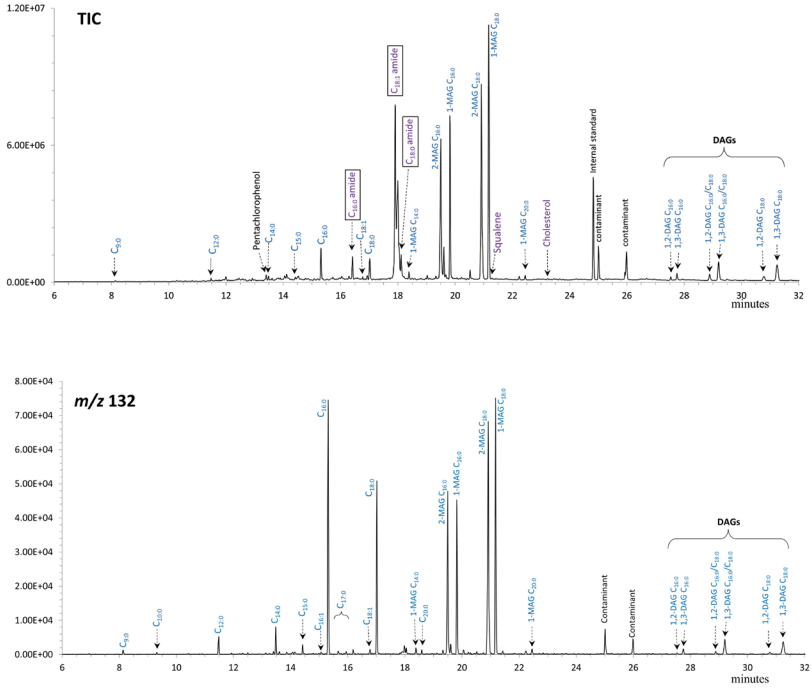

Figure 5 – Total Ion Chromatogram (TIC, Fig.5a) and Extracted Ion Chromatogram (*m/z* 132, Fig.5b) of sample C - 66_F1. Cn:x, fatty acids with carbon length n and number of unsaturation x; MAG, Monoacylglycerols; DAG, diacylglycerols

2.3.4 Sample D - 64-G-IX.35 (GC-MS)

Sample 64-G-IX.35 - Cano /72/5 presented a chemical signature which is aligned with sample B6. In fact, it is particularly clear the sample waterproofing with pitch produced from *Pinaceae* resins, namely by the presence of abietic acid, dehydroabietic acid (the dominant peak), 7-oxo--dehydroabietic acid, pimaric acid, isopimaric acid, and simonellite (Fig.6). No clear evidence of wine markers was detected, besides benzoic acid.

