ESTUDOS ARQUEOLÓGICOS DE OEIRAS

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ACTAS DO IX CONGRESSO IBÉRICO DE ARQUEOMETRIA
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Editores Científicos: M. Isabel Dias e João Luís Cardoso

APRESENTAÇÃO

A Nona Edição do Congresso Ibérico de Arqueometria (CIA IX) decorreu em Lisboa de 26 a 28 de Outubro de 2011 nas instalações da Fundação Calouste Gulbenkian. A proposta e compromisso da organização deste evento foi feita pelo Grupo de Geoquímica Aplicada & Luminescência no Património Cultural (GeoLuC) (IST/ITN), dois anos antes na Assembleia Geral da Sociedad de Arqueometría Aplicada al Patrimonio Cultural (SAPaC), e foi aceite por unanimidade.

Com esta decisão, a SAPaC consolida uma linha de actuação, cujo objectivo é difundir e fomentar a colaboração entre os grupos de investigação arqueométrica que trabalham na Península Ibérica. Este objectivo viu-se reforçado e reflectido na composição dos novos órgãos sociais dirigentes da SAPaC, eleita durante a celebração do IX Congresso em Lisboa, que incorpora deste então investigadores portugueses e espanhóis, sendo presidida pela Doutora M. Isabel Dias (IST/ITN, Portugal).

As Actas que aqui se apresentam são uma prova tangível da via integradora desta IX edição do Congresso, verificando-se existir equilíbrio numérico entre os trabalhos apresentados por grupos de investigação portugueses e espanhóis, evidenciando-se mesmo um incremento de projectos em que participam conjuntamente investigadores dos dois países, mostrando o grande interesse que desperta a Arqueometria, em si mesma de natureza interdisciplinar, e os objectivos comuns partilhados pela comunidade científica ibérica.

Definitivamente, este Congresso constituiu um ponto de encontro dos investigadores da disciplina, tendo contribuído para a troca de experiências e o aprofundar de conhecimentos nas diversas metodologias e técnicas aplicadas à caracterização do nosso património histórico e cultural.

A publicação dos trabalhos do CIA IX nos *Estudos Arqueológicos de Oeiras* (EAO), órgão científico do Centro de Estudos Arqueológicos do Concelho de Oeiras/Câmara Municipal de Oeiras, constituiu uma oportunidade única e vantajosa para ambas as partes, já que esta inédita parceria entre uma entidade vocacionada para a investigação e uma Câmara Municipal permitiu uma sinergia de interesses quanto aos custos da publicação deste número e a sua adequada distribuição nacional e internacional. A escolha de uma revista periódica constituiu sem dúvida, a melhor opção, para a garantia de uma divulgação adequada. E a revista sobre a qual recaiu a escolha, prontamente homologada pelo Senhor Presidente da Câmara Municipal de Oeiras, Dr. Isaltino Morais, responde sem dúvida àquele requisito: além de constituir uma referência no panorama editorial nacional em matéria de publicações arqueológicas, com 18 números publicados desde 1991, mantém permuta com cerca de 200 revistas periódicas especializadas, todas de

Arqueologia e Património Arqueológico, especialmente de Espanha, França, Itália, Alemanha, Polónia, Reino Unido, Mónaco e Marrocos, para além de Portugal, incluindo as publicações mais importantes produzidas naqueles países.

Esperamos, deste modo, com a publicação deste volume, ir ao encontro dos interesses de todos os participantes do CIA IX, de todos os que contribuíram com os seus trabalhos para a excelente qualidade deste volume, dos interesses dos associados da SAPaC, dos munícipes de Oeiras, e da comunidade científica nacional e internacional no domínio da arqueometria e da arqueologia.

