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FIRST ARCHAEOMETRIC STUDY OF THE IBERIAN CERAMIC PRODUCTION FROM TWO SITES IN THE NORTHERN ORETANIA: ALARCOS AND EL CERRO DE LAS CABEZAS (CIUDAD REAL, SPAIN)

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ABSTRACT

The *oppida* at Alarcos and El Cerro de las Cabezas (Ciudad Real, Spain) were two of the most important Iberian sites in the Proto-history (8th-3th centuries BC.) the Northern Oretania. Archaeological studies have been published since the 80s; however this paper is the first that provides archaeometric results from the chemical and mineralogical analysis of 107 ceramic fragments of Iberian common pottery. They have been analysed through X-ray diffraction (XRD) and X-ray fluorescence (XRF). From the chemical results, a statistical study has been carried out, which has allowed us to create reference groups (RG) and to determine the economic relationships between the two sites studied and their areas of influence. At a technological level, the results from the mineralogical analysis have allowed us to estimate the firing temperatures (EFT) and to classify the ceramic fragments into 3 fabrics with their respective temperature ranges.

KEYWORDS: Iberian pottery, Iberian Proto-History, Alarcos, Cerro de las Cabezas, chemical analysis, mineralogical analysis, statistical treatment

1. INTRODUCTION

The current province of Ciudad Real, in the centre of the Iberian Peninsula, is included in the group of territories that were part of the Northern Oretania, a wide region where a large number of settlements from the Iberian Era has been discovered. Two of the most remarkable ones, because of their size and importance within their own territorial area, are the Iberian sites of Alarcos and El Cerro de las Cabezas. Both settlements have been subject of numerous archaeological campaigns since the 80s, which has allowed us to, know better the life of the inhabitants who occupied these hills and controlled in large part the regions of Calatrava and La Mancha respectively (Fig. 1).

Figure 1. Map of the Alarcos and El Cerro de las Cabezas and main Iberian sites in the Northern Oretania (Ciudad Real, Castilla-La Mancha).

The Iberian site of Alarcos (Poblete-Ciudad Real, Ciudad Real) is located only 8 km away from the capital of the province, Ciudad Real, and it occupies a hill that rises 100 m above the valley of the Guadiana River. The first inhabitants took advantage of the raised position that allowed them to defend themselves and to control the ancient communication routes. The choice of this place is not an accident. The Iberians used to build theirs villages on these headlands and fortified them, in a typical construction called an *oppidum*. In Alarcos, the earliest archaeological remains are dated to the late Bronze Age, but the hill was occupied uninterruptedly from the 9th century BC to the 1st c. AD. The place was reused by the Muslims and Christians from the 8th century until its permanent abandonment in the 13th century. Alarcos is also known for being the place were Christian and Muslim troops clashed in the battle of 1195, which entailed the defeat and the abandonment of the castle-palace by the Christian Army (De Juan et al. 1995).

Figure 2. Map of the Iberian Site of Alarcos.

The occupation during the Iberian Age is divided into three different stages, the Ancient and the Middle Iberian period, and a lesser-known one, the Late Iberian period, which coincides with the abandonment of the settlement. During the Ancient Iberian period (6th-5th centuries BC), the *oretani* of Alarcos extended over the whole hill (33ha). The Acropolis was probably placed on top of it, where the medieval castle was built. In this phase, the inhabitants used the quartzitic formations of the northern slope as a natural defence, and they also fortified the southern part. The oldest of the three documented necropolis, which is located in the Iberian Neighbourhood, belongs to this period (Fig. 2) (Fernández and García Huerta 1998).

In the 5th-4th centuries BC (Middle Iberian Age), the site of Alarcos reached its maximum expansion due to population growth. This society developed a hierarchy with appearance of a ruling class that controlled production surpluses and the profits derived from commercial activities. These political and social changes are reflected in the different finds associated with this period: the sanctuary, the great store, the Iberian neighbourhood and the second necropolis (Fig. 2). The sanctuary of Alarcos is located between the main street and the medieval wall. It was occupied from the 5th to 1th century BC, but it was also reused by medieval inhabitants, which has not allowed a complete excavation. Among the most remarkable archaeological objects are 60 ex-votos, some brooches (*fibula*), a small head of the divinity Astarte and ceramics associated with funeral rites (Caballero and Mena 1987). The great store is a rectangular space (400m^2) with a storage capacity of 1200 m^3 to 75.000 kg of grain, where a circular bread oven was discovered. A large number of cereals and beans was also recovered there, that are actually further evidences of the economic, storage and food processing use of this place (García Huerta and Morales 2009; 2011, García Huerta *et al*. 2006; 2020: 58-67) (Fig. 3).

Figure 3. Great Store. Red circle: Bread Oven.

The Iberian neighbourhood is composed of two areas (eastern and western) separated by a main street (40m long-5m wide) that forms a homogenous set. One of the houses, the tripartite building, has been considered as the place from where the ruling classes controlled the surpluses and the commercial activities (Fernández 2009).

Related to this period, a second necropolis was found, where some animal stone sculptures and a celto-italic helmet (5th and 3rd century BC respectively) were recovered (de Juan *et al*. 2004). In 2013, the third necropolis was discovered in the southern part of the hill, beside the Guadiana River. The findings are not published yet.

Alarcos was probably abandoned in the 3rd century BC due to the arrival of the Romans, although the sanctuary area remained in use until the 1th century BC. In fact, other nearby sites such as El Cerro de las Cabezas or El Cerro de las Nieves (Pedro Muñoz, Ciudad Real) were also abandoned in the same period, but in these cases it was because of the clashes between the Carthaginians and the local populations (Morales 2010).

El Cerro de las Cabezas (Valdepeñas, Ciudad Real) is an Iberian *oppidum* located 70 km away from Alarcos on a hill that rises 150 m above the Jabalón River, a tributary of the Guadiana River. It is situated in a strategic position due to the trade routes that communicated the south with the north (Andalusia-northern plateau) and the west with the east (Extremadura-Peninsular East) (Pérez and Vélez 1994; Blánquez 2020). This site was also occupied during the later Bronze Age and remained populated until 225 BC when it was destroyed by the Punic troops commanded by Anibal (Vélez and Pérez 1996). In the later Bronze Age-Early Iron Age the flattest and lowest areas were populated. The archaeological materials recovered from this period, especially ceramics, show the relationships between El Cerro de las Cabezas and the Urnfield Culture and also with Tartessos (Esteban *et al.* 2003).

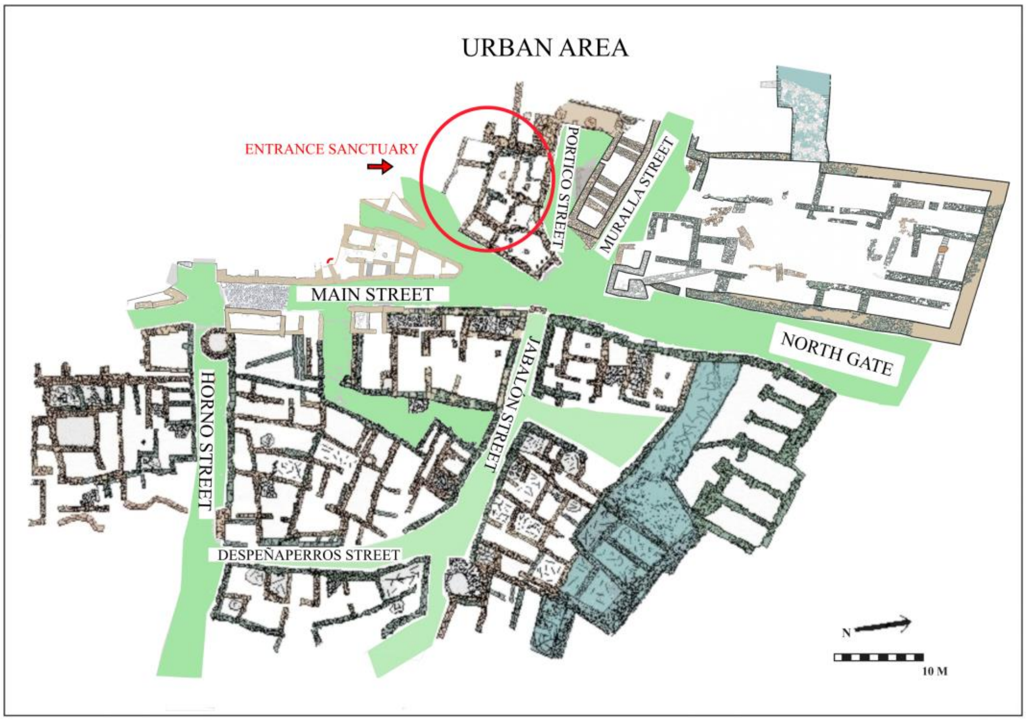

Figure 4. Urban Area of the Iberian site of El Cerro de las Cabezas. Black circles= potteries kilns.

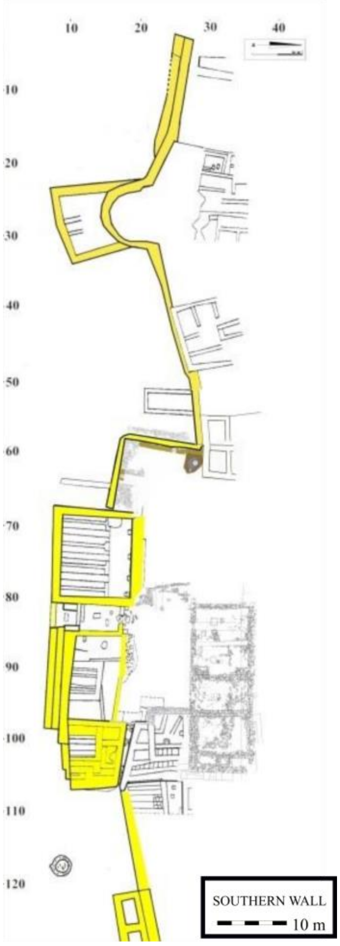

Figure 5. Southern Wall with bastions and casemates.

The Iberian period (6th to 3rd centuries BC) is divided into three phases in which the distribution of the village gradually changed according to economic and social transformations (Pérez and Vélez 1994). During these four centuries, the urban area was mainly situated on the southern slope, using a small part of the 14 ha of the site. The northern and the eastern slopes were used as natural defences.

In the initial phase (6th to 5th centuries BC) the following facts should be highlighted: the reuse of earlier ancient buildings that became larger, the elaboration of the first wheel-made ceramics, and specially the construction of the defensive walls that surrounded a large part of the hill. The acropolis was built at the highest point in the 4th century BC. It was a heptagonal enclosure from where the ruling class would exercise economic and political control (Vélez and Pérez 2000; Vélez *et al.* 2004; Torres *et al.* 2014).

