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MRS, EDXRF and GC–MS analysis for research on the ritual and funerary areas of *Cerro de los Vientos* (Baeza, Jaén, Spain). Native and Eastern Mediterranean influences

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ABSTRACT

This paper is about the results obtained from the physico-chemical analysis of the Orientalizing (7th century BC) archaeological materials retrieved from the ritual and funerary areas of the site *Cerro de los Vientos* (Baeza, Jaén, Spain). The samples under study are native tradition and Phoenician ceramic vessels, and beads of several colours. The analysis used Micro Raman Spectroscopy (MRS) with portable and laboratory equipments, Energy Dispersive X Ray Fluorescence (EDXRF) and Gas Chromatography-Mass Spectrometry (GC–MS). Joint use of these techniques allowed to examine the interaction between the native community of *Cerro de los Vientos* and the Eastern Mediterranean influence by populations of Phoenician origins.

Use of MRS and EDXRF helped identify the mineral and elemental composition of the decorations of the native and Phoenician ceramic vessels, and also of the red and blue beads. Hematite, goethite, amorphous carbon and graphite were recorded in the native pottery. Hematite and manganese oxide were recorded in the Phoenician pottery. The red beads were made of carnelian, a variety of chalcedony that is not found in the Iberian Peninsula. The blue beads were made of glass paste.

GC–MS contents analysis identified beeswax in most vessels, both native and Phoenician. The beeswax in native and Phoenician vessels from the area for the funerary feast and in the funerary urns laid in the graves leads to an interpretation in terms of hydromel consumption during feasts and offerings of honey in urns.

1. Introduction

The archaeological site *Cerro de los Vientos* lies on the hillside after which it is named, on the basin of the Guadalquivir, in the administrative term of Baeza (Jaén, Spain). The roadworks on motorway A-316 unearthed 14 structures, of which at least three were identified as cremation graves. All were partially dug into the bedrock. Despite the archaeological damage of the site, two distinct areas seemed to emerge: a ritual and a funerary one (Fig. 1). The site *Cerro de los Vientos* sets in the so-called Orientalizing period (7th century BC) (Lechuga and Soto, 2017).

The data gathered during site excavation and the research on the archaeological materials retrieved from the structures outline the material footprint of a small cemetery dating back to a complex period of hybridisation and of social and cultural transformation. The Orientalizing period witnessed the transition from the recent Late Bronze to the Early Iberian period. At that time, the local elites tried to strengthen their prevalence based on privileged links with the Phoenician trade networks, by which they gained access to exotic items as symbols of power in their communities (e.g. black and red oxidation firing wheelmade pottery, silver and bronze rings and beads made of valuable materials). In a way, the native elites were both actors and beneficiaries of the Phoenician colonization, insofar as they were the emerging aristocracies that would over time lead to the development of the culture of the Iberians (Aubet, 1978).

The ritual area consists of two fairly damaged oval-shaped cavities on the ground of approximately one meter in diameter (structures E28 and E32). The lack of bone remains suggests that these cavities were not graves (Fig. 2). The ceramic group of structure E28 is particularly worthy of note. Three of the nine vessels retrieved from this structure are native tradition, hand-made, reduction firing bowls. Two of these bowls (14-1021 and 8-1022) show black, red and yellow geometric decoration. The ceramic group of this structure also contains a set of

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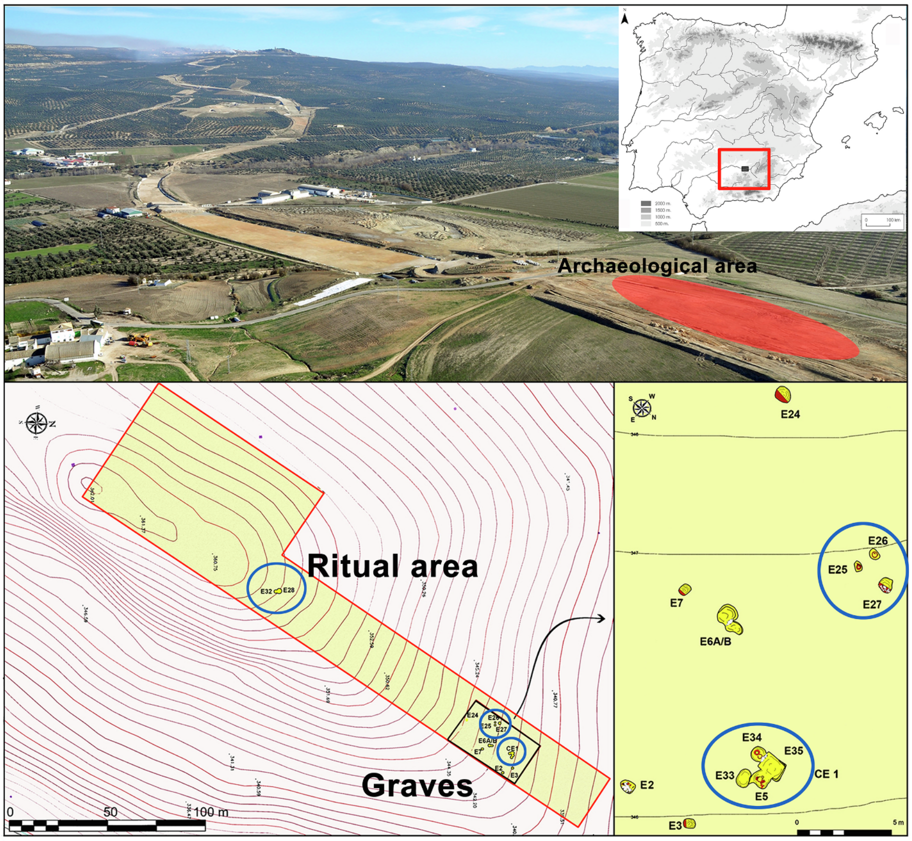

Fig. 1. *Cerro de los Vientos*. The ritual and funerary areas.

Fig. 2. Structures E28 and E32.

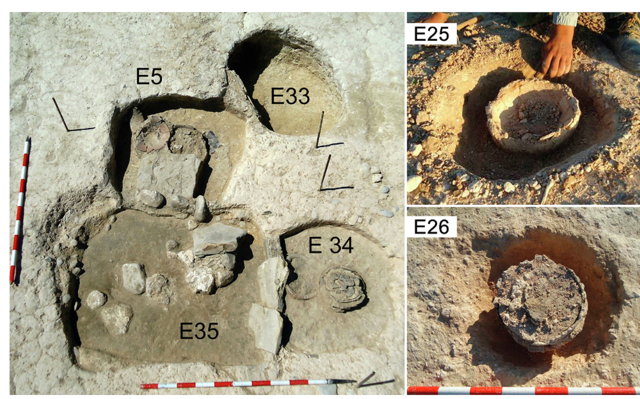

Fig. 3. Structures E5, E35, E33, E34, E26 and E25.

three storage vessels with red decoration and a plate decorated with black and red bands (15-1020), all of a distinct Phoenician influence and manufactured by oxidation firing. The two remaining items in this ceramic group are two slow wheelmade, oxidation firing bowls or plates. Not showing the black and red decoration and being rather crudely made, they are hypothesized to be imitations of Phoenician plates like the one found in this structure.

The second area holds the remaining structures, including the cremation graves which display similar building properties: they are rather shallow (10–30 cm) circle/oval-shaped graves arranged in two groups: one southeast of structures E2, E3, E5, E33, E34, and E35, and the other northeast of structures E6A, E6B, E7, E24, E25, E26 and E27 (Fig. 3).

In the first of the above two groups, structure E2 housed two handmade pottery pots, one of them decorated with bronze studs. Structure E3 was empty and the four remaining structures (E5, E33, E34 and E35) make a stratigraphically related set. This is where the most relevant material was recorded. Structure E35, empty of materials, is a sort of lobby leading to structures E5 and E34. Structure E5 held three ceramic vessels as grave goods: a hand-made bowl engraved with vegetal and animal motifs, a ceramic reel stand with black, red and yellow geometric motifs, and a medium/high carinated plate in the stand. A large reduction firing urn with red vegetal motifs lay next holding the cremated remains of an adult female (Trancho and Robledo, 2016; Lechuga and Soto, 2017). Items for personal adornment were also retrieved from inside the urn: a silver ring, two orange beads, remains of what appears to be a double spring fibula and a bronze falcated knife. Structure E33, separated from structure E5 by a stone, did not show any archaeological material.

The cremated remains of grave E34 are of a subadult of unknown sex for the very few remains available for identification (Trancho and Robledo, 2016; Lechuga and Soto, 2017). The remains were kept in a hand-made, reduction firing polished urn alongside elements for personal adornment, e.g. a ring and an open, bronze bracelet with geometric decoration incised at the ends, and an iron spearhead. The grave goods included a wheelmade plate with red decoration.

The main structure of the second group is structure E26, a grave where a hand-made ceramic urn contained the cremated remains of a subadult female (Trancho and Robledo 2016; Lechuga and Soto, 2017). The grave goods included an unidentified iron item, three bronze rings and seven globular beads of a bluish colour. Six of those beads had the typical form of eye beads. They were in a poor state of preservation and had lost the eye decoration. Structure E25 had an empty, reduction firing urn with red decoration. The remaining structures, i.e. structures E6A, E6B, E7, E24 and E27, did not retain any archaeological materials.

This paper aims at the physico-chemical analysis of non-metallic items retrieved from the abovementioned ritual and funerary áreas. Specifically, the focus is on the decoration of the native and Phoenician type pottery, on the composition of the orange and blue beads, and on the organic remains in the ceramic group, regardless of whether they have decoration or not. The results obtained should help define more accurately the properties and origin of the native and non-native items, and would thus result in a better understanding of how the hybridisation between native tradition and the Phoenician tradition took place. These objectives should also supply additional information about:

- The manufacturing processes used for Orientalizing native pottery and the use of vegetal carbon and/or graphite. The archaeometric analyses carried out this far on this type of pottery in other sites of the same period are not conclusive (Rincón, 1981; García and Morales, 2017; Celestino et al., 2018). As a result, a good part of the research on the subject is based on weak results or on scientifically inadequate visual observation.
- The influence and continuity of the Phoenician decoration model on the later native communities who were identified as Iberians from the 6th century BC.
- Data on the rites performed in the funerary and the ritual areas. The identification of specific chemical markers is expected to cast light on the type of organic offerings or the materials used during the rite, and on whether their origin can be considered of the local tradition or the result of an Eastern Mediterranean influence.