Pela Comissão organizadora do CIA IX, Presidência da SAPaC
e comissão editorial deste volume dos Estudos Arqueológicos de Oeiras,

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Oeiras, 31 de Outubro de 2012

ARCHAEOMETRIC STUDY OF IBERIAN POTTERY FROM "EL CASTILLEJO" (ALAMEDA, MALAGA, SPAIN)

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Resumen

El yacimiento arqueológico de "El Castillejo" está situado en la colina homónima, muy cerca del casco urbano de Alameda (Málaga, España). El estudio de sus materiales de superficie muestra un largo periodo de ocupación, desde el Calcolítico hasta los inicios del Imperio Romano. La mayor parte de los restos abarcan desde el Bronce Final (hacia los siglos VIII-VII a.C.), como consecuencia del comercio con los asentamientos costeros fenicios, hasta época Ibérica (hacia el siglo III a.C.), con la construcción de un oppidum. En este trabajo se han estudiado 17 fragmentos de cerámicas pintadas, principalmente ibéricas. Las muestras han sido analizadas utilizando fluorescencia de rayos-X (WDXRF) y difracción de rayos-X acoplada al método de Rietveld (RQPA). La microestructura de las muestras ha sido observada utilizando microscopía electrónica de Barrido (SEM). Los pigmentos han sido analizados utilizando micro-Raman.

Palabras clave: Difracción de rayos-X, Microscopía electrónica de barrido, Fluorescencia de rayos-X, micro-Raman, Cerámica ibérica

Abstract

The "El Castillejo" archaeological site is placed in the homonymous hill, very close to the village of Alameda (Malaga, Spain). The study of the surface materials showed a wide period of occupation, from the Copper Age to the Early Roman Empire. The major part of the evidences dates from the Late Bronze Age (close to VIII-VII centuries BC), as a consequence of the trade with the Phoenician coastal settlements, to Iberian times (around III century BC) when an *oppidum* was built. In this work, 17 painted sherds, mainly Iberian, have been selected. The samples have been analyzed using X-ray fluorescence (WDXRF) and X-ray diffraction coupled to the Rietveld Method (RQPA). The microstructure of the samples have been observed using Scanning Electron Microscopy (SEM). The pigments have been analyzed using micro-Raman.

Keywords: X-ray powder diffraction, Scanning electron microscopy, X-ray fluorescence, micro-Raman, Iberian pottery

1 – INTRODUCTION

The "El Castillejo" archaeological site is placed in the homonymous hill, very close to the village of Alameda (Malaga, Spain). It is a smooth hill with a wide top, between two small brooks (now covered), so it is a very strategic place for human settlement (Fig. 1). Its first study was carried out in the late eighties by the

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Diputación de Málaga, in the frame of the local urban development. The field prospection provided evidences of a wide variety of archaeological materials including a high amount of pottery. In addition to the handmade pottery, there is some wheeled pottery, like polished grey wares, Iberian amphorae, and even Phoenician *pithoi*. The study of those materials pointed to a probable early occupation in the Copper Age, followed by a wider development in the Late Bronze Age (close to VIII-VII centuries BC) and later (RECIO RUÍZ, 1998, p. 458). The most important occupation of the settlement is, without any doubt, the Iberian period, with the building of an *oppidum* at the top of the hill (about V-III centuries BC). The major part of the ceramic evidences are also from this period, including the typical Iberian painted wares (LÓPEZ GARCÍA, 2006, p. 113). The most recent chronological evidences of the site date from the Early Roman Empire.
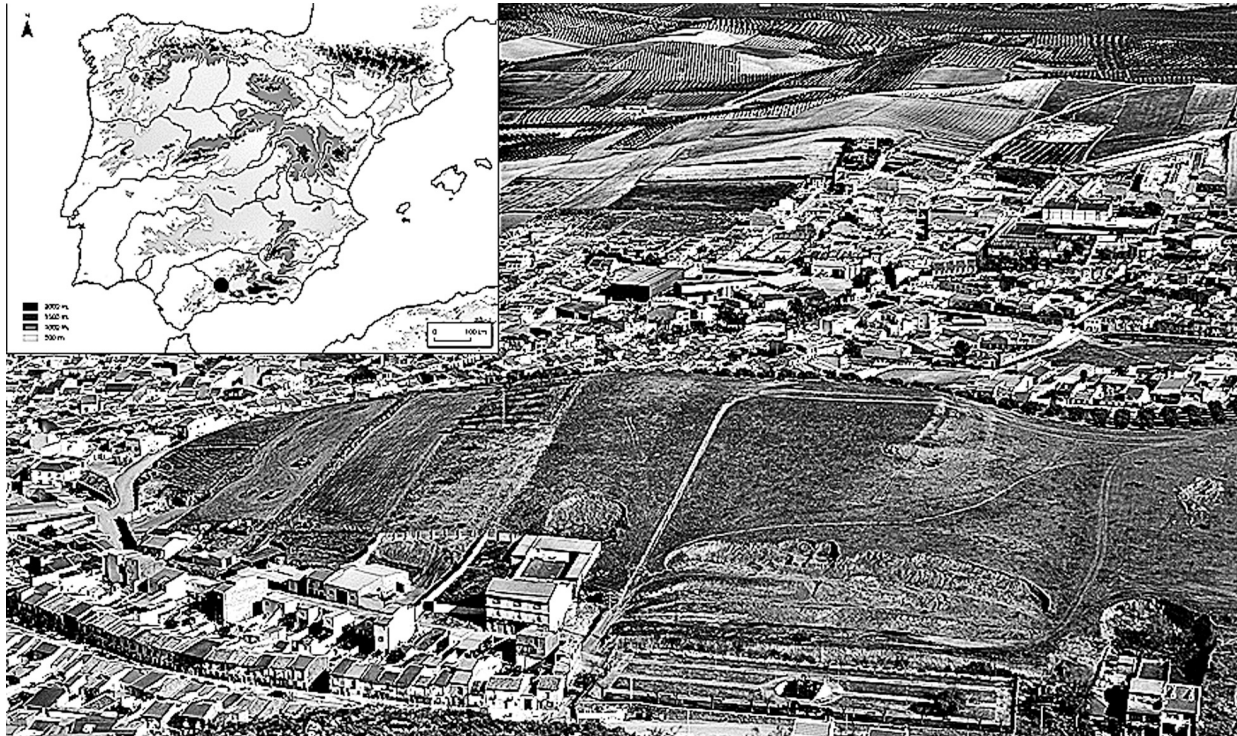