The urban planning of the village started in the second phase (5th to 4th centuries BC) from the Main Street (4m wide) that distributed the space in 4 different neighbourhoods and from whence it gave rise to other secondary streets. In Fig. 4, the predominance of the rectangular room is observed; some of them are identified as workshops, bread ovens, and ceramic-metallic kilns. During this phase, the walls reached the maximum perimeter (1750 m), and they were reinforced using a defence system called cyclopean masonry, formed by a double wall and other defensive elements such as casemates, posterns and bastions (Fig. 5) (Torres *et al.* in press). The first Attic ceramics appeared at the end of 6th or the beginning of 5th century BC, used by archaeologists as an index fossil, coinciding with the disappearance of the autochthonous hand-made ceramics (Madrigal 2020).

In the last phase (3rd century BC) a social growth is determined from the extension of the village to the southwest, although a great number of rooms remained occupied. The Social and economic growth is also reflected in the creation of new kilns, workshops and in the specialization of labour. Regarding to religiosity, it is noteworthy that there were three sanctuaries all used during the 3rd century BC. The most remarkable is an *entrance sanctuary* located on the main street, which was used as a religious centre, but also as a place to control the access to the village (Fig. 4) (Moneo *et al.* 2001; Vélez and Pérez 2010).

The site of El Cerro de las Cabezas, one of the most important and populated sites in the Iberian period, was sharply and definitively abandoned in the 3rd century BC.

All the ceramic fragments analysed in this work are classified as Iberian Common Pottery and abbreviated in Spanish as IBC (*Ibérica común*). This pottery is characterised by its purified earthenware body, a high quality ceramic worked on the potter's wheel with pictorial decoration (Mata and Bonet 1992). The process to produce this ceramic could be summarized in the following way. The first step was the selection and extraction of the clays from ponds[1]. Then, the clays were decanted to remove the largest inclusions. Thus, the potters obtained a refined clay body without colourations. Before firing, the pieces were modelled during wheel-throwing and after that, they could receive a slip coating called an engobe (a clay-water mixture) or they could be polished to obtain a smooth and brilliant surface.

Once dried, the pieces were decorated with pictorial and geometrical motifs (lines, concentric and semi concentric circles, bends, etc.) that evolved into a more sophisticated decoration in the last centuries; like human and animal figures, floral and epigraphic motifs, that led to the professionalization of the potters (Taradell and Sanmartí 1980). This sophisticated decoration may indicate the professionalization of the iberan potters. may be an evidence for The colour palette employed in decoration is rather limited, predominantly reds (vinous and matte), beiges, oranges and browns, coming from different mineral oxides. Another type of decoration is the stamp, which was elaborated when the pieces were not entirely dry (leather-hard). The site of El Cerro de las Cabezas is considered to be one of the most important centres of stamped pottery production in the 3rd century BC (Fernández Maroto *et al.* 2007). Afterwards, the pieces were fired (single-firing) in an oxidizing atmosphere, reaching high temperatures, from 800ºC to 1000ºC (Antón 1973; Rincón 1985; Buxeda and Madrid 2003; Tsantini 2007; Cultrone *et al.* 2011, Compaña *et al.* 2012; Rodríguez Corral, 2017; Liritzis *et al.,* 2020).

The aim of this work is to archaeometrically characterize 107 ceramic fragments of the Iberian Common Pottery (IBC) from Alarcos and El Cerro de las Cabezas, in order to establish reference groups and especially their provenance. The provenance concept is defined by Picon (1973), Weigand *et al.* (1977), and is used by other authors like Harbottle (1982), Hein *et al.* (2002) or Buxeda (2001). In summary, provenance studies are carried out to determine the origin of the raw materials used to produce the ceramics.

In the historical context in which our study is based, Northern Oretania in the 5th-3rd centuries BC, the commercial and economic relationships between the settlements that occupied this territory with other regions in the Iberian Peninsula have been deeply researched by Morales (2010) and García Huerta and Morales (2011). In both Alarcos and El Cerro de las Cabezas a large number of archaeological materials, especially ceramics have been found, which came from contacts with the Greeks and Phoenicians, whose presence in the Iberian Peninsula was even before the 5th century BC. On the basis of the chemical results and the subsequent provenance study, it will be possible to go deeply into these commercial, cultural and economic relationships.

2. MATERIALS AND METHODS

In this work 107 sherds have been analysed. These include 53 fragments from Alarcos, 37 of them recovered from the great store and 16 from two Iberian rooms located next to it. Another 54 sherds (50 IBC and 4 amphorae) coming from different parts of El Cerro de las Cabezas have been analysed (Figs. 4-5). Most of the analysed samples have been archaeologically or artistically dated between the 5th- 4th centuries BC.

The chemical and mineralogical characterisation has been performed using X-ray fluorescence and X-ray diffraction, which are two of the most used techniques in this kind of work (Peña-Pozas *et al.* 2011). For sample preparation, the following process has been carried out: mechanical removal of the pictorial decoration using an IsoMet® 100 Buehler cutting saw,

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[1] Currently, it is unknown for us the exact location from where they obtained the raw materials, but both sites are located close to rivers with deposits of clay.

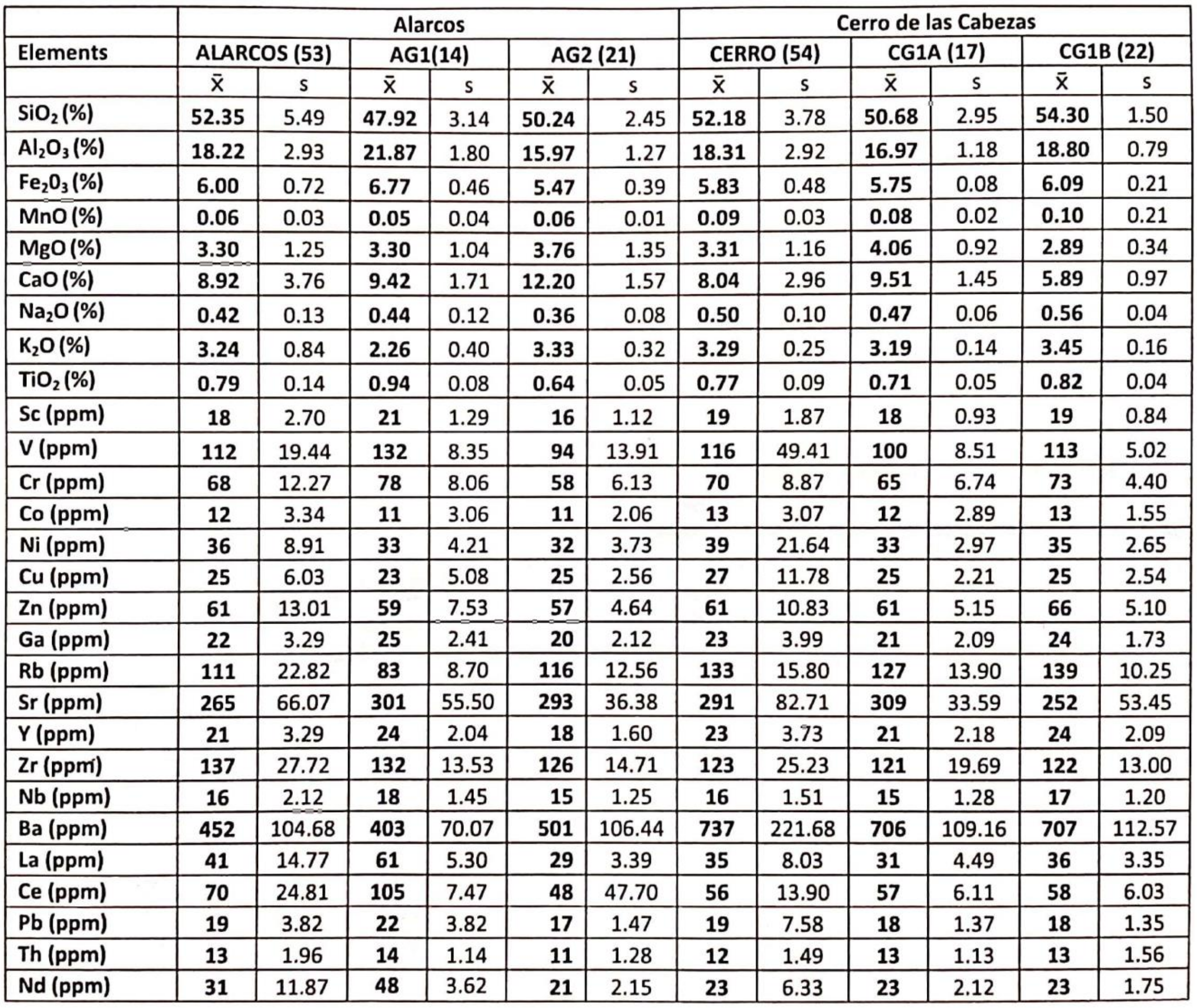
ultrasonic cleaning, drying in ovens at 100°C, grinding and homogenizing in agate mills. For each ceramic fragment, 10 g has been used.

The mineralogical analysis has been performed using X-ray diffraction (XRD), working with powdered samples (about 1 g) below 50 µm. The equipment used to determine the mineral phases is a Philips PW-1710 diffractometer with an automatic divergence slit and graphite monochromator, applying the copper Ka radiation wavelength (λ = 1.5405). The interpretation of results has been carried out using the X'Pert Highscore 3.0 software.

The chemical analysis has been obtained through X-ray fluorescence (XRF) using a MagiX Super Q Version 3.0 X-ray fluorescence spectrometer (Philips). The trace elements have been analysed using pressed pellets (8 g). To obtain the major and minor elements, it was necessary to make glassy pills using 0.5 g of powdered sample with 5 g of lithium tetraborate. The equipment used to fuse the mixture is a semi-automatic Philips Perl'x 3 fuser. The loss on ignition has been determined by calcination of the samples at 1100°C for 5h (for calibration standards see **APPENDIX** Tables A1 and A2).

The elements identified by XRF are Al_2O_3, Fe_2O_3, SiO_2, MgO, MnO, TiO_2, K_2O, CaO, Na_2O, Sc, V, Cr, Co, Ni, Cu, Zn, Ga, Rb, Sr, Y, Zr, Nb, Ba, La, Ce, Pb, Th and Nd. The average and standard deviation of the chemical elements used in our study are presented in Table 1. The statistical analysis has been performed on the chemical results using the free R Software for statistical computing and graphics.

Table 1 Chemical results for Alarcos and El Cerro de las Cabezas 107 ceramics with average and standard deviation for the chemical groups obtained through the statistical analysis. Major and minor elements in wt.%; trace elements in ppm (parts per million). $\bar{x}$=average, s= standard deviation.