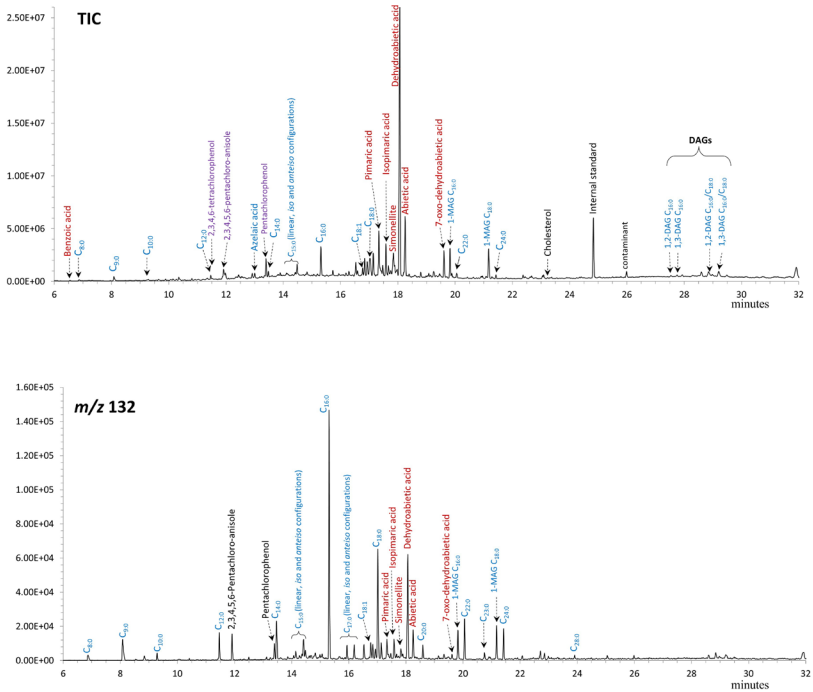

Figure 6 – Total Ion Chromatogram (TIC, Fig.6a) and Extracted Ion Chromatogram (*m/z* 132, Fig.6b) of sample D - 64-G-IX.35. Cn:x, fatty acids with carbon length n and number of unsaturation x; MAG, Monoacylglycerols; DAG, diacylglycerols

2.3.5 Sample 3495 – 71.CRY.N.cano (LC-MS/MS)

In sample 3495 originating from a Conimbriga 45-46 amphora, Vitisin A molecules were detected (Fig.7). Vitisin A is a pyranoanthocyanin formed during the fermentation of red and black grapes indicating the presence of wine residues (Elezi et al. In preparation).

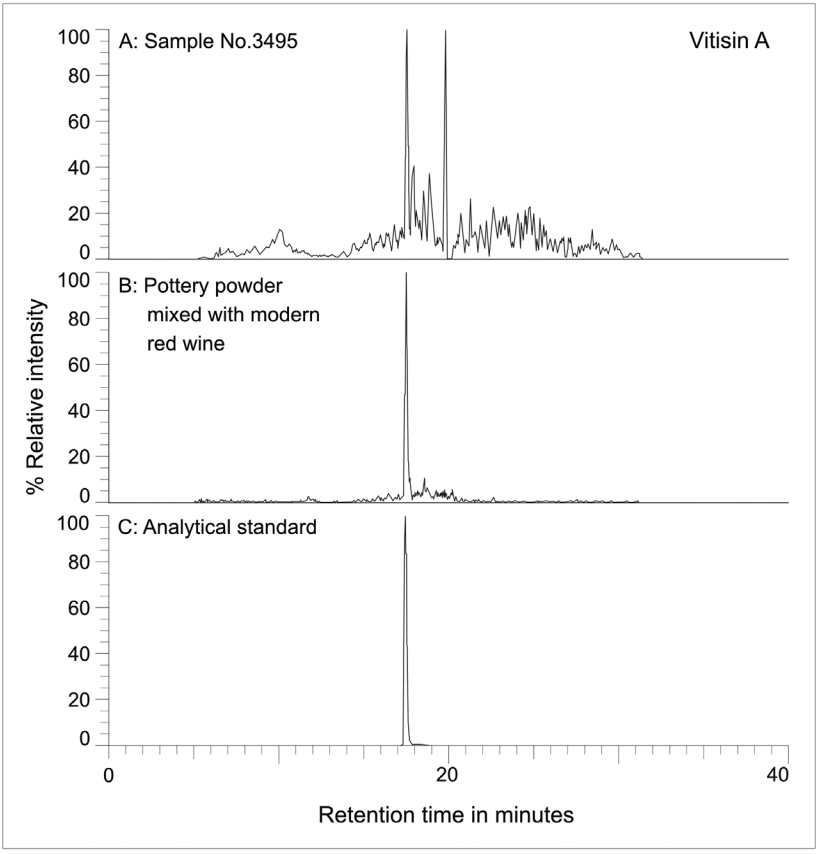

Figure 7 – LC-MS/MS chromatograms showing the response signal of Vitisin A in a) the archaeological sample 3495, b) a mixture of pottery powder with modern red wine used as a positive control, c) commercial analytical standard.

2.3.6 Sample 3496 – 66.U(11) (LC-MS/MS)

Sample 3496, a fragment from an imitation of Gauloise 4 amphora, showed similar chemical traces to sample 3495, with the detection of Vitisin A molecules (Fig.8).