Fig. 1 – The "El Castillejo" *oppidum* in January-2012.

Unfortunately, our knowledge of the "El Castillejo" *oppidum* is limited to the two surface works already cited. Here, we report a characterization of the recovered ceramics, in order to get more information of the supplies of the *oppidum*, from an archaeometric approach.

2 – SAMPLING

All the provided material came from old surface prospections, so we have chosen those materials which probably would yield the most useful information. A series of 17 painted shreds, very likely contemporary to the *oppidum,* were chosen. All of them are painted, mostly in the Iberian way, with reddish and brownish final colors. Some of them show a typical "sandwich" fabric (oxidizing firing near the surfaces, and reducing firing in the core). Macroscopically, some of these sandwich fabrics are very similar to the Phoenician fabrics

from the Coast of Malaga (GONZÁLEZ PRATS, 1986, p. 292-294; RAMÓN TORRES, 1995, p. 256-257), also present in the site, so it was an interesting question to determine whether they were related to them, or not. The inventory of the analysed samples is listed in Table 1.

3 – EXPERIMENTAL TECHNIQUES. RESULTS

Freshly broken surfaces of the samples were initially observed, photographed and described at 10× and 20× using a stereomicroscope Olympus SZX7, equipped with a digital camera Olympus SC30. Later, the microstructure of representative samples was also observed in a JEOL JSM-6490LV scanning electron microscope, after gold-coating, using secondary electrons and an acceleration voltage of 20 kV.

Table 1 – Inventory of the analyzed samples.

Sample	Description	Firing	Pigment
IBE001	Pithoi wall	Ox	Black
IBE002	Pithoi wall	Ox/Red	Black, Red
IBE003	Amphora wall	Red	Brown, Red
IBE004	Bowl bottom	Ox	Brownish red
IBE005	Bowl bottom?	Ox/Red	Red
IBE006	Dish?	Ox/Red	Red
IBE007	Dish?	Ox	Red
IBE008	Pithoi wall?	Ox/Red	Red
IBE009	Amphora wall	Ox/Red	Brown
IBE010	Amphora wall	Ox/Red	Brown
IBE011	Amphora wall	Red	Red
IBE012	Amphora wall	Ox/Red	Brown, Red
IBE013	Amphora wall	Ox/Red	Wine-red
IBE014	Amphora wall	Ox/Red	Brown, Red
IBE015	Amphora wall	Ox/Red	Wine-red
IBE016	Amphora wall	Ox/Red	Wine-red
IBE017	Amphora wall	Ox	Blackish

After their macroscopic study, small pieces of the samples were reduced to a fine powder in an agate mortar. The powdered samples were analyzed using X-ray diffraction, coupled to the Rietveld Method (RQPA) and X-Ray fluorescence. This method has given good results in former works (COMPAÑA *et al.* 2010).