	Alarcos						Cerro de las Cabezas					
Elements	ALARCOS (53)		AG1(14)		AG2 (21)		CERRO (54)		CG1A (17)		CG1B (22)	
	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s
SiO_2 (%)	**52.35**	5.49	**47.92**	3.14	**50.24**	2.45	**52.18**	3.78	**50.68**	2.95	**54.30**	1.50
Al_2O_3 (%)	**18.22**	2.93	**21.87**	1.80	**15.97**	1.27	**18.31**	2.92	**16.97**	1.18	**18.80**	0.79
Fe_2O_3 (%)	**6.00**	0.72	**6.77**	0.46	**5.47**	0.39	**5.83**	0.48	**5.75**	0.08	**6.09**	0.21
MnO (%)	**0.06**	0.03	**0.05**	0.04	**0.06**	0.01	**0.09**	0.03	**0.08**	0.02	**0.10**	0.21
MgO (%)	**3.30**	1.25	**3.30**	1.04	**3.76**	1.35	**3.31**	1.16	**4.06**	0.92	**2.89**	0.34
CaO (%)	**8.92**	3.76	**9.42**	1.71	**12.20**	1.57	**8.04**	2.96	**9.51**	1.45	**5.89**	0.97
Na_2O (%)	**0.42**	0.13	**0.44**	0.12	**0.36**	0.08	**0.50**	0.10	**0.47**	0.06	**0.56**	0.04
K_2O (%)	**3.24**	0.84	**2.26**	0.40	**3.33**	0.32	**3.29**	0.25	**3.19**	0.14	**3.45**	0.16
TiO_2 (%)	**0.79**	0.14	**0.94**	0.08	**0.64**	0.05	**0.77**	0.09	**0.71**	0.05	**0.82**	0.04
Sc (ppm)	**18**	2.70	**21**	1.29	**16**	1.12	**19**	1.87	**18**	0.93	**19**	0.84
V (ppm)	**112**	19.44	**132**	8.35	**94**	13.91	**116**	49.41	**100**	8.51	**113**	5.02
Cr (ppm)	**68**	12.27	**78**	8.06	**58**	6.13	**70**	8.87	**65**	6.74	**73**	4.40
Co (ppm)	**12**	3.34	**11**	3.06	**11**	2.06	**13**	3.07	**12**	2.89	**13**	1.55
Ni (ppm)	**36**	8.91	**33**	4.21	**32**	3.73	**39**	21.64	**33**	2.97	**35**	2.65
Cu (ppm)	**25**	6.03	**23**	5.08	**25**	2.56	**27**	11.78	**25**	2.21	**25**	2.54
Zn (ppm)	**61**	13.01	**59**	7.53	**57**	4.64	**61**	10.83	**61**	5.15	**66**	5.10
Ga (ppm)	**22**	3.29	**25**	2.41	**20**	2.12	**23**	3.99	**21**	2.09	**24**	1.73
Rb (ppm)	**111**	22.82	**83**	8.70	**116**	12.56	**133**	15.80	**127**	13.90	**139**	10.25
Sr (ppm)	**265**	66.07	**301**	55.50	**293**	36.38	**291**	82.71	**309**	33.59	**252**	53.45
Y (ppm)	**21**	3.29	**24**	2.04	**18**	1.60	**23**	3.73	**21**	2.18	**24**	2.09
Zr (ppm)	**137**	27.72	**132**	13.53	**126**	14.71	**123**	25.23	**121**	19.69	**122**	13.00
Nb (ppm)	**16**	2.12	**18**	1.45	**15**	1.25	**16**	1.51	**15**	1.28	**17**	1.20
Ba (ppm)	**452**	104.68	**403**	70.07	**501**	106.44	**737**	221.68	**706**	109.16	**707**	112.57
La (ppm)	**41**	14.77	**61**	5.30	**29**	3.39	**35**	8.03	**31**	4.49	**36**	3.35
Ce (ppm)	**70**	24.81	**105**	7.47	**48**	47.70	**56**	13.90	**57**	6.11	**58**	6.03
Pb (ppm)	**19**	3.82	**22**	3.82	**17**	1.47	**19**	7.58	**18**	1.37	**18**	1.35
Th (ppm)	**13**	1.96	**14**	1.14	**11**	1.28	**12**	1.49	**13**	1.13	**13**	1.56
Nd (ppm)	**31**	11.87	**48**	3.62	**21**	2.15	**23**	6.33	**23**	2.12	**23**	1.75

3. RESULTS

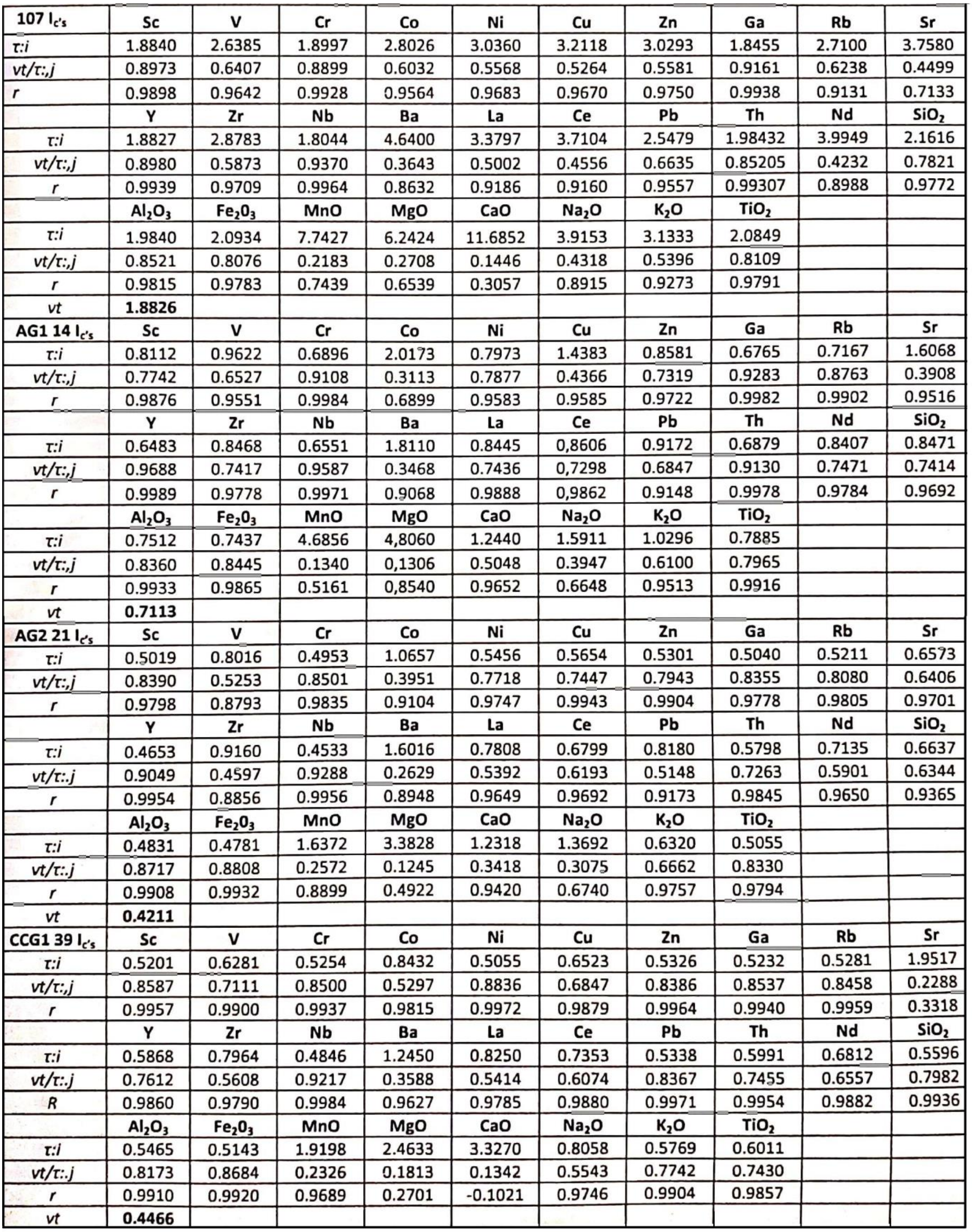
3.1. Chemical and statistical analysis

The first estimates performed from the chemical analysis have allowed us to determine the existence of different groups in both sites, indicating that there is a heterogeneous production. In order to confirm these preliminary results, it is necessary to use statistical techniques to process the chemical composition and to establish the different reference groups.

For the statistical treatment the 28 chemical elements shown in Table 1 have been used. In this table they are expressed as the mean ($\bar{x}$) and standard deviation (s) of the total of the individual ceramics ($I_{c's}$) to Alarcos and El Cerro de las Cabezas together within the 3 - 4 chemical groups determined from the statistical analysis: AG1 and AG2 to Alarcos, and CG1 (CG1A and CG1B) to El Cerro de las Cabezas. The elements W and Sn have been removed from the statistical treatment because of the regression limits of the FRX equipment. There are also some elements which could have been altered during burial process like P_2O_5 (Lemoine and Picon 1982) which has been also removed, and CaO (Buxeda and Cau 1995), MnO (Picon 1985) or MgO.

In order to compare the variability of the analytical corpus, the compositional variation matrix (CVM) defined by Aitchison (1986) or Buxeda and Kilikoglou (2003) has been calculated. This matrix includes the total variation (vt), a value that quantifies and summarizes the variability of the chemical composition. The value used theoretically to define a chemical composition as heterogeneous is around 0.3. If the vt is above this value, the chemical dataset should be considered as polygenic and not monogenic. A polygenic set could mean that a part of the $I_{c's}$ analysed has a different provenance. However, to reach this conclusion, the CVM must be treated before as Aitchinson proposed (1981; 1982). He observed the existence of the spurious relationships between the different variables, i.e. the inexistent relation between them. The chemical composition provides relative information, not an absolute; therefore it is necessary to make a logratio transformation. This solution has also been extended by Aitchinson (1986) and by Buxeda (1999), who applied this formula to ceramic studies, using the additive logratio transformation (ALR) or the centred logratio transformation (CLR). The ALR transformation used in this work and developed by Guirao *et al.* (2014), implies the use as a divisor element, the one which provides the lowest chemical variability. This value is also obtained from CVM (Table 2). In this table are briefly expressed the most important values: the τ.i, the vt/τ.i, the r and the vt for the whole data set (107 $I_{c's}$), and for the different groups defined from both sites. The simbol *vt* is the total variation and *τ.i* is a greek Word (tau). τ.i is the sum of the values of each column of the MVC; It refers to the origin of compositional variation. In addition, these values can be given in relation to the value of the total variation (vt / τ.i), the lower this value, the greater the variation imposed by the element in question.