The procedure uses various approaches but is consistent as regards the objectives. It covered three stages:

i) Micro Raman Spectroscopy (MRS) and Energy Dispersive X-Ray Fluorescence (EDXRF) analysis of the mineral and elemental composition of the painted decoration on the ceramic vessels
ii) Gas Chromatography-Mass Spectrometry (GC–MS) analysis of chemical markers in the contents of the vessels
iii) MRS and EDXRF analysis of the mineral and elemental composition of the orange and blue beads.

2. Sampling

This paper researched 28 items, 19 of which were ceramic vessels and nine were beads. In the ceramic group, eleven vessels were reduction firing hand-made items, and the remaining eight were wheelmade, oxidation firing pottery. Four of the hand-made vessels had red, yellow and/or black decoration, whereas six of the wheelmade vessels had black and/or red decoration (Fig. 4). The group comprehends all the ceramic vessels retrieved from the six structures (four funerary and two ritual) that had materials at the time of their archaeological excavation.

The research sample also included a set of beads: two hollow perforated orange beads retrieved from urn 5-1011, which lay in funerary structure E5, and seven bluish globular beads retrieved from funerary urn 7-29 in structure E26 (Table 1) (Fig. 5).

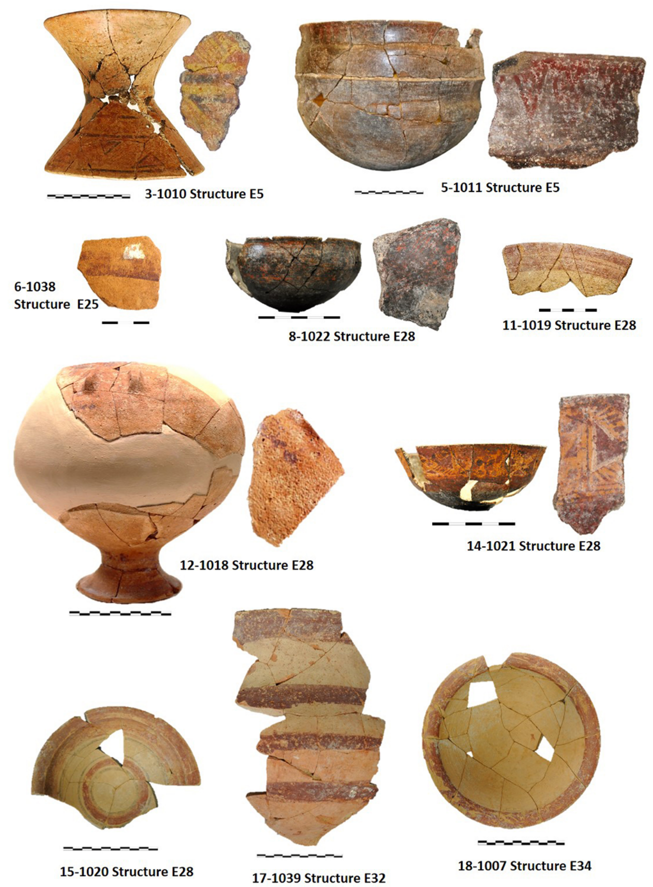

Fig. 4. Decorated vessels under study.

The ten decorated vessels and the orange and blue beads were analysed mineralogically (MRS) and elementally (EDXRF). The contents analysis used GC–MS on the bottom part and/or the body of the 19 vessels, according to the remains available and to their size (26 ceramic samples under study).

3. Method

3.1. Micro Raman Spectroscopy (MRS)

Two Raman spectrometers were used for the analysis of the samples. The portable equipment proved especially useful for large and also for irregularly-shaped items.

The portable equipment used was a BWS445-785S innoRam™ Raman spectrometer (B&WTEK, Inc., Newark, USA) with a 785 nm excitation laser (maximum power of 300 mW) and a 4.5 cm^{-1} spectral resolution. The Raman microprobe can be mounted on a tripod with motorized XYZ axis (MICROBEAM S.A, Barcelona, Spain) or on a microscope sampling stage (B&WTEK, Inc., Newark, USA). The measurements were carried out with a 50x objective. Experimental conditions were: exposure time ranged from 100 to 1000 ms, maximum 60 acquisitions and a spectral range between 60 and 3000 cm^{-1}.

The smaller items were studied with a Renishaw 'in via' Reflex Spectrometer coupled with a confocal Leica DM LM microscope (CICT, University of Jaén), equipped with an argon ion laser (514.5 nm, 25 mW) and a diode laser (785 nm, 300 mW), and a Peltier-cooled CCD detector, calibrated to the 520.5 cm^{-1} line of silicon. The maximum laser output power was eventually reduced using neutral density filters. The spectra were acquired using the 50x objective, in the 100 to 3200 cm^{-1} region, with spectral resolution of *ca.* 2 cm^{-1} (1800 lines/mm grating) and *ca.* 1 cm^{-1} (1200 lines/mm grating). Acquisition time

Table 1
Materials under analysis. NHP: native hand-made pottery, PWP: Phoenician wheelmade pottery, PWNI: Phoenician wheelmade native imitation. NDS: Non-determinate shape.

Vessel/sample	Structure	Type	Context	Decoration-colour	Analysis
1-1040a	E2	NHP, pot	Grave	–	GC–MS
2-1040b	E2	NHP, pot	Grave	–	GC–MS
3-1010	E5	NHP, reel stand	Grave	Red, yellow, black	EDXRF, MRS, GC–MS
4-1036	E5	NHP, storing vessel	Grave	–	GC–MS
5-1011	E5	NHP, urn	Grave	Red	EDXRF, MRS, GC–MS
6-1038	E25	PWP, NDS	Grave	Red	EDXRF, MRS, GC–MS
7-29	E26	NHP, urn	Grave	–	GC–MS
8-1022	E28	NHP, bowl	Ritual area	Red, black	EDXRF, MRS, GC–MS
9-1050	E28	PWNI, bowl/plate	Ritual area	–	GC–MS
10-1029	E28	PWP, storing vessel	Ritual area	–	GC–MS
11-1019	E28	PWP, NDS	Ritual area	Red	EDXRF, MRS, GC–MS
12-1018	E28	PWP, storing vessel	Ritual area	Red	EDXRF, MRS, GC–MS
13-1024	E28	NHP, bowl	Ritual area	–	GC–MS
14-1021	E28	NHP, bowl	Ritual area	Red, yellow	EDXRF, MRS, GC–MS
15-1020	E28	PWP, plate	Ritual area	Red, black	EDXRF, MRS, GC–MS
16-1023	E28	PWNI, bowl/plate	Ritual area	–	GC–MS
17-1039	E32	PWP, storing vessel	Ritual area	Red	EDXRF, MRS, GC–MS
18-1007	E34	PWP, plate	Grave	Red	EDXRF, MRS, GC–MS
19-1034	E34	NHP, urn	Grave	–	EDXRF, MRS, GC–MS
20-1066	E5	Bead	Grave	Orange	EDXRF, MRS
21-1005	E5	Bead	Grave	Orange	EDXRF, MRS
22-1062-1	E26	Bead	Grave	Blue	EDXRF, MRS
23-1062-2	E26	Bead	Grave	Blue	EDXRF, MRS
24-1033-1	E26	Bead	Grave	Blue	EDXRF, MRS
25-1033-2	E26	Bead	Grave	Blue	EDXRF, MRS
26-1033-3	E26	Bead	Grave	Blue	EDXRF, MRS
27-1033-4	E26	Bead	Grave	Blue	EDXRF, MRS
28-1033-5	E26	Bead	Grave	Blue	EDXRF, MRS

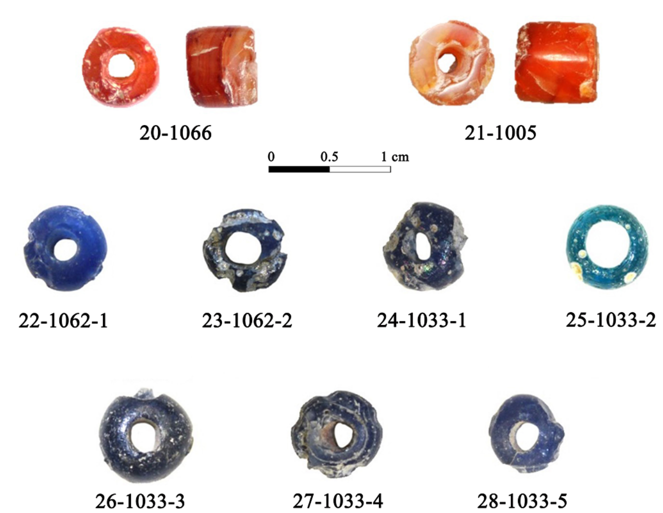

Fig. 5. The orange beads from structure E5 and the bluish beads from structure E26 under study.

was set between 10 and 60 s per accumulation, and the maximum number of accumulations was 15.

3.2. Energy dispersive X-Ray Fluorescence (EDXRF)

An energy dispersive X-ray microfluorescence spectrometer (M4 Tornado, Bruker) was used for this paper (CICT, University of Jaén). This spectrometer is equipped with a microfocus X-ray tube with a Rh anode, a polycapillary lens for X-ray focussing, and a 30-mm^2 energy dispersive detector (SDD), calibrated to Kα_1 line of Zr standard (15.775 keV). The sample chamber incorporates an XYZ motorized stage for sample positioning. A high resolution microscope (10x, 100x) is used to position the sample on the desired distance from the polycapillary. To increase the sensitivity of the low Z elements, the sample chamber can be brought under vacuum. For the analysis of the samples, a spot size of 25 µm was chosen at an operating X-ray tube voltage of 50 kV and intensity of 600 µA. The ESPRIT software quantification procedure used relies on a modified version of Sherman equation with correction of matrix effects for higher accuracy.

Three measurements were carried out for each coloured area, ceramic paste and also for the beads. In the former, the areas where colours are best preserved were used as measurement areas. High resolution microcospy coupled to the EDXRF equipment allowed further delineation of the areas so as to avoid sectors with discontinuous or fragmented decoration. This focus on areas with the thickest decoration allowed to prevent or significantly to reduce X-ray penetration into the underlying ceramic paste. Potential interference was prevented by measuring the ceramic paste of each vessel and comparing the results with the decorated areas, especially as regards Mn and Fe concentrations. X-ray penetration was also assessed by recording multi-element mappings in specific areas with varying decoration thicknesses to test the influence of the ceramic paste composition on the decoration results.

3.3. Gas chromatography-mass spectrometry (GC–MS)

3.3.1. Archaeological samples

Approximately 4 g of the bottom or of the body of the vessel were sampled. The fragments of archaeological vessels selected for analysis were wrapped each in a piece of dark paper and stored in a freezer at least at −20 °C until their analysis. The fragments were then taken out of the freezer and a sample was collected. Any remains of soil were removed with an electric hand-drill. The sample was then ground to the appropriate size in an agatha mortar. 2 g were taken for the analysis.