Laboratory X-ray powder diffraction data (LXRPD) for the pastes were collected on the PANanalytical X'Pert PRO MPD diffractometer of University of Malaga. The powder XRD patterns were recorded in all cases in Bragg–Brentano reflection configuration by using a Ge(111) primary monochromator (CuKα_1) and the X'Celerator detector. The XRPD patterns were recorded between 5 and 80º in $\theta/2\theta$ mode with a step size of 0.017º (2θ). The X-ray tube worked at 45 kV and 40 mA. All XRPD data treatments (identification, cluster analysis and mineralogical quantification) were carried out with PANalytical HighScore Plus 2.2.d software. The results of the phase quantification are given in Table 2. The mineral names cited in this work are abbreviated in the usual way in Geology (KRETZ, 1983).

Table 2 – Mineralogical composition (wt %) of the pastes of the studied samples from the Rietveld analysis of LXRPD data.

Sample	Qtz	Cal	Mca[1]	Pl[2]	Kfs[3]	Hem	Px[4]	Geh	Ant	Rt	Dol	Arg	FT (°C)[5]	R_{wp}[6]
IBE001	48.6(3)	10.4(2)	13.0(6)	14.5(5)	9.5(6)	2.0(2)	–	2.0(2)	–	–	–	–	750-850	7.34
IBE002	53.8(3)	12.3(9)	10.7(6)	18.6(5)	2.7(4)	0.4(1)	–	1.3(2)	–	–	–	–	750-850	5.88
IBE003	55.9(3)	17.1(2)	8.1(5)	11.9(4)	–	–	5.2(5)	–	0.5(1)	1.4(1)	–	–	800-900	6.46
IBE004	34.4(2)	42.5(9)	11.3(5)	3.0(2)	1.3(2)	–	–	–	–	–	1.5(1)	–	700-800	6.30
IBE005	72.2(6)	15.1(8)	12.8(4)	–	–	–	–	–	–	–	–	–	700-800	13.32
IBE006	70.0(3)	10.6(2)	16.6(7)	2.8(4)	–	–	–	–	–	–	–	–	700-800	6.76
IBE007	24.9(4)	-	7.0(9)	15.0(7)	44.2(1)	0.6(2)	8.4(8)	–	–	–	–	–	850-950	8.46
IBE008	69.7(3)	5.8(2)	7.2(6)	10.1(5)	–	–	5.9(6)	1.2(2)	–	–	–	–	800-900	7.57
IBE009	33.0(2)	15.6(4)	1.9(3)	18.7(5)	–	–	4.2(4)	26.6(3)	–	–	–	–	750-850	6.15
IBE010	26.1(2)	49.2(5)	4.4(4)	2.5(3)	–	–	–	6.7(2)	–	–	–	11.0(3)	750-850	5.96
IBE011	54.8(3)	8.8(4)	3.9(4)	9.9(4)	2.7(3)	–	14.4(9)	5.5(2)	–	–	–	–	800-900	5.34
IBE012	35.8(2)	20.9(5)	–	19.9(5)	–	–	2.1(4)	21.4(3)	–	–	–	–	750-850	6.78
IBE013	55.5(3)	33.4(5)	9.5(5)	-	–	–	–	–	–	–	1.56(7)	–	700-800	9.07
IBE014	27.2(2)	21.7(4)	2.6(4)	19.9(5)	–	–	5.5(5)	23.1(3)	–	–	–	–	750-850	6.26
IBE015	72.3(4)	18.4(6)	8.0(5)	–	–	0.40(9)	–	1.0(4)	–	–	–	–	800-900	8.22
IBE016	65.2(3)	29.6(6)	5.1(5)	–	–	–	–	–	–	–	–	–	700-800	7.69
IBE017	34.8(2)	18.5(5)	8.0(6)	12.7(4)	–	0.5(1)	18.8(7)	6.7(3)	–	–	–	–	850-950	5.41