Table 2 The compositional relative variation of AG1, AG2 and CCG1 from Alarcos and Cerro de las Cabezas (107 ceramics)



107 $I_{c's}$	**Sc**	**V**	**Cr**	**Co**	**Ni**	**Cu**	**Zn**	**Ga**	**Rb**	**Sr**
τ:i	1.8840	2.6385	1.8997	2.8026	3.0360	3.2118	3.0293	1.8455	2.7100	3.7580
vt/τ:,j	0.8973	0.6407	0.8899	0.6032	0.5568	0.5264	0.5581	0.9161	0.6238	0.4499
r	0.9898	0.9642	0.9928	0.9564	0.9683	0.9670	0.9750	0.9938	0.9131	0.7133
	Y	**Zr**	**Nb**	**Ba**	**La**	**Ce**	**Pb**	**Th**	**Nd**	**SiO_2**
τ:i	1.8827	2.8783	1.8044	4.6400	3.3797	3.7104	2.5479	1.98432	3.9949	2.1616
vt/τ:,j	0.8980	0.5873	0.9370	0.3643	0.5002	0.4556	0.6635	0.85205	0.4232	0.7821
r	0.9939	0.9709	0.9964	0.8632	0.9186	0.9160	0.9557	0.99307	0.8988	0.9772
	Al_2O_3	**Fe_2O_3**	**MnO**	**MgO**	**CaO**	**Na_2O**	**K_2O**	**TiO_2**		
τ:i	1.9840	2.0934	7.7427	6.2424	11.6852	3.9153	3.1333	2.0849		
vt/τ:,j	0.8521	0.8076	0.2183	0.2708	0.1446	0.4318	0.5396	0.8109		
r	0.9815	0.9783	0.7439	0.6539	0.3057	0.8915	0.9273	0.9791		
vt	**1.8826**									
AG1 14 $I_{c's}$	**Sc**	**V**	**Cr**	**Co**	**Ni**	**Cu**	**Zn**	**Ga**	**Rb**	**Sr**
τ:i	0.8112	0.9622	0.6896	2.0173	0.7973	1.4383	0.8581	0.6765	0.7167	1.6068
vt/τ:,j	0.7742	0.6527	0.9108	0.3113	0.7877	0.4366	0.7319	0.9283	0.8763	0.3908
r	0.9876	0.9551	0.9984	0.6899	0.9583	0.9585	0.9722	0.9982	0.9902	0.9516
	Y	**Zr**	**Nb**	**Ba**	**La**	**Ce**	**Pb**	**Th**	**Nd**	**SiO_2**
τ:i	0.6483	0.8468	0.6551	1.8110	0.8445	0,8606	0.9172	0.6879	0.8407	0.8471
vt/τ:,j	0.9688	0.7417	0.9587	0.3468	0.7436	0,7298	0.6847	0.9130	0.7471	0.7414
r	0.9989	0.9778	0.9971	0.9068	0.9888	0,9862	0.9148	0.9978	0.9784	0.9692
	Al_2O_3	**Fe_2O_3**	**MnO**	**MgO**	**CaO**	**Na_2O**	**K_2O**	**TiO_2**		
τ:i	0.7512	0.7437	4.6856	4,8060	1.2440	1.5911	1.0296	0.7885		
vt/τ:,j	0.8360	0.8445	0.1340	0,1306	0.5048	0.3947	0.6100	0.7965		
r	0.9933	0.9865	0.5161	0,8540	0.9652	0.6648	0.9513	0.9916		
vt	**0.7113**									
AG2 21 $I_{c's}$	**Sc**	**V**	**Cr**	**Co**	**Ni**	**Cu**	**Zn**	**Ga**	**Rb**	**Sr**
τ:i	0.5019	0.8016	0.4953	1.0657	0.5456	0.5654	0.5301	0.5040	0.5211	0.6573
vt/τ:,j	0.8390	0.5253	0.8501	0.3951	0.7718	0.7447	0.7943	0.8355	0.8080	0.6406
r	0.9798	0.8793	0.9835	0.9104	0.9747	0.9943	0.9904	0.9778	0.9805	0.9701
	Y	**Zr**	**Nb**	**Ba**	**La**	**Ce**	**Pb**	**Th**	**Nd**	**SiO_2**
τ:i	0.4653	0.9160	0.4533	1.6016	0.7808	0.6799	0.8180	0.5798	0.7135	0.6637
vt/τ:.j	0.9049	0.4597	0.9288	0.2629	0.5392	0.6193	0.5148	0.7263	0.5901	0.6344
r	0.9954	0.8856	0.9956	0.8948	0.9649	0.9692	0.9173	0.9845	0.9650	0.9365
	Al_2O_3	**Fe_2O_3**	**MnO**	**MgO**	**CaO**	**Na_2O**	**K_2O**	**TiO_2**		
τ:i	0.4831	0.4781	1.6372	3.3828	1.2318	1.3692	0.6320	0.5055		
vt/τ:.j	0.8717	0.8808	0.2572	0.1245	0.3418	0.3075	0.6662	0.8330		
r	0.9908	0.9932	0.8899	0.4922	0.9420	0.6740	0.9757	0.9794		
vt	**0.4211**									
CCG1 39 $I_{c's}$	**Sc**	**V**	**Cr**	**Co**	**Ni**	**Cu**	**Zn**	**Ga**	**Rb**	**Sr**
τ:i	0.5201	0.6281	0.5254	0.8432	0.5055	0.6523	0.5326	0.5232	0.5281	1.9517
vt/τ:,j	0.8587	0.7111	0.8500	0.5297	0.8836	0.6847	0.8386	0.8537	0.8458	0.2288
r	0.9957	0.9900	0.9937	0.9815	0.9972	0.9879	0.9964	0.9940	0.9959	0.3318
	Y	**Zr**	**Nb**	**Ba**	**La**	**Ce**	**Pb**	**Th**	**Nd**	**SiO_2**
τ:i	0.5868	0.7964	0.4846	1.2450	0.8250	0.7353	0.5338	0.5991	0.6812	0.5596
vt/τ:.j	0.7612	0.5608	0.9217	0.3588	0.5414	0.6074	0.8367	0.7455	0.6557	0.7982
R	0.9860	0.9790	0.9984	0.9627	0.9785	0.9880	0.9971	0.9954	0.9882	0.9936
	Al_2O_3	**Fe_2O_3**	**MnO**	**MgO**	**CaO**	**Na_2O**	**K_2O**	**TiO_2**		
τ:i	0.5465	0.5143	1.9198	2.4633	3.3270	0.8058	0.5769	0.6011		
vt/τ:.j	0.8173	0.8684	0.2326	0.1813	0.1342	0.5543	0.7742	0.7430		
r	0.9910	0.9920	0.9689	0.2701	-0.1021	0.9746	0.9904	0.9857		
vt	**0.4466**									

3.1.1. Alarcos

The results from Alarcos (53 $I_{c's}$) show a high chemical variability due mainly to CaO, MnO and MgO, as shown in the element uniform chart (Fig. 6) and also in the vt, being 1.9198. The most plausible explanation is a multiple provenance (different sites or different source-areas). From the chemical and statistical analysis two groups have been defined: AG1 and AG2 (Fig. 7).

AG1 is formed by 14 samples recovered from two rooms outside the great store. This group has a vt of 0.7113, due mainly to the high variability of MgO ($\tau.i$= 4.8060; $vt/\tau.i$=0.1306) and MnO ($\tau.i$=4.6856; $vt/\tau.i$=0.1340). The element with least variability, Y ($\tau.i$= 0.6483; $vt/\tau.i$=0.9688) is used as the divisor element in the logratio transformation. The variability of MgO and MnO is due to the disparate values (MgO, A52=1.42%; A41=5.37%; MnO, A42=0.02%; A38=0.08%). In the case of Na_2O, it could be related to an alteration process during burial (Buxeda 1999). These 14 samples have been initially considered as local products although their chemical compositions are not comparable with the other chemical group of Alarcos.

AG2, composed of 21 $I_{c's}$ has a vt of 0.4211. This value is slightly higher than the theoretical 0.3 mentioned above but it should be still considered as a monogenic production. The variability is mainly due to MgO ($\tau.i$= 3.3828; $vt/\tau.i$=0.1245) – according to Table 2 $vt/\tau.i$=0.1245 and its consequently high standard deviation (*s*=1.38). The other elements are again MnO ($\tau.i$= 1.6372; $vt/\tau.i$=0.2572) - according to Table 2 $\tau.i$= 1.6372; $vt/\tau.i$=0.2572, Ba ($\tau.i$= 1.6016; $vt/\tau.i$=0.2629) - according to Table 2 $vt/\tau.i$=0.2629 and Na_2O ($\tau.i$= 1.3692; $vt/\tau.i$=0.3075) - according to Table 2 $\tau.i$= 1.3692 and in lesser extent CaO, Co and Zr.

In the Fig. 7 there are 11 $I_{c's}$ (A6, A15, A16, A28, A45, A32, A48, A33, A8, A21 and A29) that remain outside the two groups defined, so it could mean that they have another provenance. Samples A32 and A33 are fragments of handles and they present a different composition visible to the naked eye, therefore both samples could have been made in Alarcos although their chemical compositions are quite different. There is a small non-calcareous group (A13, A18 and A30) and others 5 $I_{c's}$ (A19, A26, A23 and A36) that are situated near to AG2. Therefore, 18 of the 53 $I_{c's}$ analysed remain unclassified.

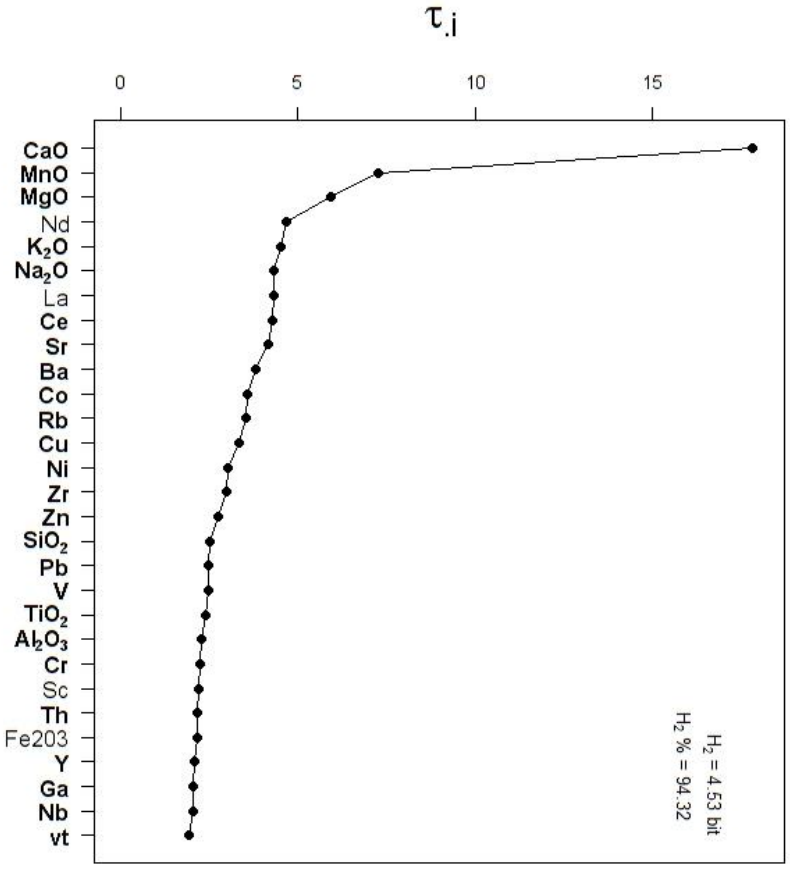


Figure 6. ***Uniform Chart of the 53 samples of Alarcos: the y-axis corresponds to τ.i and the x-axis to the 28 studied elements.***