3.3.2. Lipid extraction

Extraction is in accordance with the procedure described in Evershed et al. (1990). Ten µL of tetratriacontane (internal standard) and 10 mL of the mixture chloroform/methanol ($CHCl_3$:MeOH) (2:1 v/v) were added to 2 g of the ground ceramic fragment. Lipids were extracted with ultrasound for 15 min. The solution was centrifuged (3500 rpm, 5 min) eliminating the remaining ceramic and removing the supernatant where the lipids are solved. This process was repeated twice. The extract portions were combined into one for solvent evaporation under N_2 stream. The dry extract was solved again in 500 µL of $CHCl_3$. The extracts were stored in a deep freeze (-20 °C) until required for GC–MS.

3.3.3. Derivatization

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

3.3.4. GC–MS analysis

The analyses were performed using a gas chromatography equipment (model Thermo TraceGC Ultra) coupled to a Thermo DC Q II mass spectrometer. The samples were introduced by on-column injection into a 15 m × 0.25 mm I.D. fused silica capillary column, coated with poly (dimethylsiloxane) stationary phase with 0.1 µm film thickness. Helium was used as the carrier gas (purity 99.99%) at a flow speed of 1.2 mL/min. The GC oven temperature program was as follows: initially at 50 °C, held for 2 min, ramp to 350 °C at 10 °C/min, held for 10 min.

The mass spectrometer conditions were an emission current of 100 µA, an electron energy of 70 eV and an ionization source temperature of 300 °C. Total ions measurements were obtained in the mass spectrometer that scanned from m/z 50–900 at a scan rate of 1.15 scan/s. The GC–MS capillary interface was maintained at a temperature of 350 °C.

4. Results

4.1. Ceramic vessels: decoration

The data obtained by MRS and EDXRF analysis from the decorations show a contrast between native ceramic materials and materials of Phoenician origin or influence. Although red pigment is obtained in both groups from hematite (α-Fe_2O_3) in various concentrations, the contrast lies in the raw materials used for yellow and black, and in the firing used (Table 2).

In native pottery, yellow is obtained from goethite [FeO(OH)] (vessels 3-1010 and 14-1021), as shown by the Raman bands (90, 164, 204, 243, 296, 389, 547 cm^{-1} in vessel 3-1010) and high Fe_2O_3 concentration (between 26,07 and 37,22 wt%) (Fig. 6). As regards black, MRS analysis recorded amorphous vegetal carbon and graphite. Amorphous carbon is used for colouring the surface of the vessel (vessel 8-1022, Raman bands at 1370, 1609 cm^{-1}), whereas graphite is used for stripes or lines (vessel 3-1010, Raman bands at 1355, 1587, 1624, 2459, 2708, 2954 cm^{-1}) (Coccato et al., 2015; Bokobza et al., 2015) (Fig. 7).

The use of goethite and amorphous carbon also shows that decoration followed firing, because both damage colours: Goethite turns into hematite above 250–300 °C and turns red as goethite becomes dehydrated (De Faria and Lopes, 2007; Romero et al., 2013). Amorphous carbon alters from 200 °C. On average, organic carbon is not completely eliminated until temperatures of about 500 °C are reached (Garrison, 2016).

Wheelmade pottery of the Phoenician type no longer uses yellow, and it uses manganese oxide instead of amorphous carbon for black. This innovation allowed vessel decoration with black and red before oxidation firing without the risk of colour changes at high temperatures. In the vessel studied (vessel 15-1020), the Raman spectrum records this type of oxide, even if it does not identify whether it is bixbyite (α-Mn_2O_3) or hausmannite (Mn_3O_4) (Fig. 8).

MRS identification of the various types of manganese oxides is not without problems (Angeli et al., 2019; Centeno et al., 2012; Julien et al., 2004, Sepúlveda et al., 2015). Manganese oxides and oxyhydroxides show high thermal opacity and absorption. This gives rise to amorphous materials or new mineral phases under medium-high laser intensity. An increase in the temperature of the crystal network generally modifies the displacement of the Raman bands as the spinel structure of hausmannite (Mn_3O_4) is formed. Another difficulty for the identification of manganese compounds is the possibility of the manganese ion to coexist in varying degrees of oxidation. This gives rise to totally different oxides and oxyhydroxides. Finally, some types may be converted into others as a result of geologically-, anthropogenically- or climatically-induced reactions (Ospitali et al., 2006; De Benedetto et al., 2011; Tuñón et al., 2016).

The most common form of manganese mineral in nature is pyrolusite (β-MnO2). During firing, pyrolusite first converts into bixbyite (above 450 °C) and then combines with iron oxide to form jacobsite (above 950 °C) (Schweizer and Rinuy, 1982). The identification of bixbyite or hausmannite in vessel 15-1020 signals mineral phases prior to the formation of jacobsite, because the necessary temperature has not been reached. Laser-induced modifications can be discarded considering the low power applied during the study of black decorations (3mW).

Table 2
MRS results. NHP: Native hand-made pottery, PWP: Phoenician wheel-made pottery. References: 1) http://rruff.info/hematite; 2) De Faria and Lopes, 2007; 3) http://rruff.info/goethite; 4) http://rruff.info/graphite; 5) Bokobza et al., 2015; 6) Parras et al., 2010; 7) Coccato et al., 2015; 8) Julien et al., 2004; 9) Tuñón et al., 2016; 10) Angeli et al., 2019.

Vessel/Type	Structure	Colour	Wavenumber/cm^{-1}	Identification	Ref.
3-1010 NHP	E5	Red	223, 291, 408, 498, 608	Hematite	1, 2
		YellowBlack	90, 164, 204, 263, 296, 389, 547	Goethite	2, 3
			1355, 1587, 1624, 2459, 2708, 2954	Graphite	4, 5
5-1011 NHP	E5	Red	222, 288, 406	Hematite	1, 2
6-1038 PWP	E25	Red	223, 242, 290, 408, 496, 610	Hematite	1, 2
8-1022 NHP	E28	Red	223, 294, 411, 493, 611	Hematite	1, 2
		Black	1370, 1609	Amorphous carbon	6, 7
11-1019 PWP	E28	Red	223, 290, 407, 607	Hematite	1, 2
12-1018 PWP	E28	Red	222, 241, 288, 404, 498, 604	Hematite	1, 2
14-1021 NHP	E28	Red	223, 292, 407, 606	Hematite	1, 2
		Yellow	145, 201, 294, 396, 508	Goethite	2, 3
15-1020 PWP	E28	Red	142, 223, 243, 290, 407, 499, 607	Hematite	1, 2
		Black	320, 480, 605, 700, 829	Bixbyite/Hausmannite	8, 9, 10
17-1039 PWP	E32	Red	224, 290, 480, 496, 608, 1306	Hematite	1, 2
18-1007 PWP	E34	Red	223, 243, 289, 407, 499, 607	Hematite	1, 2

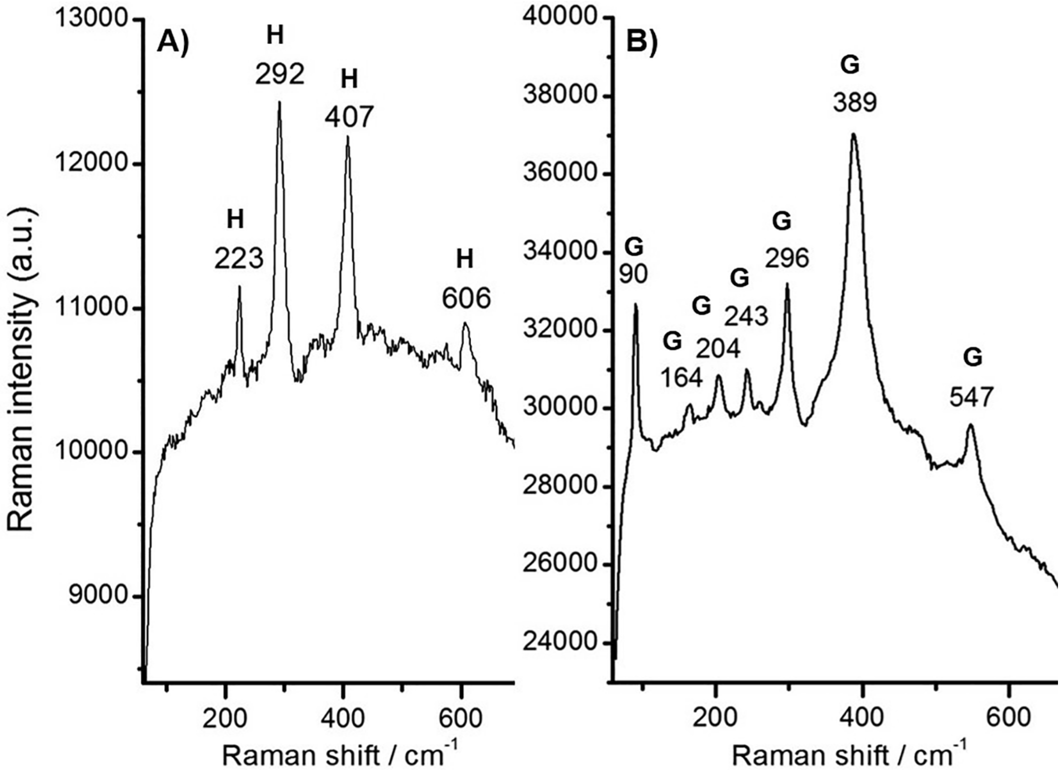

Fig. 6. A: Raman spectrum of the red decoration of vessel 14-1021 (H: hematite). Experimental conditions of portable Raman spectrometer: 50x objective, 30 acquisitions of 300 ms, 785 nm diode laser, 45 mW. B: Raman spectrum of the yellow decoration of vessel 3-1010 (G: goethite). Experimental conditions of portable Raman spectrometer: 50x objective, 60 acquisitions of 500 ms, 785 nm diode laser, 45 mW.