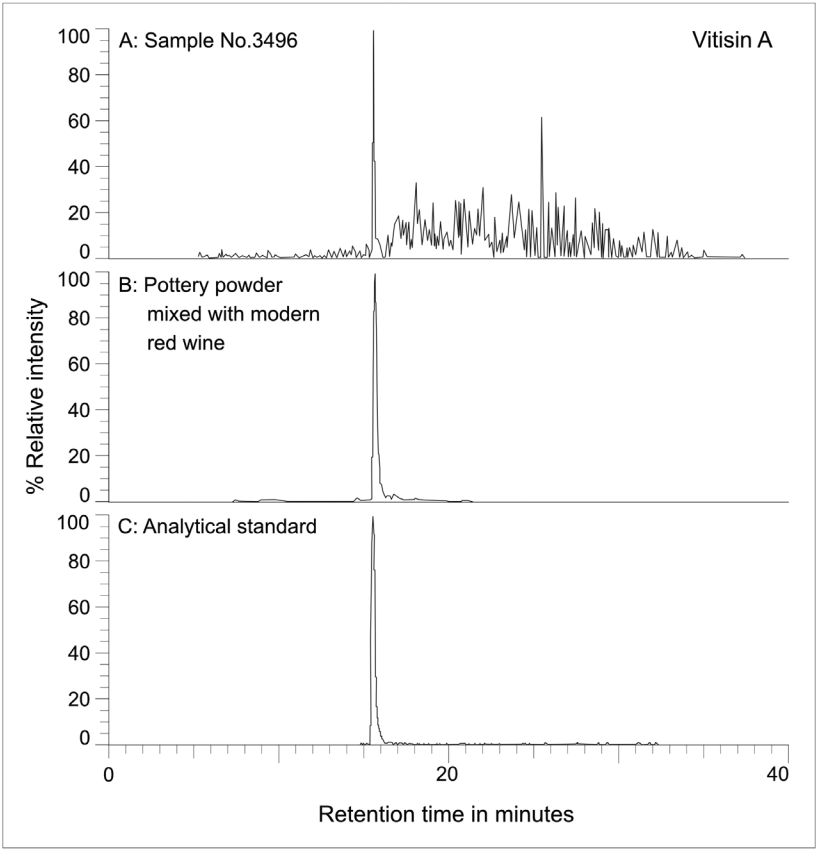

Figure 8 – LC-MS/MS chromatograms showing the response signal of Vitisin A in a) the archaeological sample 3496, b) a mixture of pottery powder with modern red wine used as a positive control, c) commercial analytical standard.

2.3.7 Other (LC-MS/MS)

Other analyses proved inconclusive.

3. A preliminary overview of research results

3.1 The analyses

We would like to present a first assessment of content analyses in selected amphorae types, from Conimbriga, with the purpose of pointing some general ideas on the wine consumption and production in the town and of establishing some basic principles for future research, which is obviously a necessity.

We are dealing with three sets of analyses:

– In the first place, the analyses carried out in 2015 by GC-MS on two amphorae of the Conimbriga 45-46 class. The sampled vases were the definition vases for the class, two well preserved exemplars from protected stratigraphical contexts (a drain in the forum, a deposition layer in a demolished shop).
– Second, we have the four analyses by GC-MS on other classes of presumably regional productions of amphorae. Provenance contexts for the fragments do not offer the same selected characteristics, but scarcity of material does not allow to be demanding on the matter.
– The same applies to the third set of analyses by LC-MS/MS, on a large sample of amphora typologies, mostly assumed to be wine carriers (but two of them of disputed use).

Results of those analyses are divided in 5 different groups:

a) First set of GC-MS; conclusive results for wine (2 samples);
b) Second set of GC-MS; unclear results, probably wine (3 samples);
c) Second set of GC-MS, inconclusive results (1 sample);
d) Set of LC-MS/MS; conclusive results for wine (2 samples);
e) Set of LC-MS/MS; inconclusive results (13 samples).

3.2 Typological implications

The analyses described above were made on 19 samples of 12 different typologies. With these, we must bear in mind the results of the first analyses, mentioned in a) just above. The typological and chronological characteristics of the sampled amphorae are as follows:

3.2.1 Dressel 1

Sample 1 - 67 CRY 7 (6)- solid bottom eroded at the tip, coated with resin. Probable Dressel 1 amphora with paste typical of the Campania area. Found in the Franco-Portuguese excavations, more precisely from the Forum. Chronology republican to the beginning of the principate.