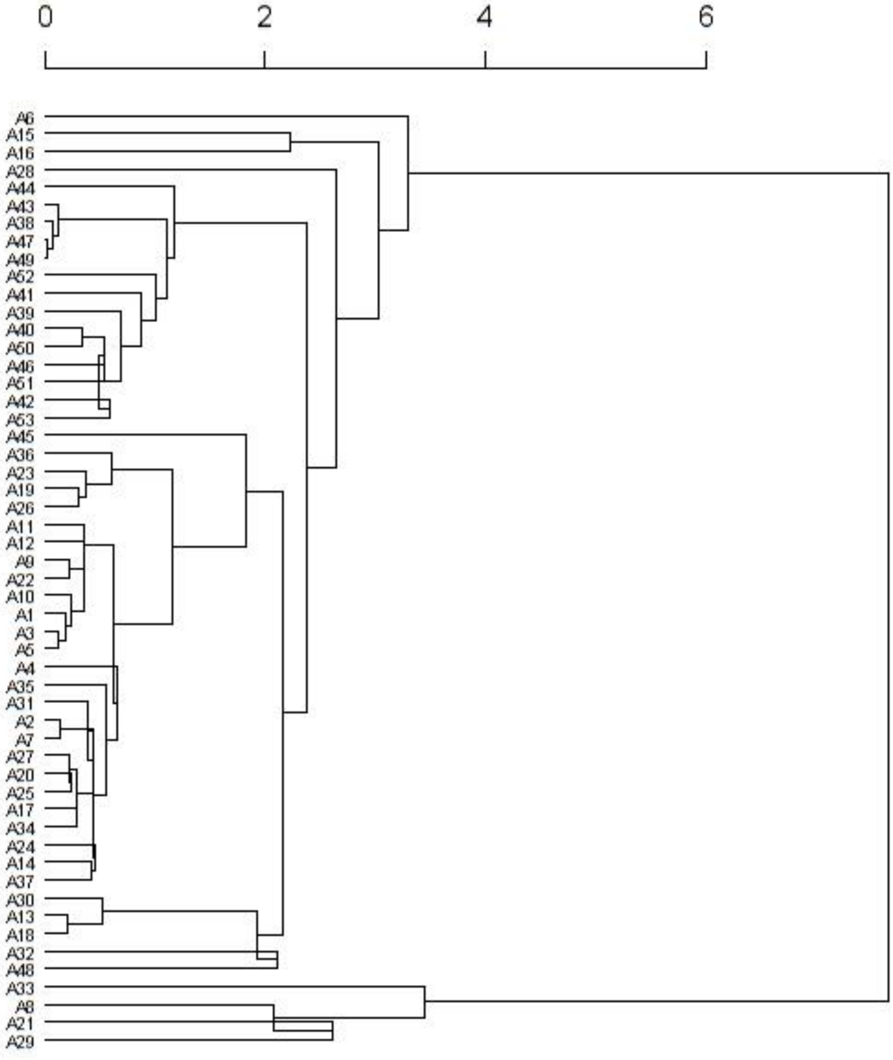


Figure 7. Dendrogram of Alarcos showing the main groups. The method of grouping used is by centroid links and the squared Euclidean distance.

There are some plausible explanations to interpret the differences between AG1 and AG2. From the chemical results it can be affirmed that AG2 is the reference group (RG) of Alarcos, but it is not so evident in the case of AG1 and this group could have another provenance. Another option would be the use of other raw materials richer in aluminium and with lower levels of SiO_2 and K_2O. In order to demonstrate this option, it would be interesting to identify the source area(s) from where the raw materials were recollected in Alarcos.

3.1.2. Cerro de las Cabezas

The calculations based on the 54 sherds show a polygenic sampling, with a vt of 1.2081. The variability is due to the MnO, MgO, CaO, Sr, Zn and Na_2O (Fig. 8). However, in Fig. 9 a large cluster can be observed, CG1, formed by a great number of individuals (39 - 54). This CG1 has been subdivided into two groups, CG1A and CG1B composed for 17 and 22 $I_{c's}$ respectively, in order to analyse their own particularities.

CG1 is characterised by a 0.4466 vt, whose greater variability is due again to the CaO (*τ.i*= 3.3269; *vt/τ.i*=0.1342) - according to Table 2 *τ.i*=3.3270, the MgO (*τ.i*= 2.4632; *vt/τ.i*=0.1813) - according to Table 2 *τ.i*=2.4633, the MnO (*τ.i*= 1.919; *vt/τ.i*=0.2326) - according to Table 2 *τ.i*=1.9198, and the Sr (*τ.i*= 1.9517; *vt/τ.i*=0.2288), being the element with less variability. The CaO is in fact the main element that separates CG1 into two subgroups, CG1A being the most calcareous (Table 1).

The vt for CG1A and CG1B are 0.3484 and 0.2972 respectively. Therefore, both groups can be classified as monogenic and the differences between them are not sufficient to consider that these two subgroups have a different origin. In fact, exist a small group formed by CC50-CC38-CC28-CC29 and CC49 could also be part of the CG1. These 5 $I_{c's}$ are mainly conditioned by their high levels of CaO and low levels of SiO_2 and TiO_2 compared to the average of group CG1.

Individuals CC20, CC26 and CC27 compose a little group but with considerable chemical differences because of their low levels of SiO_2 and their high levels of Al_2O_3. The MgO levels of these three samples (around 1%) are far below the average of MgO in El Cerro de las Cabezas ($\bar{x}$=3.64%). In fact, these ceramic fragments have their own mineralogical composition which will be discussed in the next section.

Individuals CC32, CC8, CC48, CC16, CC32, CC9, CC44 and CC45 are isolated, maybe because they have a different provenance. The amphorae analysed (ANF1-4) are grouped in CG1, so it is deduced that they were made in el Cerro de las Cabezas.

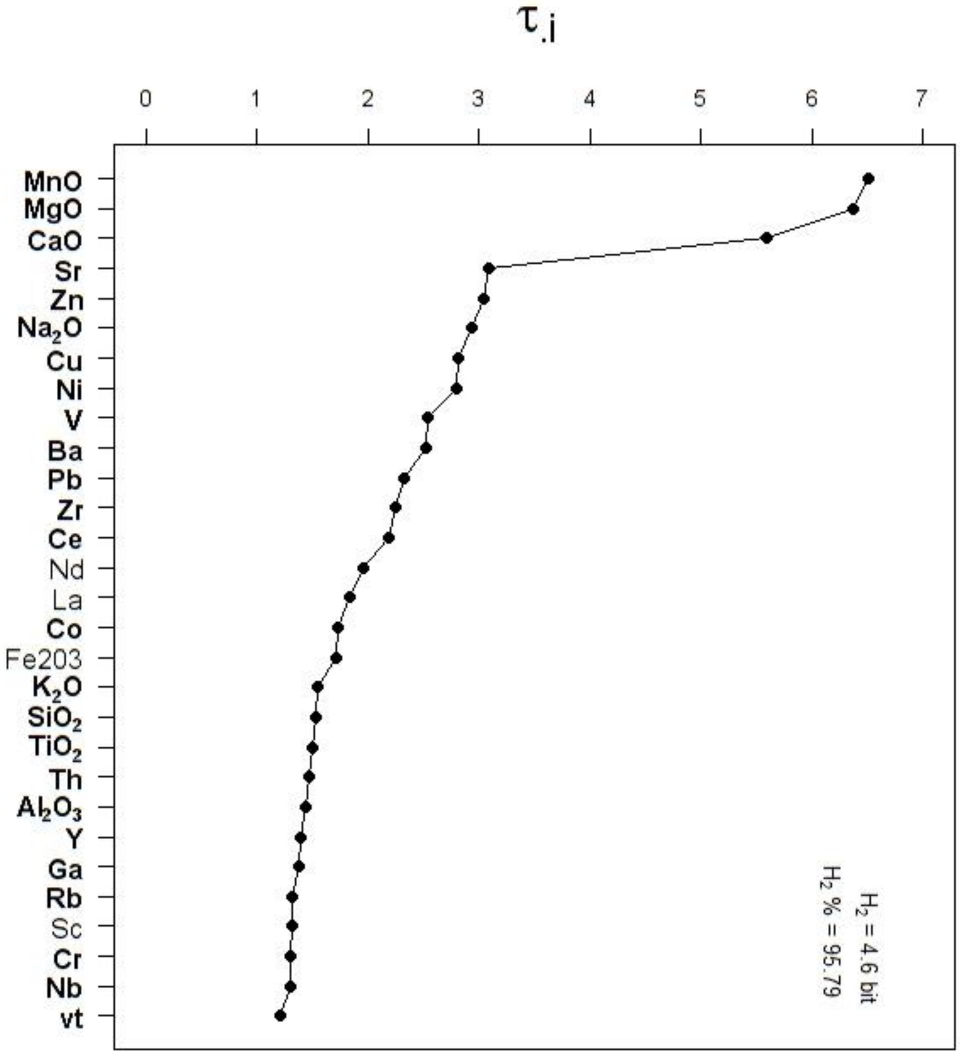


Figure 8. Uniform Chart of El Cerro de las Cabezas: the y-axis corresponds to τ.i and the x-axis to the 28 studied elements.