[1]Mica is accounted for as sericite. [2]Plagioclases are accounted for as albite and anorthite. [3]Potassium-feldspars are accounted for as orthoclase. [4]Pyroxenes are accounted for as diopside and augite. [5]FT stands for the estimated firing temperature. [6]Rwp is a disagreement factor which gives an indication of the quality of the fit.

Quantitative chemical analysis were performed at the Application Development Center of PANalytical BV (Almelo, Netherlands), using an Axios XRF spectrometer. The operation conditions were a maximum voltage 60 kV and a maximum current 100 mA, using a 27 mm collimator mask and a measuring time 20 min. The spectra were analyzed by means of the Omnian software of PANalytical BV. The chemical data, expressed as oxides wt %, are given in Table 3.

Table 3 – Normalized chemical composition of the pastes of the studied samples (XRF) expressed as oxide contents (wt %).

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	MnO	Fe_2O_3	NiO	CuO	ZnO	Rb_2O	SrO	ZrO_2	BaO
IBE001	0.634	2.706	16.724	55.504	0.344	0.226	2.444	12.588	0.902	0.194	7.217	0.023	0.011	0.018	0.011	0.037	0.049	0.369
IBE002	0.737	2.788	17.570	54.157	0.443	0.133	2.425	13.087	0.902	0.173	7.248	0.019	0.005	0.020	0.012	0.038	0.041	0.201
IBE003	0.496	3.762	16.977	54.590	0.243	0.273	3.340	11.669	0.827	0.181	7.180	0.018	0.015	0.020	0.015	0.044	0.040	0.309
IBE004	0.325	3.105	12.715	44.511	0.236	0.323	3.218	28.250	0.765	0.082	5.647	0.015	0.010	0.015	0.010	0.097	0.032	0.643
IBE005	0.259	1.700	16.395	57.283	0.157	0.093	2.883	13.083	0.758	0.071	6.644	0.013	0.009	0.012	0.011	0.041	0.045	0.542
IBE006	0.445	2.390	16.031	62.858	0.186	0.104	3.174	7.475	0.696	0.061	6.198	0.008	0.011	0.019	0.013	0.039	0.033	0.259
IBE007	1.046	1.202	22.221	57.744	0.189	0.336	3.642	3.946	0.901	0.170	7.324	0.006	0.006	0.014	0.020	0.065	0.072	1.097
IBE008	0.264	2.820	15.459	57.596	0.240	0.240	3.722	12.391	0.690	0.113	6.089	0.015	0.007	0.013	0.015	0.059	0.033	0.234
IBE009	0.247	1.586	15.370	41.613	0.290	0.228	1.857	30.299	0.620	0.153	7.154	0.020	0.011	0.014	0.012	0.107	0.018	0.400
IBE010	0.293	0.991	11.180	35.008	0.175	0.110	1.663	44.406	0.560	0.143	4.956	0.015	0.005	0.014	0.012	0.094	0.016	0.358
IBE011	0.350	3.705	16.029	51.896	0.398	0.207	2.963	15.085	0.886	0.230	7.753	0.016	0.011	0.019	0.015	0.039	0.045	0.352
IBE012	0.202	1.547	14.951	41.846	0.175	0.199	1.846	30.743	0.614	0.169	7.099	0.015	0.011	0.014	0.011	0.098	0.020	0.440
IBE013	0.249	1.461	13.311	51.616	0.326	0.131	2.307	25.047	0.538	0.133	4.338	0.012	0.013	0.013	0.013	0.070	0.024	0.401

Table 3 – Normalized chemical composition of the pastes of the studied samples (XRF) expressed as oxide contents (wt %).