Sample 2 - 70 TH I 22- Solid bottom eroded at the tip, coated with resin. Probable Dressel 1 amphora with paste typical of the Campania area. Found in the Franco-Portuguese excavations, more precisely from Southern Bath. Chronology republican to the beginning of the principate.

3.2.2 Dressel 2-4

Sample 3 - A 17 S 2 (5)- Solid bottom, rounded at the end, most likely belonging to a Dressel 2-4 amphora of Italic manufacture. It comes from the excavations of the House of the Fountains. Chronology prior to the last quarter of the 2nd century AD.

Sample 4 - 72 B F1 (1A)- Solid bottom rounded at the end, most likely belonging to a Dressel 2-4 amphora of Italic manufacture. From the excavations of J. Alarcão in the House of the Swastika Mosaic. Chronology: 1st century AD.

3.2.3 Haltern 70

Sample 5 - 69 TEM I N (3)- Filled-in bottom, conical profile with a rounded base. Probable Haltern 70 amphora of Baetic (Guadalquivir)

manufacture. From the Franco-Portuguese excavations, specifically from the Forum. Chronology: will be around the end of the 1st century BC until the 1st century AD.

Sample 6 - 68 ESP 3 (1)- Filled-in bottom, conical profile with a rounded base, belonging to a probable Haltern 70 amphora of Baetic manufacture (Guadalquivir). They come from Franco-Portuguese excavations, more specifically from the Forum. Its chronology will be around the end of the century BC until the 1st century AD.

3.2.4 Rhodian

Sample 7 - 66 G VIII 41 (7)- Belly of probable Rhodian-type amphora, from the eastern Mediterranean (Fouilles VI, nº46). They come from Franco-Portuguese excavations, more specifically from the Forum. Its chronology will be before the 19th century. AD

3.2.5 Dressel 28

Sample Sample 8 - 67 CRY 2 (4)- Bilobed frame edge of a probable Dressel 28 of probable Lusitanian manufacture. They come from Franco--Portuguese excavations, more specifically from the Forum. Its chronology will be from the second half of the 1st century AD.

3.2.6 Conimbriga 45-46

Sample Sample 9 - 71 CRY N Cano- Conímbriga amphora belly 45/46 of local/regional manufacture. They come from Franco-Portuguese excavations, more specifically from the Forum. Its chronology will be around the second half of the 1st century AD. GC-MS analysis published in Correia et al. 2015, see 4.1 a), above; LC-MS/MS analysis, see 3.3.5 above. Fig. 9, 18.

3.2.7 Gauloise 4 (local production)

Sample A - B6 – Rim rounded to the outside, straight neck and elliptical section handle. Probable imitation of a Gauloise 4 amphora, of local production. There are some incisions on the external surface of the neck. From the excavations of J. Alarcão, more precisely from zone B, from the courtyard between the Stores to the South of Road and House of the Swastika. Flavian period. GC-MS analysis, see 3.3.1 above.

Sample B - 72 B F1 (7) – Rim with rounded section, thickened on the external side. Concave curve lip. Probable imitation of a Gauloise 4 amphora, of local production. There are incisions on the outer wall of the neck. Same provenance and chronology. GC-MS analysis, see 3.3.2 above.

Sample C - 66 F (1) - Rim with rounded section, thickened on the external side. Concave curve lip. Probable imitation of a Gauloise 4 amphora, of local production. There are incisions on the outer wall of the neck. Same provenance and chronology. GC-MS analysis, see 3.3.3 above.

Sample 10 - 66 U (11)- Gauloise 4 imitation amphora belly of local/regional production. They come from the Lojas south of Via, from Professor Alarcão's excavations. Its chronology will be before the 3rd century AD. LC-MS/MS analysis, see 3.3.6 above. Fig. 9, 5.

3.2.8 Dressel 2-4 (local production)

Sample D - 64 G IX 35 - canalização/72/5- Infilled bottom with rounded base. Likely a local imitation of Dressel 2-4. From the Franco-Portuguese excavations, specifically from the drainage of the Flavian forum. Flavian period. GC-MS analysis, see 3.3.4 above.