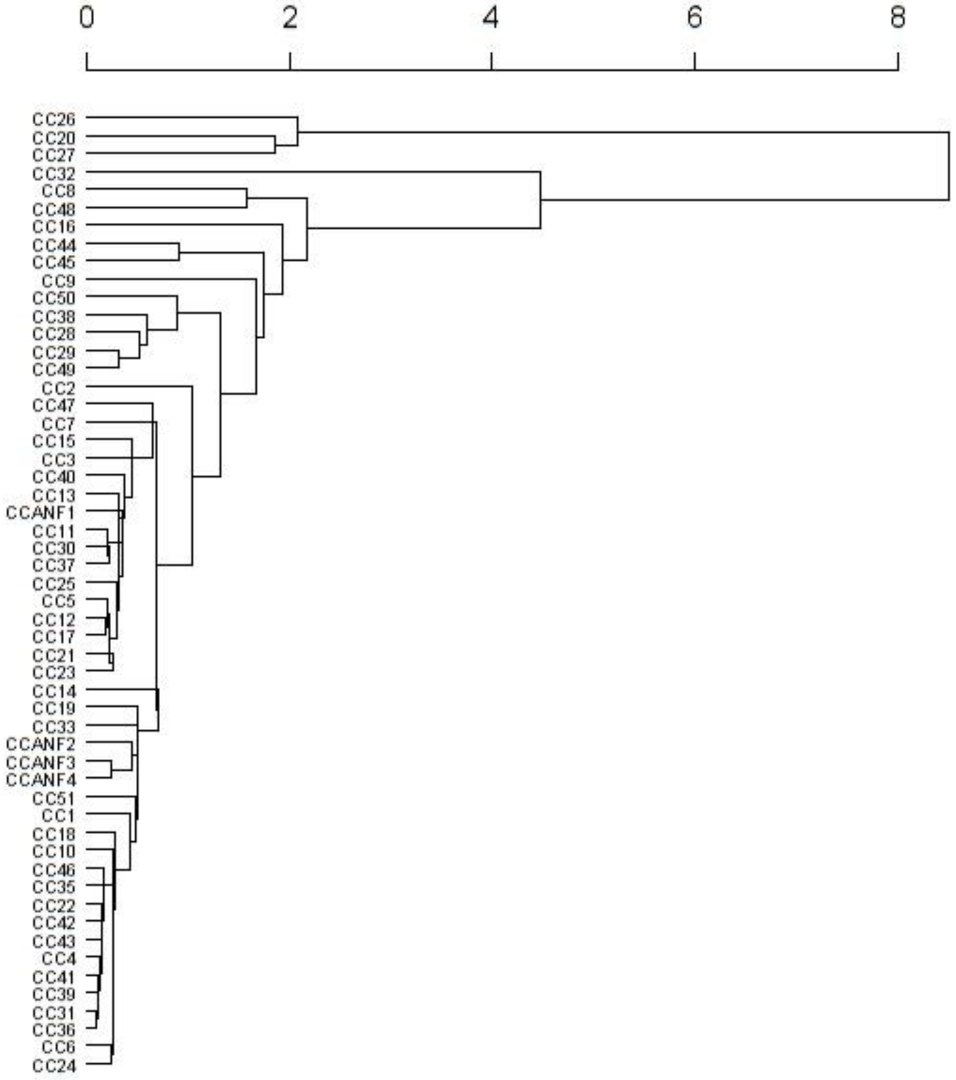


Figure 9. Dendrogram of El Cerro de las Cabezas showing the main groups.

3.1.3. Alarcos and Cerro de las Cabezas

In Table 2 are presented the values from the CVM which indicate a vt of 1.8826, the elements with greater variability being CaO, MnO, MgO, Ba, Nd and Na_2O. Nb is used in the logarithmic transformation. The cluster analysis presented in Fig. 10 is calculated using the 28 elements used thus far. However, the same dataset has been treated by removing certain chemical elements like MgO, MnO, even using only trace elements. MgO and MnO are two elements that could have been altered during burial, and together with CaO, they have always been the elements which have provided the greatest variability in the dataset. It is impossible to present each of the calculations performed. Therefore, the interpretation that follows is actually a summary of all these. The clusters repeated in all the calculations and also the new possible groupings have been taken into consideration.

3.1.4 Classified Samples from Alarcos: AGs and CC/CCG(s) from El Cerro de las Cabezas

- **AG1** is formed again by the same 14 samples from Alarcos recovered from two rooms next to the great store. Sample A48 was left out of this group in Fig. 7 and also in Fig. 10, but probably belongs to AG1.
- **AG2** was presented as a group composed of 21 $I_{c's}$ which can be subdivided into two small groups (Fig.7). This first group (8 - 21), is well defined in those published calculations and also in those that are not (A1, A3, A5, A9, A10, A11, A12,

A22). A35 should be also included to AG2. The other 12 $I_{c's}$ are somehow linked (A2, A4, A7, A14, A17, A20, A24, A25, A27, A31, A34, A37) but in this poorly defined cluster are also some samples coming from CG1A from El Cerro de las Cabezas (CC3, CC7, CC11, CC15, CC30, CC37, CC40 and CCANF1). Thus, if an euclidean distance higher than 1.5 is taken as a reference, this group becomes a larger cluster with non-provenance defined. However, in calculations removing MgO and MnO, this subgroup of Alarcos remains fully bonded, and it only confirms the joining of CC7, CC11 and CC37 to this RG. This could mean that these three samples were made with the same raw materials as those of Alarcos.

- **CCG1A** was initially composed by 17 $I_{c's}$ (Fig.8), but subsequent calculations provided a new perspective. In Fig. 10, 7 of these 17 $I_{c's}$ (CC47, CC25, CC5, CC12, CC17, CC21 and CC23) are located within the larger group from El Cerro de las Cabezas, a distribution that is repeated in other calculations. CC2 moves away from the reference groups and CCANF1 and CC13 bind to CC28, an $I_{c's}$ close to the CCG1A in Fig. 8. The remaining samples (7 $I_{c's}$) have already been explained in the AG2 section.
- **CCG1B** formed by 22 $I_{c's}$ remains the same, but includes 7 samples from CCG1A and 4 $I_{c's}$ from Alarcos (A23, A19, A26 and A36), therefore these last ceramics would have been made in El Cerro de las Cabezas instead of Alarcos. A new CVM has been calculated for these 33 $I_{c's}$, with a vt 0.3399, which confirms that this group has a monogenic origin.

3.1.5 Unclassified Samples

From the dendrogram (Fig.10) it stands out that there are a significant number of samples not classifiable into any reference group. In the case of Alarcos, 13 of the initial 14 $I_{c's}$ are kept isolated from the main groups even in the calculations from which various chemical elements have been removed. Only A48 would join AG1. In El Cerro de las Cabezas the same initial 10 $I_{c's}$ unclassified in Fig. 9 remain in the same way.

From the chemical and statistical analysis, it can be said that the ceramics in El Cerro de las Cabezas present a more homogenous production output. The subgroups CG1A and CG1B from El Cerro are part of a single reference group (RG) with more than 33 $I_{c's}$, including 4 samples from Alarcos. For Alarcos there are two RG composed of 14 $I_{c's}$ (AG1) and 22 $I_{c's}$ (AG2) respectively. As it was discussed above, the vast majority of the samples in Alarcos come from the great store, a reception and food storage place, which probably received ceramics from the farmland areas under Alarcos influence, confirming the dominant role of this *oppidum* over the rest of the nearby settlements. It has been also determined that there existed some kind of commercial or economic exchanges between Alarcos and El Cerro de las Cabezas.

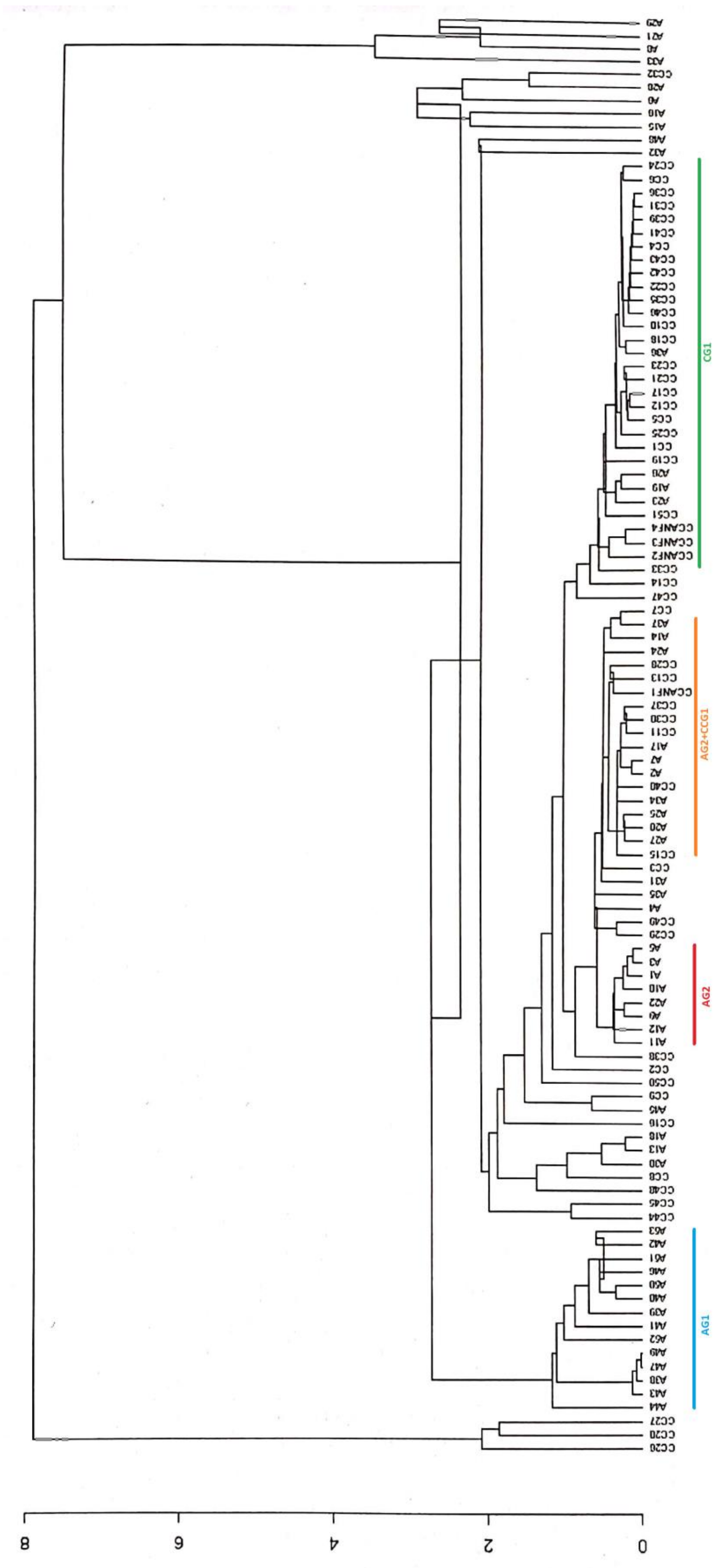


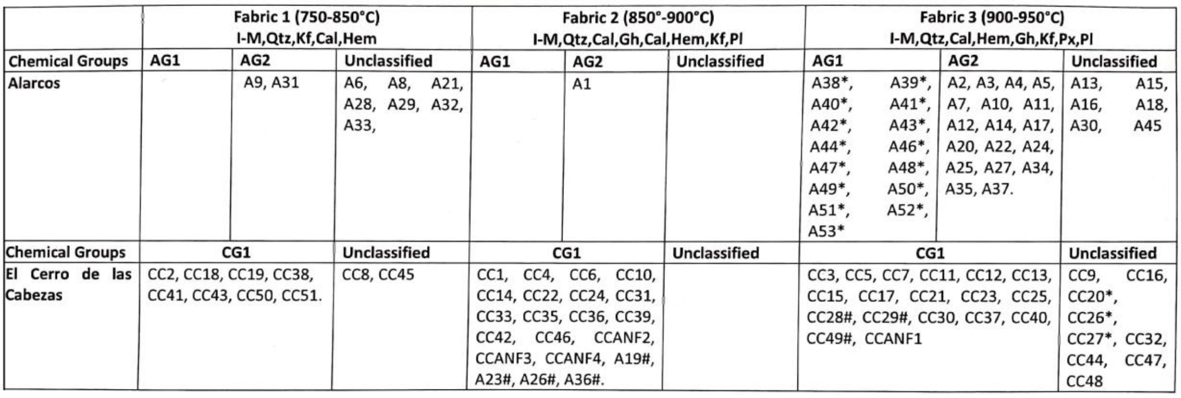
Figure 10. Dendrogram of Alarcos and El Cerro de las Cabezas showing the main groups.