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	MnO	Fe_2O_3	NiO	CuO	ZnO	Rb_2O	SrO	ZrO_2	BaO
IBE014	0.237	1.771	14.887	41.347	0.221	0.269	1.854	30.663	0.591	0.193	7.329	0.013	0.010	0.016	0.011	0.101	0.024	0.462
IBE015	0.237	2.062	12.509	56.103	0.321	0.279	2.551	19.490	0.626	0.129	5.203	0.012	0.011	0.013	0.010	0.063	0.023	0.356
IBE016	0.237	1.883	15.191	55.320	0.327	0.216	1.972	17.316	0.677	0.192	6.001	0.009	0.012	0.017	0.007	0.060	0.034	0.527
IBE017	0.372	2.359	14.734	49.939	0.194	0.281	2.907	21.667	0.790	0.073	6.374	0.011	0.008	0.012	0.012	0.093	0.027	0.146

Raman spectra of the pigments of selected samples (IBE002, IBE004, IBE013, IBE014 and IBE015) were collected using a Bruker Senterra micro-Raman spectrometer at University of Málaga. Samples were placed on the microscope stage and a 633 nm laser beam was focused on the various pigmented areas of each specimen by using an Olympus objective with a 20x magnification. Raman spectra were recorded from 100 to 1800 cm⁻¹, and during every measurement the samples were exposed to the laser beam for 25 seconds, taking from 6 to 10 accumulations. The 633 nm laser radiation was used as excitation source and the laser beam power was chosen as low as possible to avoid for mineralogical changes (typically the laser power at the sample was kept to 2 mW).

4 – DISCUSSION

The cluster analysis of LXRPD raw data gives a first grouping, but very interesting (Fig. 2). Discarding the three obvious outliers (samples IBE005, IBE007 and IBE010), the remaining samples form three clear clusters. The mineralogy of cluster-1 samples is characterized by an intermediate amount of calcite and gehlenite. Cluster-2 samples are characterized by a high amount of calcite, and almost complete absence of gehlenite. Conversely, cluster-3 samples show a high amount of calcite and gehlenite. Taking into account the relatively high amounts of calcite in most of the samples, this may be correlated to the different degree of firing (TRINDADE *et al.*, 2009). The approximate firing temperatures given in Table 2 are a rough estimate both from the mineralogy and the SEM microstructure of the samples. Nevertheless, the technological differences which may be inferred from these evidences are too hard and intricate to be discussed here, and will be the subject of a future and wider work.
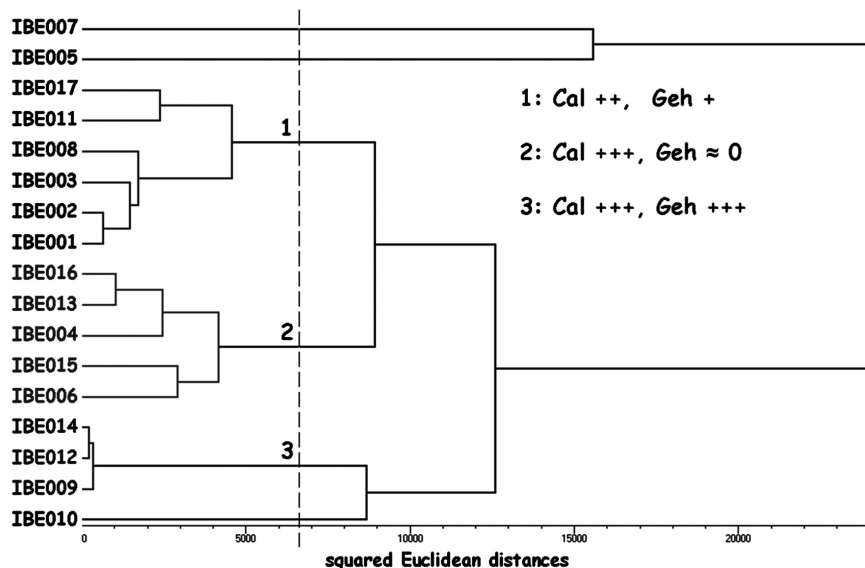

Fig. 2 – Dendrogram of Euclidean distances, average linkage method, calculated using LXRPD raw patterns.