Sample 11 - 2004 IWT (4)- Hollow bottom, with rounded base of a probable imitation Dressel 2-4 amphora, of local/regional production. From the Insula west of the Bath. Flavian period. Fig. 9, 9.

3.2.9 Dressel 28 (local production)

Sample 12 - 64 B 13 (3)- Rim in bilobed frame of a probable Dressel 28 amphora of local/regional production. From the House of the Swastica, J. Alarcão's excavations. Its chronology points to the 1st century AD. Fig. 9, 2.

3.2.10 Indetermined local production (Conimbriga 1)

Sample 13 - 2004 IWT (2)- Subtriangular rim with a straight neck, from which two elliptical section handles of a Conimbriga 1 amphora emerge. From the Insula west of the Baths. Its chronology will be pre--Flavian or Flavian. Fig. 9, 16.

3.2.11 Lusitanian 3

Sample 14 - 2012 R2 D (13)- Lusitanian 3 amphora belly and bottom. Lusitanian production. From the street north of the Forum. 2nd century AD.

3.2.12 Lusitanian 9

Sample 15 - 2018 G XVII 35/40 (6)- Lusitanian amphorae and rim 9. Its production is Lusitanian. From the Amphitheatre. Its dating falls between the end of the 20th century. II AD until the beginning of the 4th and 5th centuries. AD.

We present a summary of analytical conclusions described above in relation to these typologies.

	Typology											
Assumed content for the typology	Dressel 1	Dressel 2-4	Haltern 70	Rhodian	Dressel 28	Conimbriga 45-46	Imit. Gauloise 4	Imit. Dressel 2-4	Imit. Dressel 28	Conimbriga 1	Lusitanian 3	Lusitanian 9
Wine	e)	e)	e)	e)	e)							
Fish											e)	e)
Unknown a), b) and d) = wine transport						a), d)	b), c), d), e)	b), e)	e)	b), e)		

Table 3 – Summary of results

The determination that we are dealing with locally produced amphorae was made by concurrent analyses. In 2015[19] chemical analyses of Conimbriga 45-46 amphorae were compared to the evidence available at the time, which was the characterization of ceramic building materials;[20] the results were very clear: there was no significant difference between the composition of the clays used in one and the other class of materials.

Recently, other classes of amphorae that have emerged in our study of the collection from Conimbriga, fragments that show easily recognisable forms, but with fabrics that do not match the known productions of those formal classes, were analysed in combination with local coarse orange pottery, to which they seem – at the naked eye – absolutely similar. The early conclusions about the class Conimbriga 45-46 were confirmed, and the empirical appreciation was proved right: these amphorae of forms Dressel 2-4, Dressel 28, Gauloise 4 and the ones that we should call Conimbriga 1 were produced with the same material that was used to produce the abundant orange pottery retrieved in every Roman context in the town.[21]