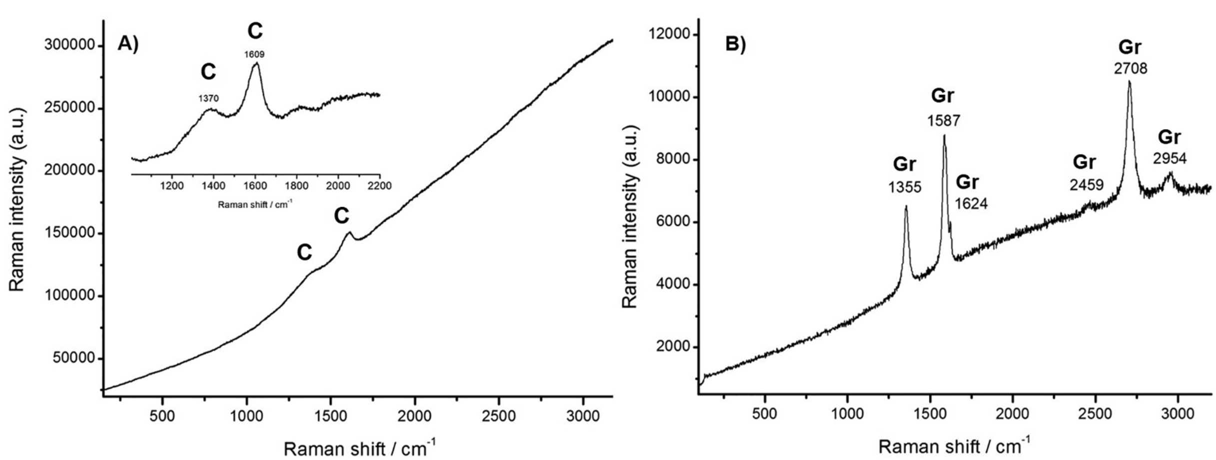

Fig. 7. A: Raman spectrum of amorphous carbon (C) in vessel 8-1022 with detail of baseline correction. Experimental conditions of Renishaw 'in via' Reflex Spectrometer: 50x objective, 10 accumulations of 10 s, 514.5 nm argon ion laser, 2.5 mW. B: Raman spectrum of graphite (Gr) in vessel 3-1010. Experimental conditions of Renishaw 'in via' Reflex Spectrometer: 50x objective, 10 accumulations of 10 s, 514.5 nm argon ion laser, 2.5 mW.

Elemental analysis of the samples with black decoration confirms the various ways in which black can be obtained. Only the Phoenician vessel (15-1020) did show high concentrations of MnO (2,12 wt%). In the rest of the vessels, 3-1010 with a black decoration band and 8-1022 with black background, the concentration of MnO does not exceed 0,13 wt% (Table 3).

4.2. Ceramic vessels: contents

GC–MS analysis identified chemical markers of contents in 10 out of 19 vessels. This high success rate (52.6%) reveals excellent preservation of the organic remains. The substance in question was beeswax in nine vessels. In the remaining vessel, the chemical markers point to vegetable fat. The vessels with chemical markers belong to a ritual structure (E28) and to four funerary structures (E5, E25, E26, E34) (Table 4).

The typical chemical markers of beeswax identified in nine vessels match totally or partially the range of markers that are typical of this substance (Regert et al., 2001; Colombini and Modugno, 2009; Pollard et al., 2017; Roffet-Salque et al., 2015) (Fig. 9, Table 4):

- Series of C23 to C33 n-alkanes displaying a unimodal distribution with a strong odd-over-even predominance
- Series of W40-W54 carbon number palmitic acid wax esters with a main constituent that contains 46 carbon atoms.
- Long-chain alcohols, resulting from hydrolysis of wax esters, with an

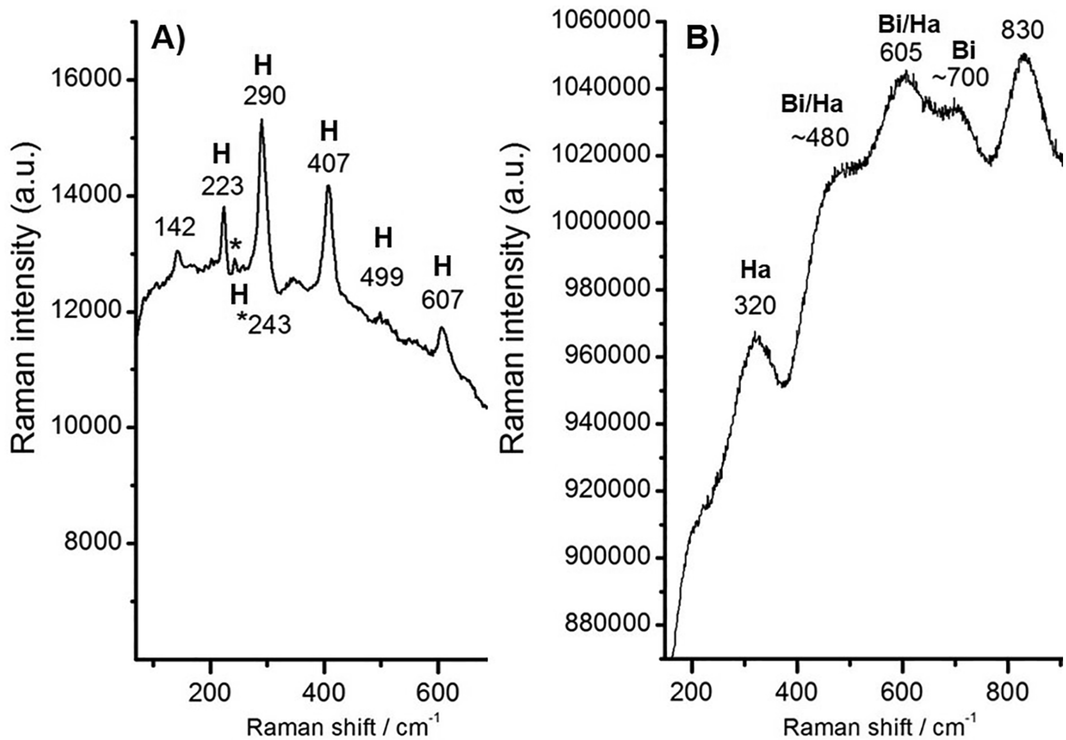

Fig. 8. Vessel 15-1020. A: Red decoration. Raman spectrum of hematite (H). Experimental conditions of portable Raman spectrometer: 50x objective, 40 acquisitions of 300 ms, 785 nm diode laser, 45 mW. B: Black decoration. Raman spectrum of bixbyite(Bi)/hausmannite(Ha). Experimental conditions of Renishaw 'in via' Reflex Spectrometer: 50x objective, 10 accumulations of 10 s, 785 nm diode laser, 15 mW.

even carbon number usually from C24(OH) to C34(OH), maximizing at C30(OH).

- Series of W(OH)40–W(OH)50 carbon number hydroxymonoesters.

Beeswax is relatively resistant to degradation for its hydrophobic nature. Hence, if protected from extensive microbial attack and/or exposure to high temperatures during anthropogenic manipulation, the aforementioned chemical characteristics can be used for recording its presence (Roffet-Salque et al., 2015).

The remaining vessel, urn 6-1038 of structure E25, shows a radically different lipidic profile and one that clearly points to a vegetable fat: oleic (C18:1) and linoleic unsaturated fatty acids (C18:2), and the vegetable sterols stigmasterol and sitosterol. The high concentration of lauric acid and of linoleic acid may signal that the vegetable fat inside the vessel had a closer fat profile to that of nuts than to that of wild olives (Pollard and Heron, 2008) (Fig. 10, Table 5).

4.3. Beads

The two orange beads are made of carnelian. Carnelian belongs to the general group of chalcedony gems which are characterised by the presence of micro- or crypto-crystalline varieties of silica. The main component of chalcedony is α-quartz; it also contains a minor amount of moganite and a lot of opacifying and colouring impurities (Economou et al., 2010; Lauwers et al., 2016). Moganite is the monoclinic silica polymorph identified by its main band at 501 cm^{-1} (Pop et al., 2004; Bersani and Lottici, 2016). The interpretation of the Raman spectra allows to confirm unambiguously the presence of both quartz and moganite (Table 6, Fig. 11).

EDXRF elemental analysis of the orange beads reveals approximately 99 wt% of SiO_2 and a low concentration of Fe_2O_3, specifically below 0,11 wt% in both beads (Table 7). This difference in composition separates this element from jasper, as the latter variety of chalcedony has high concentrations of Fe. The use of organic dyes to enhance red colouration can be discarded due to the absence of their characteristic Raman bands. Furthermore, the low Fe concentration reveals no thermal modifications in the structure of carnelian, which may indicate that the beads were deposited in the funerary urn after the cremation of the body (Lauwers et al., 2016).

In the case of the group of seven blue beads recovered from the structure 26, the obtaining of Raman spectra of good quality was prevented by the strong fluorescence backgrounds caused by the beads matrices. EDXRF analysis reveals the chemical composition of a soda lime silicate glass (the SiO_2 concentration ranging between 72,96 and 82,99 wt%), even if clearly altered by postdepositional factors as a result of which the beads became dealkalized. They also show a low percentage of Na_2O, K_2O and MgO, as well as relatively enriched stable oxides, e.g. SiO_2, Al_2O_3 and CaO. The cobalt recorded in all the samples (*ca.* 0,1 wt%) is enough to suggest that the blue colour was obtained deliberately by addition of Co(II) to the glass matrix of the beads, by use of some kind of mineral salt rich in cobalt or materials rich in cobalt (García-Heras et al., 2003; Smirniou and Rehren, 2013; Oikonomou et al., 2018). Sample 23-1062-2 stands out for the lowest SiO_2 percentage (72,96 wt%) and for the highest concentrations of CaO (14,08 wt%) and of CuO (0,35 wt%). This combination suggests addition of Egyptian blue as colouring agent. No evidence of the use of calcium antimoniate as opacifier in the beads was recorded (Table 7).

5. Discussion

The archaeological interpretation of the data suggests an interpretation of the funerary rite, a construal of the interaction between native and Phoenician decoration, and the analysis of the cultural referents of the Mediterranean and of the Near East.

5.1. Ceramic decoration

Regarding decoration, the site *Cerro de los Vientos* represents the confluence of the native and the Phoenician traditions. A large number of the pottery researched is according to the native tradition of hand-

Table 3
EDXRF results of decorated ceramic vessels. Results shown (wt%) are the average of three analyses per colour. P: ceramic paste; R: red colour; B: Black colour; Y: yellow colour; W: white colour; Struct.: structure; s.d.: standard deviation; bdl: below detection limit.