The cluster analysis of chemical composition data also provide a very interesting grouping (Fig. 3). The analysis is carried out through the logratio transformations proposed by Aitchison (AITCHISON, 2003). It is interesting to check that, again, sample IBE007 is an outlier. This made us re-examine carefully sample IBE007. This new more careful examination allows determining that was not a painted potsherd, but internally slipped in red. It is not an Iberian nor a Phoenician ware, but a Pompeian Red Ware. Moreover, it is clearly a sample of fabric 1 as defined by Peacock (PEACOCK, 1977, p. 149-153), which is well known to be Campanian. This fabric is the commonest Pompeian Red fabric found in the Mediterranean region, and it is often found in Spain. It is also defined as fabric 2 in the *Tarraconensis* by Aguarod (AGUAROD OTAL, 1991, p. 381-382).

Excluding this potsherd, there are two clear clusters in Fig. 3, named A and B. The presence in cluster A of clear Phoenician fabrics (samples IBE002 and IBE006, for instance) and the presence in cluster B of clear Iberian amphorae fabrics (samples IBE012 and IBE013, for instance), without mixture, suggest that both clusters point out to different production sources. Cluster A seems to group those samples from the coast, because some of these fabrics are clearly produced there, as cited *supra*. Conversely, cluster B likely groups those indigenous fabrics whose provenance is still unknown. They may be local, because there are some, still unpublished, potter workshops close to the village. There are other possibilities in the Antequera depression, even in the proximities, like the possible Iberian workshop in the "Cerro de la Virgen", Fuente de Piedra (RECIO RUIZ, 2007), whose archaeometrical study is still not carried out.
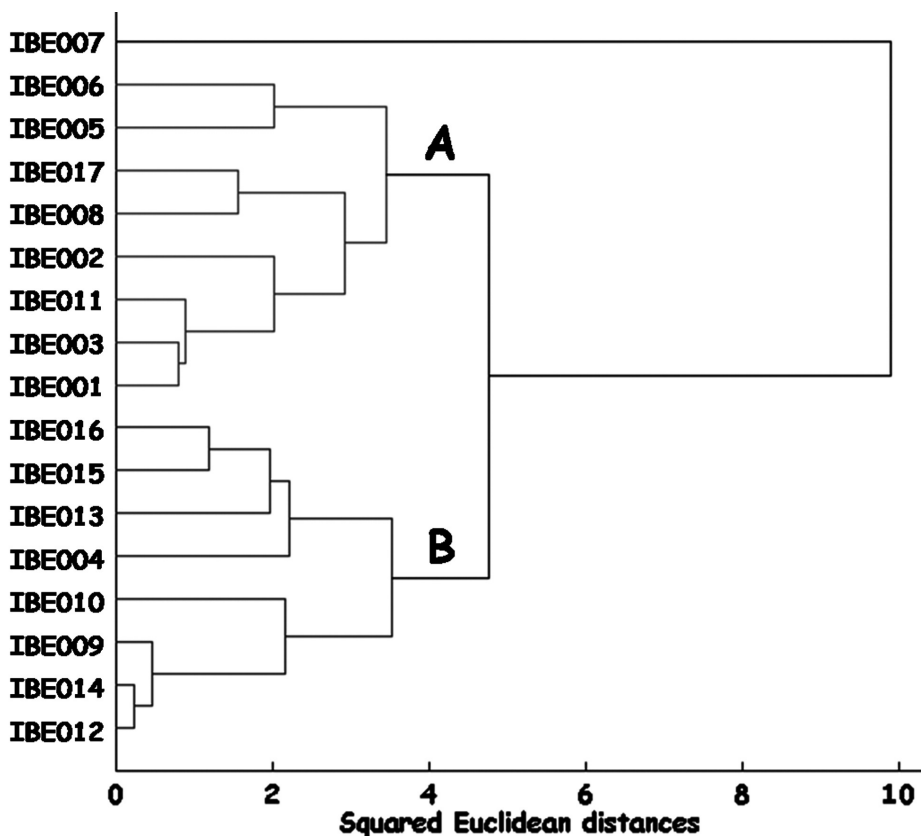