3.2. Mineralogical Analysis

The XRD study of the 107 ceramic fragments has been carried out to determine the different crystalline phases. From the results obtained, these samples have been grouped according to their composition and their estimated firing temperature (EFT). The mineralogical and chemical results have been compared to determine the particular characteristics of each chemical group.

Every sample analysed has a similar composition based on quartz (SiO_2), calcite ($CaCO_3$), potassium feldspars ($KAlSiO_8$), gehlenite (sorosilicate), pyroxenes (calcosilicate or magnesium silicate), hematite (Fe_2O_3) and phyllosilicates (illite-muscovite). During the firing process, the primary mineral phases of the ceramics decompose and form new mineral phases (firing phases). Specifically, the absence or presence of some minerals, allows the firing temperatures to be estimated. Table 3 presents the distribution of the samples according to their EFT in three fabrics (Whitbread 1989), being on the same line as the samples published in Guirao *et al*. (2013). The great majority of the analysed sherds are calcareous (CaO>4-5%), which significantly affects the formation of particular types of new minerals phases. There are also a significant number of samples whose CaO levels are lower than 4-5% (A8, A13, A15, A16, A18, A21, A30, CC8, CC24 and CCANF2) which remain unclassified in relation to the reference groups determined. Samples A13, A18 and A30 form a little group in Figs 7 and 10, and samples A8, A15, A16 and A21 from Alarcos and CC8 from El Cerro de las Cabezas are in the same situation. Only samples CC24 and CCANF2 (CaO=4.59% and 4.24%) take part of the CG1B (Fig. 9). The presence of CaO in ceramics is usually related to primary calcite, but it should also be considered as a secondary calcite formed during burial (Buxeda and Cau 1995). In fact, this phenomenon has been determined in 5 non-calcareous samples (A13, A18, CC8, CC24 and CCANF2) which also present other firing phases like enstatite or magnesium and ferric olivine. Dolomite, a calcium magnesium carbonate has also been detected, a mineral that indicates low firing temperatures because it should be totally decomposed at 800°C. However, this mineral has been found in some samples with an EFT higher than 850°C (Baziotis *et al.,* 2020; Xanthopoulou *et al.,* 2021) what means it could have a secondary origin. At any rate, set criteria for firing temperature determination in ancient ceramics involving e.g. presence of mullite or muscovite or illite and associated observed kinetic reactions have been discussed elsewhere, for temperatures ca 800°C to 1000°C (Baziotis et al., 2020; Xanthopoulou et al., 2021).

Table 3 ***Classification of 107 ceramic samples according to their EFT and place of origin: an asterisk indicates the samples with talc; a hash indicates reclassified samples.***



	Fabric 1 (750-850°C) I-M,Qtz,Kf,Cal,Hem			Fabric 2 (850°-900°C) I-M,Qtz,Cal,Gh,Cal,Hem,Kf,Pl			Fabric 3 (900-950°C) I-M,Qtz,Cal,Hem,Gh,Kf,Px,Pl		
Chemical Groups	**AG1**	**AG2**	**Unclassified**	**AG1**	**AG2**	**Unclassified**	**AG1**	**AG2**	**Unclassified**
Alarcos		A9, A31	A6, A8, A21, A28, A29, A32, A33,		A1		A38*, A39*, A40*, A41*, A42*, A43*, A44*, A46*, A47*, A48*, A49*, A50*, A51*, A52*, A53*	A2, A3, A4, A5, A7, A10, A11, A12, A14, A17, A20, A22, A24, A25, A27, A34, A35, A37.	A13, A15, A16, A18, A30, A45
Chemical Groups	**CG1**		**Unclassified**	**CG1**		**Unclassified**	**CG1**		**Unclassified**
El Cerro de las Cabezas	CC2, CC18, CC19, CC38, CC41, CC43, CC50, CC51.		CC8, CC45	CC1, CC4, CC6, CC10, CC14, CC22, CC24, CC31, CC33, CC35, CC36, CC39, CC42, CC46, CCANF2, CCANF3, CCANF4, A19#, A23#, A26#, A36#.			CC3, CC5, CC7, CC11, CC12, CC13, CC15, CC17, CC21, CC23, CC25, CC28#, CC29#, CC30, CC37, CC40, CC49#, CCANF1		CC9, CC16, CC20*, CC26*, CC27*, CC32, CC44, CC47, CC48

Individuals A38 - A53 (except A45) from Alarcos (AG1) and three more from el Cerro de las Cabezas (CC20-CC26-CC27) present reflections at 3.11 Å and 9.35 Å, assigned to talc, a magnesium silicate. This is a strange mineral not present in a wide area and rarely used in ancient pottery. It could be used as a flux in low-fired clay bodies, but, in this case, it is an incognito that remains unclarified. Only a ceramic fragment from the iberroman site of La Bienvenida-Sisapo also (fig. 1) has talc (Fernández Ochoa and Zarzalejos 2011/12). This site is near to Almadén, an area which is known for its cinnabar mines (exploited by the Romans) and where the presence of talc is also documented (Higueras 1997). The samples with talc in Alarcos are 14, forming a single group in Figs 7 and 10. Individuals CC20, CC26 and CC27, also with talc, are not located in any group. AG1 has a very different chemical and mineralogical composition compared to the other samples from Alarcos. It would be interesting to analyse more samples from Alarcos and La Bienvenida to determine the provenance of this group from the traceability of the talc.

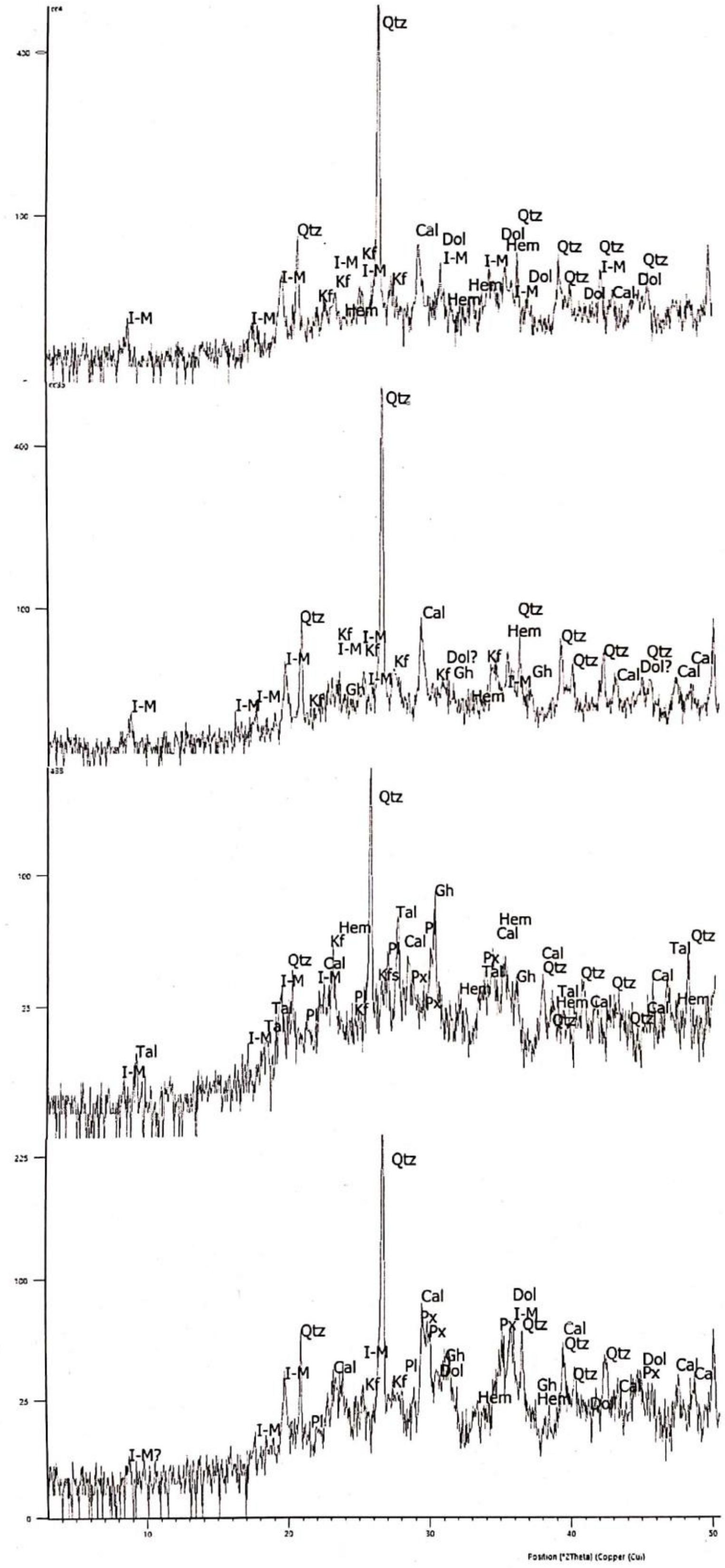


Figure 11. Upper diffractogram: CC4, representing Fabric 1; central diffractograms: CC35, representative Fabric 2; bottom diffractograms: A34 and A39, representing Fabric 3. I-M, illite-muscovite; Pl, plagioclase; Kf, Potassium Felspars; Qtz, quartz; Cal, calcite; Px, pyroxene; Gh, gehlenite; Dol, Dolomite; Tlc, Talc; Hem, Hematite. Abbreviations after Kretz (1983).

In Table 3 all the samples are classified according to their EFT and their chemical groups:

Fabric 1 (F1): It is formed by samples with primary crystalline phases corresponding with phyllosilicates (illite-muscovite - 10 Å), quartz (3.34 Å), calcite (3.03 Å), dolomite (2.88-2.89 Å), hematite (2.69-2.70 Å) and potassium feldspars (3.24 Å). The presence of these minerals, all of them associated with low firing temperatures, and the absence of gehlenite and pyroxenes suggest an EFT between 750°C and 800/850°C (Fig. 11, upper diffractogram).

Fabric 2 (F2): This group is characterized by an initial presence of gehlenite, an Al-rich silicate which it is formed from the decomposition of calcium and aluminium in calcareous ceramics around 900°C (Maniatis *et al.* 1983). Gehlenite starts to decompose at 1050°C. The phyllosilicates in fabric 2 are not completely decomposed and the calcite which decomposes around 900°C, still remains. The samples included in this group are considered as well-fired and the EFT assigned should be between 850°C and 900°C (Fig.11, central diffractogram).