19 Correia et al. 2015.

20 Triães 2003, 2016.

21 Buraca et al. i.p. a); i.p. b).

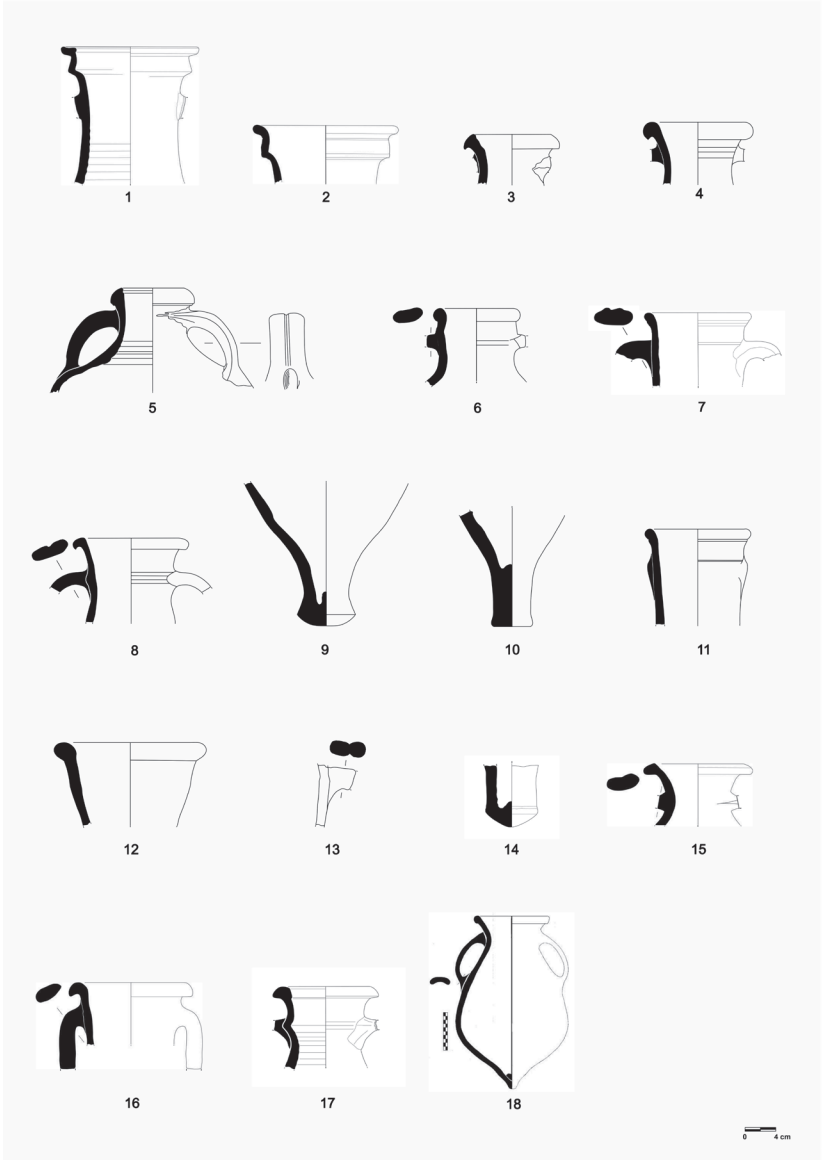

Figure 9 – Locally produced amphorae from Conimbriga. 1-2: imitation of Dressel 28; 3-8: imitation of Gauloise 4; 9-14: imitation of Dressel 2-4; 15: imitation of Gauloise 5; 16: Conimbriga 1; 17: Conimbriga 2; 18 - Conimbriga 45-46 (reconstruction).

Conclusions

The first conclusion from our work emphasizes the importance of the sampling procedure and the criteria for amphora selection it uses. Primary deposition contexts should be preferential and the ceramic fragments that, considered their good preservation, suggest they have not been subject to major post-depositional processes, offer the best possibilities for conclusive identifications of products.

Fragments that probably have moved a lot in the general sediment of the sites, subject to undetermined processes and various substances, produce inconclusive results and are of reduced value.

With these restrictions, the results of our different (in time, in method, in set of samples) analyses show no significant inconsistencies throughout. That should reassure us of the robustness of the following observation:

- Results strongly indicate that locally produced amphorae were destined to transport wine; 6 samples give indications of that, ranging from the probable to the definite, *versus* 13 inconclusive results. But future research should look to better sampling and enrich these results with further detail.

Unfortunately, discussed typologies, such as Lusitanian 3 and 9 amphorae get nonadditional indications about their purpose. Overall, and all evidence considered, from our present point of view, it is likely that some early variants of Lusitanian 3 were, at best, a multi-purpose container[22]; Lusitanian 9 still has no evidence for that, and we think a fish-products destination seems more likely.

Although the content of two amphorae was identified as red wine through the LC-MS/MS method, the understanding of what kinds of wine were imported or produced, it is dependent on further careful sampling and more analyses.

But, overall, it is important to demonstrate that there are currently available technologies that allow for such questions to be posed, something unexpected a couple of decades ago.

The opening of avenues of research on these matters will, no doubt, contribute to enhance significantly our knowledge of ancient economy, both in the agricultural and commercial domains, with valuable insights into consumption patterns, sociability and gastronomy.

22 Cf. Fabião 2019.

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