Vessels	Struct	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	V_2O_5	MnO	Fe_2O_3	CoO	CuO	ZnO	SrO	Total
3-1010 P	E5	0,43	2,06	19,43	61,85	0,36	1,46	1,03	2,80	2,04	1,18	0,10	0,03	6,93	0,03	0,02	0,03	0,04	99,77
(s.d.)		(0,13)	(0,46)	(1,70)	(1,73)	(0,04)	(0,52)	(0,18)	(0,15)	(0,12)	(0,28)	(0,08)	(0,01)	(1,29)	(0,02)	(0,01)	(0,02)	(0,00)	
3-1010 R	E5	0,93	1,21	19,19	52,15	0,79	0,42	1,85	3,27	2,27	1,18	0,09	0,29	16,10	0,07	0,04	0,04	0,07	99,90
(s.d.)		(0,01)	(0,51)	(0,49)	(2,93)	(0,13)	(0,11)	(0,23)	(0,09)	(0,23)	(0,01)	(0,01)	(0,08)	(3,16)	(0,01)	(0,01)	(0,01)	(0,01)	
3-1010 Y	E5	2,28	0,58	15,25	35,81	bdl	0,14	3,61	2,31	1,17	1,02	0,08	0,25	37,22	bdl	0,04	0,04	0,10	99,87
(s.d.)		(0,70)	(0,82)	(1,76)	(1,51)	-	(0,03)	(0,41)	(0,13)	(0,03)	(0,25)	(0,01)	(0,05)	(2,85)	-	(0,01)	(0,00)	(0,00)	
3-1010 B	E5	0,34	0,87	28,49	52,86	0,38	0,24	0,94	3,92	0,93	0,98	0,09	0,13	9,62	0,06	0,02	0,01	0,05	99,90
(s.d.)		(0,48)	(0,18)	(1,00)	(2,50)	(0,16)	(0,04)	(0,04)	(0,18)	(0,03)	(0,04)	(0,02)	(0,05)	(2,7)	(0,01)	(0,00)	(0,00)	(0,01)	
5-1011 P	E5	0,48	2,15	21,52	61,41	0,41	0,07	0,92	2,65	2,01	0,98	0,05	0,06	7,11	0,02	0,02	0,02	0,04	99,88
(s.d.)		(0,19)	(0,59)	(0,16)	(2,35)	(0,11)	(0,04)	(0,03)	(0,06)	(0,08)	(0,00)	(0,01)	(0,04)	(1,54)	(0,01)	(0,01)	(0,01)	(0,00)	
5-1011 R	E5	bdl	bdl	16,35	57,96	bdl	0,47	1,99	3,18	2,48	1,30	0,08	0,12	15,68	bdl	0,04	0,03	0,06	99,76
(s.d.)		-	-	(1,52)	(4,29)	-	(0,28)	(0,65)	(0,08)	(0,23)	(0,11)	(0,02)	(0,02)	(4,27)	-	(0,01)	(0,01)	(0,01)	
6-1038 P	E25	bdl	2,10	15,83	61,17	bdl	0,50	0,24	5,72	2,10	1,33	0,05	0,13	10,57	0,04	0,01	0,03	0,11	99,93
(s.d.)		-	(0,01)	(0,83)	(0,4)	-	(0,47)	(0,03)	(0,37)	(0,29)	(0,19)	(0,01)	(0,03)	(0,41)	(0,00)	(0,01)	(0,01)	(0,02)	
6-1038 R	E25	bdl	4,75	11,87	38,87	0,15	0,54	0,62	4,14	1,97	1,24	0,05	0,38	34,73	0,13	0,04	0,05	0,22	99,77
(s.d.)		-	(0,61)	(1,33)	(5,79)	(0,07)	(0,36)	(0,06)	(0,35)	(0,25)	(0,06)	(0,02)	(0,21)	(7,56)	(0,03)	(0,01)	(0,01)	(0,05)	
8-1022 B	E28	0,45	1,85	21,34	61,29	0,6	0,04	0,96	2,48	1,58	0,84	0,06	0,09	8,18	0,04	0,02	0,01	0,04	99,88
(s.d.)		(0,31)	(0,17)	(0,49)	(1,29)	(0,05)	(0,03)	(0,06)	(0,2)	(0,16)	(0,07)	(0,01)	(0,02)	(0,37)	(0,01)	(0,00)	(0,01)	(0,01)	
8-1022 P	E28	0,40	2,23	23,67	59,82	0,52	0,05	0,90	2,45	1,26	0,76	0,06	0,01	7,13	0,05	0,03	0,01	0,04	99,38
(s.d.)		(0,35)	(0,14)	(0,33)	(0,41)	(0,04)	(0,06)	(0,07)	(0,04)	(0,01)	(0,01)	(0,01)	(0,01)	(0,68)	(0,01)	(0,00)	(0,00)	(0,00)	
8-1022 R	E28	0,40	1,22	18,27	59,80	0,71	0,08	1,27	3,01	0,93	0,83	0,06	0,09	13,07	0,05	0,03	0,01	0,05	99,87
(s.d.)		(0,40)	(0,13)	(0,85)	(0,93)	(0,12)	(0,00)	(0,11)	(0,20)	(0,29)	(0,02)	(0,02)	(0,04)	(0,82)	(0,03)	(0,01)	(0,00)	(0,01)	
11-1019 P	E28	bdl	2,65	17,87	63,26	bdl	0,14	0,19	4,28	1,41	1,20	0,03	0,09	8,60	0,04	0,01	0,02	0,07	99,86
(s.d.)		-	(0,47)	(1,24)	(2,19)	-	(0,08)	(0,01)	(0,30)	(0,22)	(0,13)	(0,01)	(0,07)	(0,84)	(0,00)	(0,00)	(0,00)	(0,01)	
11-1019 R in	E28	bdl	4,63	13,69	47,44	0,02	0,25	0,39	3,62	1,38	1,13	0,03	0,05	26,87	0,09	0,07	0,04	0,11	99,82
(s.d.)		-	(0,3)	(1,84)	(6,27)	(0,01)	(0,20)	(0,10)	(0,63)	(0,12)	(0,18)	(0,01)	(0,01)	(8,54)	(0,03)	(0,03)	(0,03)	(0,04)	
11-1019 R out	E28	bdl	6,17	13,92	55,86	bdl	0,19	0,38	4,89	2,25	1,12	0,01	0,19	14,56	0,06	0,02	0,04	0,14	99,80
(s.d.)		-	(1,67)	(0,51)	(3,07)	-	(0,05)	(0,01)	(0,65)	(0,15)	(0,12)	(0,00)	(0,05)	(2,18)	(0,01)	(0,01)	(0,02)	(0,01)	
12-1018 P	E28	bdl	2,39	13,93	65,98	bdl	0,18	0,22	5,00	2,13	1,26	bdl	0,07	8,57	0,04	0,01	0,02	0,11	99,90
(s.d.)		-	(0,40)	(0,95)	(1,23)	-	(0,11)	(0,02)	(0,54)	(0,26)	(0,16)	-	(0,03)	(1,19)	(0,01)	(0,00)	(0,00)	(0,04)	
12-1018 R	E28	bdl	4,45	8,53	33,03	0,15	0,34	0,69	2,68	3,34	1,01	bdl	0,07	45,03	0,13	0,02	0,04	0,27	99,79
(s.d.)		-	(0,60)	(0,94)	(6,10)	(0,06)	(0,17)	(0,11)	(0,98)	(1,59)	(0,48)	-	(0,03)	(10,07)	(0,03)	(0,01)	(0,02)	(0,06)	
14-1021 P	E28	bdl	2,28	19,52	66,07	0,43	0,01	0,89	3,07	1,23	0,83	0,09	0,03	5,36	0,02	0,03	0,02	0,03	99,90
(s.d.)		-	(0,21)	(0,21)	(0,11)	(0,07)	(0,00)	(0,08)	(0,28)	(0,04)	(0,06)	(0,06)	(0,01)	(0,27)	(0,00)	(0,00)	(0,01)	(0,00)	
14-1021 R	E28	bdl	0,01	10,82	53,7	0,03	0,29	1,84	1,74	1,92	0,77	0,08	0,22	28,01	bdl	0,13	0,03	0,08	99,63
(s.d.)		-	(0,01)	(3,37)	(4,71)	(0,04)	(0,02)	(0,23)	(0,92)	(0,12)	(0,14)	(0,01)	(0,01)	(8,97)	-	(0,04)	(0,01)	(0,02)	
14-1021 Y	E28	bdl	bdl	6,56	61,62	bdl	bdl	2,26	1,67	0,59	0,56	0,06	0,10	26,07	bdl	0,09	0,03	0,04	99,60
(s.d.)		-	-	(0,35)	(5,06)	-	-	(0,08)	(0,04)	(0,09)	(0,03)	(0,01)	(0,05)	(4,46)	-	(0,01)	(0,01)	(0,01)	
15-1020 P	E28	0,35	2,46	15,07	70,45	0,58	0,05	0,97	2,70	1,41	0,70	0,06	0,08	4,95	0,02	0,02	0,02	0,07	99,92
(s.d.)		(0,01)	(0,16)	(0,57)	(0,45)	(0,02)	(0,03)	(0,05)	(0,03)	(0,08)	(0,07)	(0,01)	(0,01)	(0,29)	(0,02)	(0,00)	(0,01)	(0,01)	
15-1020 R	E28	bdl	0,69	9,74	65,51	bdl	0,01	1,32	2,62	1,50	0,99	0,07	0,09	17,11	bdl	0,10	0,02	0,08	99,82
(s.d.)		-	(0,55)	(1,55)	(2,04)	-	(0,01)	(0,01)	(0,18)	(0,15)	(0,01)	(0,02)	(0,01)	(3,36)	-	(0,03)	(0,00)	(0,00)	
15-1020 B	E28	0,06	2,65	14,00	63,94	0,60	0,18	1,42	2,35	1,90	1,12	0,07	2,12	9,40	0,01	0,02	0,03	0,09	99,92
(s.d.)		(0,08)	(0,32)	(0,06)	(2,63)	(0,16)	(0,14)	(0,11)	(0,07)	(0,42)	(0,39)	(0,01)	(0,97)	(1,93)	(0,01)	(0,00)	(0,01)	(0,01)	
17-1039 P	E32	bdl	2,66	15,02	62,57	0,55	0,14	0,67	2,90	8,71	0,64	0,05	0,04	5,75	0,02	0,02	0,01	0,06	99,77
(s.d.)		-	(0,08)	(1,03)	(2,31)	(0,04)	(0,00)	(0,02)	(0,18)	(0,52)	(0,04)	(0,01)	(0,01)	(0,47)	(0,01)	(0,00)	(0,00)	(0,01)	
17-1039 R	E32	bdl	1,69	5,15	21,61	0,13	0,20	1,89	0,02	3,05	0,83	0,21	0,24	63,70	0,01	0,08	0,04	bdl	98,82
(s.d.)		-	(0,42)	(0,12)	(1,1)	(0,11)	(0,02)	(0,00)	(0,01)	(0,57)	(0,08)	(0,01)	(0,01)	(0,20)	(0,01)	(0,01)	(0,00)	-	
17-1039 W	E32	0,39	2,88	14,75	62,87	0,56	0,26	0,66	2,45	9,58	0,59	0,06	0,03	4,69	0,01	0,02	0,02	0,06	99,89
(s.d.)		(0,28)	(0,15)	(0,49)	(2,45)	(0,14)	(0,04)	(0,01)	(0,35)	(1,16)	(0,05)	(0,03)	(0,01)	(0,92)	(0,01)	(0,01)	(0,01)	(0,01)	
18-1007 P	E34	0,11	2,13	16,21	68,33	0,41	0,10	0,99	2,58	1,27	0,91	0,07	0,14	6,58	0,02	0,03	0,02	0,06	99,93
(s.d.)		(0,16)	(0,15)	(0,27)	(0,35)	(0,00)	(0,01)	(0,01)	(0,04)	(0,17)	(0,04)	(0,01)	(0,13)	(0,52)	(0,02)	(0,00)	(0,01)	(0,00)	
18-1007 R	E34	0,44	3,07	13,01	58,50	0,33	0,14	1,52	2,91	1,52	1,22	0,11	0,10	16,72	0,02	0,07	0,03	0,11	99,81
(s.d.)		(0,38)	(1,23)	(2,83)	(2,70)	(0,29)	(0,06)	(0,31)	(0,39)	(0,17)	(0,18)	(0,03)	(0,04)	(6,79)	(0,02)	(0,03)	(0,01)	(0,03)	