Fig. 3 – Dendrogram of Euclidean distances, average linkage method, calculated using elemental analysis from XRF data.
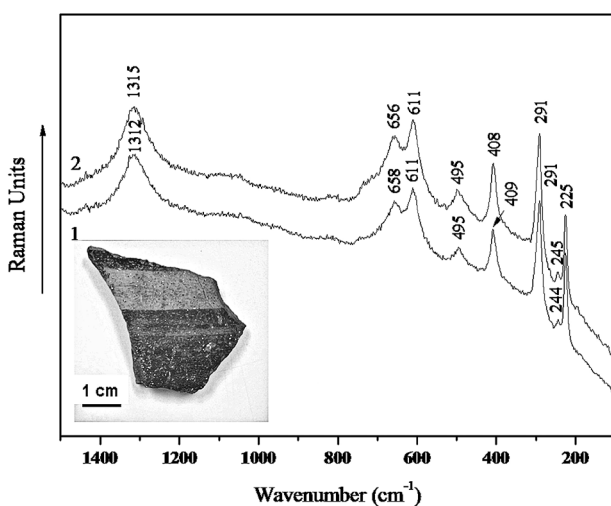
Fig. 4 – Selected Raman spectra of the brownish pigment for IBE014. The inset shows a photograph of the potsherd.
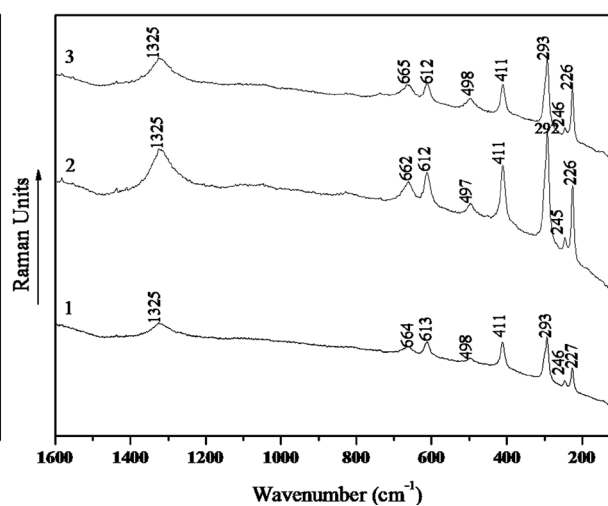
Fig. 5 – Selected Raman spectra of the reddish pigment for IBE014.

It is interesting to point out that, despite its apparent macroscopic similarity in some cases, the Phoenician and Iberian samples are clearly different, both from a mineralogical and chemical points of views. Nevertheless, a well-trained eye may be enough to perform an initial classification.

Furthermore, the Raman spectra collected on various spots through the brown and reddish colored zones of the samples clearly evidenced the use of iron oxide hematite (α-Fe_2O_3) as pigment in all studied specimens (Figs. 4 and 5). Hematite shows characteristic Raman bands at 225 (vs), 245 (m), 293 (vs), 412 (s), 498 (m) and 613 (s). The band at 662 (w) cm^{-1} can be attributed to residual traces of magnetite, Fe_3O_4 (SHEBANOVA & LAZOR, 2003a, 2003b; EL MENDILI *et al.*, 2011) Carbonaceous materials display up to four Raman bands between 1000 and 1800 cm^{-1}, including the graphite band (G-band), located at about 1580 cm^{-1}, and other three Raman features due to different kinds of defects in the graphitic structure (TUINSTRA & KOENIN, 1970). The complete absence in the collected Raman spectra of carbonaceous bands at 1350 and 1580 cm^{-1} indicates that carbon was not used as a pigment in any sample.

5 – CONCLUSIONS

This study allows establishing the following conclusions:

1. The key differences in the mineralogy of the samples are the calcite and gehlenite contents. In general, the mineralogical findings are compatible with firing conditions between 700 and 950 ºC.

2. The chemical data allow establishing the presence of, at least, two sources of pottery, with some other foreign samples. The first source is clearly the Phoenician settlements from the coast of Málaga and their characteristic fabrics. The second source is indigenous pottery, unrelated to the previous one, with a location that it has not been established, yet.

3. Hematite mixtures are the only used pigment. The use of carbonaceous pigments is completely ruled out in the studied samples, even for the blackish paintings.

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