made, reduction firing, with post-firing decoration by use of hematites, goethite, amorphous carbon and graphite (Table 2, Fig. 4). This type of ceramics, with its typical shapes (carinated bowls, cups, bell-mouthed vessels and reel stands) spread mainly over the centre and the southern half of the Iberian Peninsula (Andalusia, Castilla-La Mancha, Extremadura) in the 8th and 7th centuries BC (Pellicer, 1987–1988; García and Morales, 2017; Celestino et al., 2018).

The analysis of black decoration in native pottery casts light on some technical issues. The identification of amorphous carbon in the decoration of vessel 8-1022 questions the presumed exclusive use of graphite in this type of vessels during the Orientalizing period (Table 2). This result also confirms those obtained from similar pottery of the same chronological phase in the sites *Puente del Obispo* and *Puente Tablas* (Parras et al., 2010). Even so, use of graphite for decoration was common for the community buried in *Cerro de los Vientos*, as evidenced by its use in the decoration of vessel 3-1010 with black bands (Table 2, Fig. 4).

The same context shows oxidation firing pottery where hematite-red decoration is combined with manganese-black (Tables 2 and 3, Fig. 4). This group of ceramics is associated with the Phoenician presence from the 9th century BC. The main Phoenician colonization centres spread over the southern half of the Iberian Peninsula, from present-day province of Alicante to the mouth of river Tajo in Portugal (Aubet, 2006). Research on provenance in several sites allowed to identify western Phoenician production, i.e. the Phoenician potters of the colonies and settlements of the southern half of the Iberian Peninsula made specific shapes of amphorae, jars, dishes, pithoi and pots with local raw materials (Delgado, 2011). These ceramic types later spread over other areas, like *Cerro de los Vientos*.

Table 4
GC–MS. Chemical markers and identification. nC: n-alkanes; C(OH): long-chain alcohols; W: wax ester, W(OH): wax hydroxymonoester.

Vessel	Type	Structure	Chemical markers	Identification
3-1010 13-1024	Reel stand Bowl	E5 E28	C16:0; C18:0; nC23; nC25; nC27; nC29; nC31; nC33; C24(OH); C26(OH); C28(OH); C30(OH); C32(OH); W40; W42; W44; W46; W48; W(OH)40; W(OH)42; W(OH)44; W(OH)46; W(OH)48	Beeswax Palmitic acid, stearic acid, alcanes, alcohols, wax esters, hydroxymonosters
4-1036 7-29	Plate Urn	E5 E26	nC23; nC25; nC27; nC29; nC31; nC33; C24(OH); C26(OH); C28(OH); C30(OH); C32(OH); C34(OH); W40; W42; W44; W46; W48; W50	Beeswax Alkanes, alcohols, wax esters
8-1022	Bowl	E28	nC25; nC27; nC29; nC31; nC33; MonoP; MonoS; C24(OH); C26(OH); C28(OH); C30(OH); C32(OH); W40; W42; W44; W46; W48; W50	Beeswax Alkanes, alcohols, wax esters
5-1011 10-1029 12-1018 19-1034	Urn Storing v. Storing v. Urn	E5 E28 E28 E34	nC23; nC25; nC27; nC29; nC31; nC33; C24(OH); C26(OH); C28(OH); C30(OH); C32(OH); C34(OH); W40; W42; W44; W46; W48; W50; W(OH)40; W(OH)42; W(OH)44; W(OH)46; W(OH)48	Beeswax Alkanes, alcohols, wax esters, hydroxymonosters
6-1038	Urn	E25	C12:0; C14:0; C16:0; C18:2; C18:1; C18:0; C20:0; MonoP; MonoS; stigmasterol; sitosterol	Vegetable Fat

Even so, the Phoenician potters retained other conspicuously eastern elements, especially as regards production and decoration (Delgado, 2011; Vieira et al., 2018). Concerning black and red decoration, the closest reference pottery is Phoenician bichrome pottery of the 10th to the 8th century BC, decorated with both bands and concentric circles of alternating black and red pigments. Chemical analysis has shown that the red pigment derives from iron oxides and the black pigment from manganese oxides. Vessels made in this ware were mostly small containers such as flasks, jugs, bowls, kraters, and jars, the latter being, however, rare (Aloupi et al., 2000; Shoval and Gilboa, 2016; Shoval, 2017).

Use of manganese mineral in oxidation firing for black decoration became widespread to the extent that it was also adopted by the later native communities known as Culture of the Iberians (Tuñón et al., 2016). Oxidation firing bowls, even without manganese, started to be imitated at *Cerro de los Vientos*. The widespread use of this type of decoration is, however, not recorded in southern Iberian settlements until the 6th and 5th centuries BC. Pottery vessels with manganese oxide black and red decorations were recorded from the cemeteries *La Noria* (Fuente de Piedra, Málaga) and *Tútugi* (Galera, Granada) and the *oppidum* of *Puente Tablas* (Jaén). Manganese oxide may appear as bixbyite/hausmannite or as jacobsite according to firing temperatures, i.e. above or under 900 °C respectively (Tuñón et al., 2016).

The use of manganese oxide was a turning point as a result of which amorphous vegetal carbon remained in use only for decoration of the plaster walls inside tombs, as in the cemetery of *Tútugi* (Galera, Granada) (Parras-Guijarro et al., 2006; Sánchez et al., 2012; Tuñón et al., 2016). Black decoration based on manganese oxide remained in use in Iberian vessels until late in the 5th century BC. From the 4th century BC

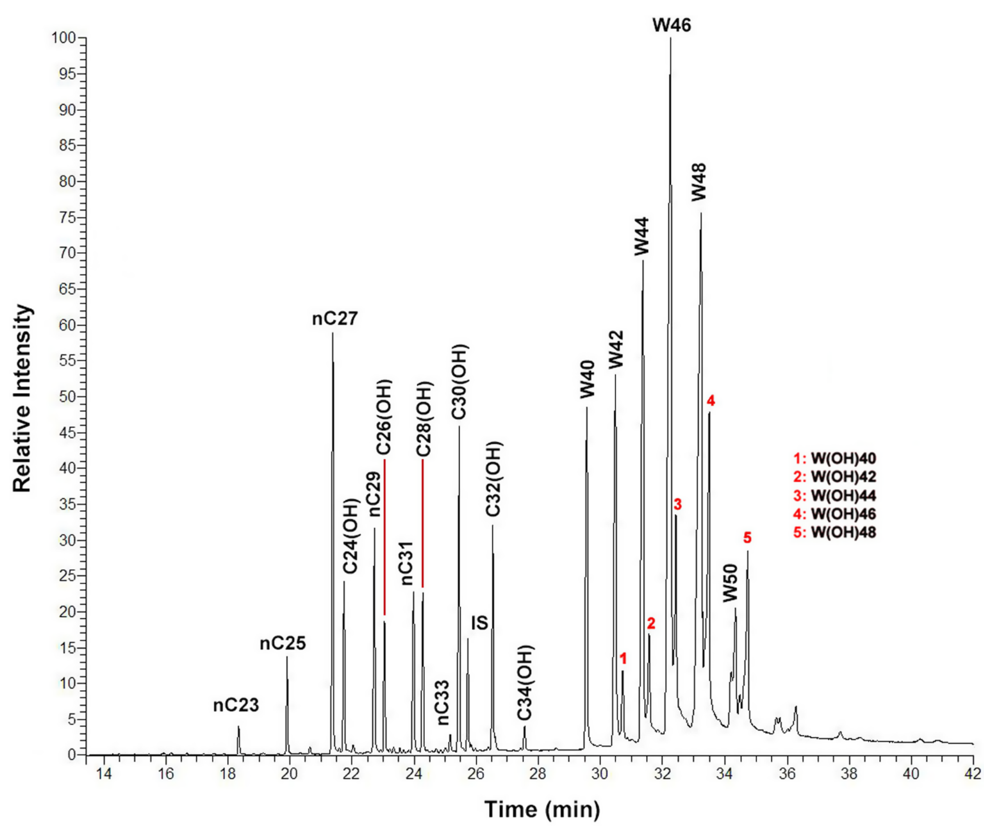

Fig. 9. GC–MS of urn 5-1011. Beeswax.

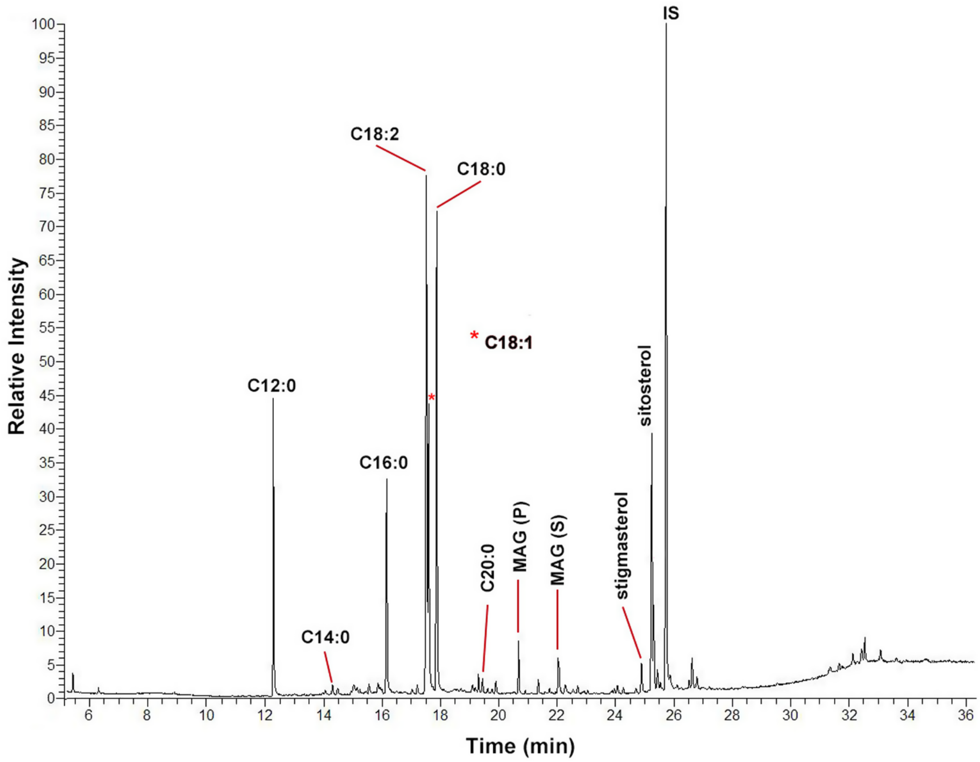

Fig. 10. GC–MS of urn 6-1038. Vegetable fat.

Table 5
Main chemical markers of vessel 6-1038, structure E25.

Chemical markers (TMSD)	*m/z* peaks
C12:0 (Lauric acid)	272 (M+), 257 (M−15)+, 73 (silyl group)
C18:1 (Oleic acid)	354 (M+), 339 (M−15)+, 73 (silyl group)
C18:2 (Linoleic acid)	352 (M+), 337 (M−15)+, 73 (silyl group)
Stigmasterol	486 (M+), 471 (M−15)+, 396, 361, 357, 73 (silyl group)
Sitosterol	484 (M+), 469 (M−15)+, 394, 379, 355, 73 (silyl group)

Table 6
Results of the MRS analysis of the beads. References: 1) Economou et al., 2010; 2) Lauwers et al., 2016; 3) http://rruff.info/quartz.

Sample	Structure	Colour	Wavenumber/cm^{-1}	Identification	Ref.
20-1066	E5	Orange	125, 204, 261, 352, 397, 463, 501, 802	Carnelian	1, 2, 3
21-1005	E5	Orange	129, 208, 262, 354, 399, 463, 501, 692, 803, 1081, 1159	Carnelian	1, 2, 3

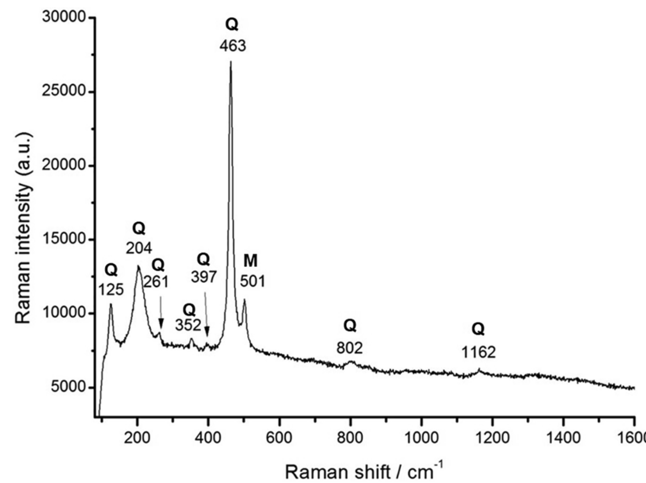

Fig. 11. Raman spectrum of 21-1066 carnelian bead. Experimental conditions of Renishaw 'in via' Reflex Spectrometer: 50x objective, 5 accumulations of 10 s, 785 nm diode laser, 3 mW. Q: quartz; M: moganite.

onwards, Iberian vessels were decorated only with shades of red obtained from hematite and cinnabar (Sánchez et al., 2012, Tuñón et al., 2016).

5.2. Beads

The carnelian and glass paste beads (Fig. 5) also refer to external influence and to a role in the funerary ritual. Both types are frequently found in the Eastern Mediterranean, but they are rare in graves of the southern and Eastern Iberian Peninsula from the 8th century BC (Martínez and Vilaplana, 2014). Their role in burial sites must have been as personal adornment, as rank or social status markers, as charms or as religious symbols. Carnelian has also been attributed beneficial health properties, so it could be related to the wish for the deceased's regeneration and durability among the living. This gem thought to cleanse blood and improve health. Carnelian has also been thought for a long time to stop bleeding and to help heal wounds (Economou et al., 2010). These roles overlap so much that it is virtually impossible to tell one from the other.

The occurrence of carnelian is particularly relevant, as it is not found in the Iberian Peninsula and the main supply areas was from Gujarat (India) and the Sahara desert in Egypt, where deposits of agatha, carnelian and onyx lay (Aston et al., 2006; Gliozzo et al., 2014; Roux, 2000). Smaller outcrops can also be found in Armenia or Ra's al-Khaymah (Brunet, 2009). The provenance of the carnelian beads of *Cerro de los Vientos* is not known, but the typology of the earliest attested carnelian ornaments in southern Spain and Portugal late in the second millennium BC (i.e. pendants with vegetal motifs) relates them

Table 7
Beads EDXRF results shown (wt%) are the average of three analyses per sample. Standard deviations (s.d.).

Bead	Na2O	MgO	Al2O3	SiO2	P2O5	SO3	Cl	K2O	CaO	TiO2	V2O5	MnO	Fe2O3	CoO	Ni2O3	CuO	ZnO	SrO	BaO
20-1066	bdl	bdl	0,20	99,38	bdl	0,06	0,06	0,01	0,06	0,02	0,05	0,03	0,08	bdl	0,01	bdl	0,01	bdl	0,04
(s.d.)	-	-	(0,05)	(0,05)	-	(0,02)	(0,01)	(0,01)	(0,00)	(0,01)	(0,02)	(0,01)	(0,03)	-	(0,00)	-	(0,00)	-	(0,00)
21-1005	bdl	bdl	0,16	99,13	bdl	bdl	0,07	0,12	0,30	0,02	0,05	0,02	0,11	bdl	bdl	bdl	0,01	bdl	0,01
(s.d.)	-	-	(0,15)	(0,64)	-	-	(0,02)	(0,02)	(0,30)	(0,01)	(0,02)	(0,01)	(0,04)	-	-	-	(0,00)	-	(0,01)
22-1062-1	1,22	1,78	7,51	78,78	1,70	0,36	1,38	0,65	4,15	0,13	bdl	0,56	1,51	0,09	0,07	0,01	0,09	0,01	bdl
(s.d.)	(1,48)	(0,45)	(0,46)	(1,93)	(1,39)	(0,10)	(0,02)	(0,05)	(0,42)	(0,01)	-	(0,01)	(0,06)	(0,01)	(0,01)	(0,01)	(0,00)	(0,00)	-
23-1062-2	0,80	bdl	7,01	72,96	0,20	0,30	0,96	0,59	14,08	0,22	bdl	0,47	1,79	0,07	0,07	0,35	0,09	0,04	bdl
(s.d.)	(1,39)	-	(1,25)	(1,69)	(0,35)	(0,18)	(0,28)	(0,10)	(1,38)	(0,05)	-	(0,05)	(0,21)	(0,03)	(0,02)	(0,14)	(0,02)	(0,02)	-
24-1033-1	1,21	0,73	7,65	80,47	bdl	0,29	1,05	0,74	5,86	0,14	bdl	0,33	1,15	0,08	0,08	0,03	0,11	0,06	0,01
(s.d.)	(1,05)	(0,52)	(2,1)	(1,57)	-	(0,15)	(0,32)	(0,27)	(0,9)	(0,02)	-	(0,05)	(0,13)	(0,01)	(0,01)	(0,01)	(0,01)	(0,01)	(0,01)
25-1033-2	1,27	0,88	6,40	81,46	bdl	0,32	1,14	0,61	5,69	0,15	bdl	0,38	1,28	0,09	0,09	0,03	0,12	0,07	0,02
(s.d.)	(0,8)	(0,27)	(0,44)	(0,48)	-	(0,10)	(0,15)	(0,10)	(0,16)	(0,01)	-	(0,02)	(0,22)	(0,01)	(0,01)	(0,01)	(0,02)	(0,03)	(0,02)
26-1033-3	1,33	1,92	8,74 (1,50)	78,01	bdl	0,53	0,69	0,78	5,65	0,10	bdl	0,50	1,39	0,10	0,11	0,01	0,12	0,03	bdl
(s.d.)	(1,47)	(1,14)		(0,82)	-	(0,09)	(0,11)	(0,36)	(1,06)	(0,02)	-	(0,04)	(0,17)	(0,01)	(0,01)	(0,00)	(0,02)	(0,01)	-
27-1033-4	2,61	1,57	5,87	80,22	bdl	0,38	1,34	0,50	5,60	0,13	bdl	0,34	1,10	0,08	0,08	0,01	0,10	0,06	bdl
(s.d.)	(0,77)	(0,15)	(0,27)	(1,25)	-	(0,13)	(0,14)	(0,09)	(0,33)	(0,01)	-	(0,05)	(0,00)	(0,00)	(0,01)	(0,01)	(0,01)	(0,02)	-
28-1033-5	0,20	1,86	6,25	82,99	bdl	0,62	0,63	0,35	5,06	0,07	bdl	0,46	1,18	0,10	0,07	0,01	0,10	0,05	bdl
(s.d.)	(0,23)	(0,35)	(0,18)	(0,41)	-	(0,10)	(0,01)	(0,00)	(0,25)	(0,00)	-	(0,03)	(0,10)	(0,01)	(0,01)	(0,00)	(0,01)	(0,02)	-

to Egyptian pendants that spread over the Mediterranean, possibly by Cypriot merchants. Dissemination of carnelian ornaments and beads from the late 9th century BC sets in the framework of contact and exchange between the Phoenician trading network and the native coastal and inland settlements of the Iberian Peninsula. Carnelian beads have been recorded in the sites of *Peña Negra* I (Crevillente, Alicante), *La Fonteta* (Guardamar de Segura, Alicante), *Pocito Chico* (Puerto de Santa María, Cádiz), and *Alarcos* (Ciudad Real) (Martínez and Vilaplana, 2014; Guirao, 2018).

Glass paste eye beads became widespread in Egypt from the middle of the second millennium BC, but their main dissemination was mainly by Phoenician trading (Palomar et al., 2009). They spread over the Iberian Peninsula similarly to carnelian, even if, like carnelian, their origin is unknown. The meaning of these beads, especially in the case of eye beads as in *Cerro de los Vientos* (Fig. 5), has been interpreted as magical or protective items. They were not just ornaments: they were medical, magical charms against the evil eye, as curses were considered to manifest themselves as diseases. Colours were major elements in their effectiveness. Blue colour was also associated with the sky, as a symbol for eternity and immortality (Vaquero, 2012).

Regarding sex distinction, the link between these items as grave goods and the sex of the deceased has not been established in most cases in the Iberian Peninsula (Martínez and Vilaplana, 2014). The beads in *Cerro de los Vientos* are an early example where beads can be associated with females. According to the anthropological analysis of the cremated bone remains (Trancho and Robledo, 2016), carnelians are linked up to an urn with remains of an adult female (urn 5-1011, structure E5). The beads of glass paste were also retrieved from an urn with bone remains of a subadult female (urn 7-29, structure E26).

5.3. Ceramic contents

The third issue about the ceramic group under study is the organic contents preserved in the vessels: mainly beeswax and vegetable fat (Table 4). Use of beeswax in Prehistory and Protohistory has been interpreted variously: for medical use, as a cosmetic, for rites, as a protection against corrosion, for paints, for boat building, as a glue, as an insulating substance, for consumption of honey, mead and other alcoholic drinks, and as fuel for lighting (Evershed et al., 1997; Kimpe et al., 2002; Łucejko et al., 2017; McGovern et al., 2013; Regert et al., 2001; Ribechini et al., 2011; Roffet-Salque et al., 2015; Parras et al., 2015).

The identification of beeswax in the ceramic vessels of *Cerro de los Vientos* helps to better understand the types of rituals carried out in the two areas already discussed above. Viewed together, the chemical data recorded and the contexts where the vessels were retrieved from (ritual and funerary) hint a likely link between beeswax and honey. The honey would have been used, transformed into hydromel, for ritual ceremonies of consumption held in structures E28 and E32 (Fig. 2). It would also be used for libations and funerary offerings in graves E5, E26 and E34 (Fig. 3).

Beeswax has been recorded for ritual and funerary purposes in bell beakers and Argaric vessels of the 3rd and 2nd millennia BC in the Iberian Peninsula. Still, only the beeswax recorded in grave 121 of the Argaric site of *Castellón Alto* (Parras et al., 2011) is a sound hypothesis. The identification of beeswax in bell beakers of the Iberian Peninsula is quite questionable, in that the experimental data of the chemical analyses have never been published (Bueno et al., 2005; Guerra, 2006). A contemporary referent of this funerary purpose has been evidenced in two graves of the cemetery of *Alarcos* (7th century BC, Ciudad Real, Spain), where beeswax was recorded in three small vessels of the Orientalizing period, two of them bowls (Sánchez et al., 2019). The sustained relevance of beeswax stands out in the Iberian period in the province of Jaén: mixed with animal and vegetable fat and/or sulphur, beeswax was part of the offerings and of the rites of the Sanctuary of *Puerta del Sol* in the *oppidum* of *Puente Tablas* (late 5th to 4th century BC) (Parras et al., 2015).

The link between beeswax and honey in the burial site of *Cerro de los Vientos* is strengthened by the classical sources and the archaeological evidence available on the use and consumption of honey in the Mediterranean before the 7th century BC. Virtually all the Egyptian funerary rites were about the afterlife, where food and drink were also necessary. Honey was, therefore, an important element of the *opening of the mouth* ritual, whereby the deceased was given the food for the afterlife. Honey has also been recorded as an offering as a result of the evidence of vessels with honey in the private graves of the 9th Dynasty (2000 BC) in Gebelein, or in view of the honeycomb found in a grave in Deir-el-Medina dating back to 1350 BC (Fernández, 2011).

Honey and its derivatives were a consumption good all over the Near East and Eastern Mediterranean (Ransome, 2004; Wilson, 2004). The Phoenician cremation site of *Al-Bass* in Tyre (Lebanon), operational between the 10th and the 7th centuries BC, sets an archaeological example despite the poor preservation as a result of the sand stratum constantly being washed and disturbed by the bed of the lower water table. Nonetheless, the remains of beeswax found inside mushroom-shaped jugs suggest that they may have contained honey or hydromel (Aubet, 2010). A funerary ritual in honour of the king's first wife in the first half of the 7th century BC, during Assyrian king Esarshaddon's reign, included libation of honey and oil in a grave (Bachvarova, 2016).

Honey was also used as a drink and as an offering to the dead and to the deities of the underworld in Greece's funerary rituals and cults since Ancient times, both in funerary pyres and for libation in graves. Homer's *The Illiad* and *The Odyssey* refer outstanding examples for the use of honey early in the 1st millennium BC. In *The Iliad*'s funeral for Patroclus, Achilles lays two amphorae of honey and oil by the pyre (scroll XXIII, 170 8), and *The Odyssey* refers Achilles' funeral wrapped in ointments and honey (scroll XIV 67-68). *The Odyssey* also refers ceremonial libation of milk and honey, wine and water, in honour of the deceased following Circe's advice (scrolls X 518, XI 26). For the transformation of Odysseus' men into pigs, Circe offered cheese, flour, and amber-coloured honey with Pramnios wine to mask her potion (scroll X, 233-236).

The occurrence of beeswax in the ritual and the funerary area of *Cerro de los Vientos* entails a particular, complementary interpretation and in relation to the abovementioned Mediterranean cultural setting.

The area of structures E28 and E32 (Fig. 2), somewhat away from the other structures and where no ritual burial has been recorded, is related to ritual consumption. Structure E28 hosts native tradition vessels (three bowls), Phoenician tradition vessels (three large storage vessels and one plate) and imitations of the latter (two bowls/plates). Beeswax is recorded in four vessels (two native bowls and two Phoenician containers). The arrangement of bowls and larger vessels as containers may suggest ritual consumption where beeswax may in turn be a hint of a related product. The simplest interpretation is for beeswax as an inside coating. Consumption as an exclusive produce of a ritual kind and of Mediterranean and Eastern origin is another interpretation. The types of vessels, the separation with respect to the funerary space and the link between beeswax and honey suggest ritual consumption, e.g. hydromel. It may also have been mixed with wine or milk, as was usual in the Mediterranean world, but this research paper did not record these chemical markers (Ransome, 2004; Wilson, 2004; Fernández, 2011).

The analysis of the funerary space contributes relevant qualifications. Two bowls, one of them with the abovementioned ceramic stand, were placed in grave E5, next to urn 5-1011 (Fig. 4) with the remains of a female, beeswax chemical markers (Fig. 9) and carnelian beads (Fig. 5). The bowl on the stand, where beeswax was recorded, must have contained a honey- or hydromel-related offering that stained the stand's inside when the bowl was filled or if it keeled over. Beeswax was also recorded in urn 7-29 of grave E26, which held the remains of a subadult female, and in urn 19-1034 of grave E34, which held the remains of a subadult of unidentified sex. The link between beeswax and honey makes sense here again. Honey was used as food for the journey to the underworld, as an offering to its deities and for literal or, as in these cremations, symbolic preservation of the deceased (Ransome, 2004; Wilson, 2004; Fernández, 2011). Therefore, honey must have been poured before the cremated remains of the deceased into the urns. Urn 5-1011 is a different case, in that the wish for durability in the afterlife was strengthened with carnelian beads and with a special offering including the bowl and the decorated stand. These and other metal items of the grave goods reveal the relevance of the female in the urn for the community of *Cerro de los Vientos*.

Urn 6-1038 of grave E25 differs substantially from the ones described above: it did not hold any type of remains, so honey was not used as food for the afterlife and as an offering. This grave may have been used as a cenotaph where honey was unnecessary because there were no remains. Vegetable fat was however recorded here, probably from some kind of rare and exclusive oil (Table 4). It may therefore suggest offering or libation on the urn. The urn in question was wheelmade and seemed to contain an unidentified iron item as grave goods.

6. Conclusions

This paper underlines the advantages derived from the interaction of various techniques of physico-chemical analysis (MRS, EDXRF and GC–MS) for research on the decoration and contents of a set of ceramic vessels of the Orientalizing period. The concept *contents* was here used in a broad sense, as the research covered both chemical markers and the items contained in the vessels.

MRS analysis and EDXRF analysis allow to identify and separate two decoration traditions in the pottery retrieved from the ritual area and from the graves of *Cerro de los Vientos*. Joint use of red (hematite), yellow (goethite) and black (amorphous carbon and graphite) decorations belong to native tradition (samples 3-1010, 8-1022 and 14-1021). The black and red decoration in oxidation firing wheelmade pottery obtained from hematite (samples 6-1038, 11-1019, 12-1018, 17-1039 and 18-1007) and/or from mangenese oxide (bixbyite/hausmannite) (sample 15-1020) finds its origin in the Phoenician influence in the Iberian Peninsula. This technique and its aesthetic result remained in the native culture of the 6th and 5th centuries BC of the southern Iberian Peninsula known as Culture of the Iberians.

The identification of the raw materials used for the orange (carnelian) and blue (glass paste) beads signals an origin other than the native communities in the Iberian Peninsula. As carnelian is not found in the Iberian Peninsula, as there is no evidence of glass paste made in the Iberian Peninsula, and as the two types of beads were widely used in the Eastern Mediterranean, it can be concluded that the beads are related with the Phoenician culture in the Iberian Peninsula from the late 9th century BC.

The archaeometric analysis could not have been completed without joint use of physico-chemical data with the archaeological context. The ritual and funerary nature of the site *Cerro de los Vientos* entails a number of connotations with an influence on the interpretation of decorations, of contents and even of the carnelian beads. Beeswax is a particularly relevant point. Its link with honey or hydromel makes sense only in a clearly ritual and funerary context, and within a native social group under the influence of the contact with other societies, specifically Phoenician and Mediterranean. The highly influential Eastern Mediterranean framework of reference stands out for its importance in this piece of research, as attested by the written sources and by the archaeological data of this cultural setting.

The community of *Cerro de los Vientos* used both native and Phoenician type vessels in consumption rituals, probably as a funerary feast with hydromel, according to the abundant records of beeswax. Hydromel was stored in Phoenician type ceramic vessels (samples 10-1029 and 12-1018) and at least served in native vessels (samples 8-1022 and 13-1024). GC–MS analysis did not identify beeswax markers in the rest of vessels of Phoenician influence (samples 11-1019, 15-1020 and 17-1039) or in Phoenician wheelmade native imitation vessels (samples 9-1050 and 16-1023).

The cremated remains were laid in native ceramic urns in the burial area, and honey offerings were made as food for the afterlife, as offerings to the deities and as a regenerating substance (samples 5-1011, 7-29 and 19-1034). The latter role must have been intended to be strengthened by the use of carnelian, as recorded in the urn of the most important female of the burial site (structure E5).

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