Fabric 3 (F3): Samples with the highest firing temperature are related to the third fabric. Phyllosilicate peaks have almost completely disappeared and relative intensity of calcite is lower than in the other groups. Further, individuals in this group have a main peak at 3.20Å and also at 2.89 Å which are assigned to plagioclase (calcium feldspars) and pyroxenes (diopside or augite) respectively. Based on these new phases, together with an increased presence of gehlenite, an estimated firing temperature between 900°C and 950°C is assigned to this group (Fig.11, bottom diffractogram).

According to Table 3, all the samples of group AG1 are located in F3. Most of the samples from AG2 are situated also in F3, and only 3 samples are in F1 and F2. Unclassified samples are distributed in the 3 fabrics proposed. Samples from El Cerro de las Cabezas are classified almost equally between F1-F2 and F3, although the large majority are in the fabrics with higher temperature assigned.

In summary, most of the samples analysed have reached optimum firing temperatures and they are within the usual ranges determined in Iberian pottery from other archaeological sites (Antón 1973; De Andrés *et al*. 1984; Rincón 1985; Soria and Córdoba 1994; Parras *et al*. 1997, Buxeda and Cau 1998; Tsantini *et al*. 2003; Buxeda and Madrid 2003; Tsantini *et al*. 2005; Tsantini 2007; Compaña *et al*. 2012). In Alarcos there is a tendency to fire at higher temperatures compared with El Cerro de las Cabezas and also with greater homogeneity, since almost all samples from Alarcos are situated in F3, and F1 and F2 are represented for those samples that remain outside the chemical groups.

4. CONCLUSION

In this study, 107 Iberian fragments from two of the most important archaeological sites in the northern Oretania have been archaeometrically studied. From the chemical statistical and mineralogical analysis, it has been determined that two reference groups exist in Alarcos (AG1 and AG2) and a big one in El Cerro de las Cabezas (CG1). AG1 has a very particular chemical and mineralogical composition that make us question their true provenance. The presence of talc in this group, and also from 3 more samples from El Cerro de las Cabezas, could be the key to discover a new production centre in the region. On this point, only AG2 should be considered as a reference group in Alarcos. On the other hand, a greater homogeneity in ceramic production has been demonstrated for El Cerro de las Cabezas in comparison with Alarcos. A plausible explanation for these differences could be in the sampling strategy. Most samples analysed from Alarcos were collected in the great store, a space where ceramic materials from other settlements that were under the influence or the economic control of Alarcos were accumulated. Even so, it is necessary to remember that the sampling from El Cerro de las Cabezas has been much more heterogeneous than at Alarcos, and any sample comes from a space related to the receipt or storage of food. From the conclusions mentioned, we consider the possibility to carry out a broader sampling in Alarcos (sanctuary, Iberian neighbourhood, etc.) and an analysis of samples from one store of El Cerro de las Cabezas.

In relation to trade flow economic relationships between the two sites studied have been confirmed, thanks to the finding of at least 4 samples recovered in the great store of Alarcos but produced in El Cerro de las Cabezas. There is also a large number of unclassified samples, some of them with chemical similarities composed of little groups, but others with a sufficiently disparate chemical composition to assure that they have another provenance. The existence of these unclassified samples is further evidence of the commercial exchanges between these production centres with other Iberian villages.

The mineralogical study has provided some significant data about the particularities of each group. All the samples from AG2 contain dolomite, and talc is only determined in AG1 and CC20-CC26-CC27. Muscovite is a micaceous mineral determined in El Cerro de las Cabezas products which could be used as an identifying mineral of this site. Regarding the EFT, 3 fabrics have been determined with different temperature ranges, whose main difference is the absence of firing phases in fabric 1. In any case, all the samples analysed are well-fired and it can be said that Iberian potters from Alarcos, El Cerro de las Cabezas and others nearby settlements (unclassified ceramics) produced a high quality ceramic and they also controlled each of the required steps for production of the most widespread pottery in the Iberian period.

In summary, in this paper are presented the first results from the archaeometric characterisation of Iberian Common Pottery from two of the most important sites in Northern Oretania, but it is only the first step of our true goal: to characterize the ceramic products from more settlements of this historical region and to deepen in the knowledge of the routes used during the pre-Roman Era.

Author Contributions

Conceptualization, D.G., M.R.G.H., A.A.; methodology, M.R.G.H., A.A; software, P.M.N..; validation, M.R.G.H. and A.A.; formal analysis, D.G.; investigation, D.G., M.R.G.H., A.A. and P.M.N.; resources, D.G.; data curation, M.R.G.H., A.A.; writing—original draft preparation, D.G.; writing—review and editing, M.R.G.H. and A.A.; visualization, D.G..; supervision, P.M.N.; project administration, M.R.G.H.; funding acquisition, M.R.G.H. All authors have read and agreed to the published version of the manuscript. All authors have read and agreed to the published version of the manuscript.

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REFERENCES

Aitchison, J. (1981) *A new approach to null correlations of proportions.* Mathematic Geology, vol. 13, No. 2, pp. 175-189.

Aitchison, J. (1982) The statistical analysis of compositional data. *Journal of the Royal Statistical Society*, vol. 44, No. 2, pp. 139-177.

Aitchison, J. (1986) *The statistical analysis of compositional data*. London, Chapman and Hall.

Antón, G. (1973) Análisis por Difracción de Rayos X de cerámicas Ibéricas Valencianas. *Serie de Trabajos Varios*, vol. 45, pp. 1-66.

Baziotis, I, Xydous, S, Manimanaki, S, Liritzis, I *et al* (2020) An integrated method for ceramic characterization: A case study from the newly excavated Kastrouli site (Late Helladic). *Journal of Cultural Heritage*, Vol. 42, pp. 274–279.

Blánquez, J. (ed.) (2020) In El oppidum oretano de El Cerro de las Cabezas: De yacimiento a Parque Arqueológico. 35 años de investigaciones arqueológicas y patrimoniales. Madrid-Valdepeñas, ediciones de la Universidad Autónoma de Madrid.

Buxeda i Garrigós, J. and Cau Ontiveros, M. A. (1995) Identificación y significado de la calcita secundaria en cerámicas arqueológicas, *Complutum*, vol. 6, pp. 293-309.

Buxeda i Garrigós, J. and Cau Ontiveros, M.A. (1998) Possibilitats i limitacions en l'estudi arqueomètric de les produccions ceràmiques ebusitanes. *Pyrenae: Revista de Prehistòria i Antiguitat de la Mediterrània Occidental*, vol. 29, pp. 97-115.

Buxeda i Garrigós, J. (1999) Alteration and contamination of archaeological ceramics: the perturbation problem. *Journal of Archeological Science,* vol. 26, pp. 295-313.

Buxeda i Garrigós, J. (2001) L'estadística i la seva aplicació en els estudis de provinença dels materials arqueològics. In *Jornades d'Arqueologia i Tecnologies de la Informació i la Comunicació Recerca*, Docència i Difusió, F. Gràcia, J. M. Gurt, C. Carreras and G. Munilla (eds.). Barcelona, Universitat de Barcelona, pp. 71-92.

Buxeda i Garrigós, J. and Madrid, M. (2003) Resultados preliminares de la caracterización arqueométrica de las ánforas ibéricas de la Laietania y la Cosetania. Las ánforas Ibéricas de la costa de Cataluña. *Documents d'Archéologie Méridionale*, vol. 27, pp. 379-403.

Buxeda i Garrigós, J. and Kilikoglou, V. (2003) Total variation as a measure of variability in chemical data sets. In: *Patterns and Process*, a Festschrift in honour of Dr. Edward V., L. van Zelst (ed.), Suitland, Sayre, pp. 185-198.

Caballero, A. and Mena, P. (1987) Los exvotos ibéricos del *oppidum* de Alarcos. In: *XVIII Congreso Nacional de Arqueología,* Zaragoza, pp. 615-633.

Compaña J.M., León L., Capel, C., Jorge, S.E., Hernández, V. and García M.A. (2012) Archaeometric study of Iberian pottery from "El Castillejo" (Alameda, Málaga, Spain). In: *Actas del IX CIA in Lisbon, Estudos Arqueológicos de Oeiras,* vol. 19, Oeiras, pp. 43-50.

Cultrone, G., Molina, E., Grifa, C. and Sebastián, E. (2011) Iberian Ceramic production from Basti (Baza, Spain): First geochemical, mineralogical and textural characterization, *Archaeometry*, vol. 53, No. 2, pp. 340-363.

De Andrés, A., De Andrés, B., Balcazar J.L. and Domínguez, E. (1984) Reconocimiento mineralógico de cerámicas ibéricas. In: *I Congreso Español de Geología*, vol. 2, pp. 205-215.

De Juan, A., Caballero, A. and Fernández, M. (1995) Alarcos medieval: La batalla de Alarcos. In: *Alarcos 95, El fiel de la Balanza, Toledo, Junta de Comunidades de Castilla La Mancha*, pp. 41-59.

De Juan, A. Fernández, M., and Caballero, A. (2004) El Cerro de Alarcos (Ciudad Real). In*: Investigaciones Arqueológicas en Castilla-La Mancha: 1996-2002*. Salamanca, pp. 365-379.

Esteban, G., Hevia, P., Pérez Avilés, J. J. and Vélez, J. (2003) La transición del Bronce Final a la Primera Edad del Hierro en el Cerro de las Cabezas (Valdepeñas, Ciudad Real). Cuadernos de Estudios Manchegos, vol. 24-25, pp. 10-46.

Fernández, M. (2009) Sistemas de almacenamiento en Alarcos: el ejemplo del edificio tripartito. In: *Sistemas de almacenamiento entre los pueblos prerromanos peninsulares,* Mª R. García Huerta and D. Rodríguez González (coords.), Cuenca, Universidad de Castilla-La Mancha, pp. 209-224.

Fernández, M. and García Huerta, R. (1998) El Urbanismo del poblado ibérico de Alarcos (Ciudad Real). In: *Los íberos, príncipes de Occidente, Saguntum No. Extra-1,* Universidad de Valencia, pp. 47-54.

Fernández Maroto, D., Vélez, J. and Pérez, J. (2007) La cerámica estampillada de tipo figurativo del cerro de las Cabezas (Valdepeñas). In: *Proceedings of the Art Iberian in Mediterranean Spain*, Alicante, pp. 211-227.

Fernández Ochoa, C. and Zarzalejos, M. (2011/2012) Un vaso cerámico con decoración dionisíaca procedente de Sisapo-La Bienvenida (Ciudad Real), *Cuadernos de Prehistoria y Arqueología de la Universidad Autónoma de Madrid*, vol. 37-38, pp. 579-597.

García Huerta, R., Morales, F. J., Vélez, J., Soria, L., and Rodríguez, D. (2006) Hornos de pan en la Oretania Septentrional, *Trabajos de Prehistoria*, vol. 63, No. 1, pp. 157-166.

García Huerta, R. and Morales, F. J. (2009) Sistemas de almacenamiento y tratamiento de alimentos entre los pueblos prerromanos en la meseta meridional. In: *Sistemas de almacenamiento entre los pueblos prerromanos peninsulares,* Mª R. García Huerta and D. Rodríguez González (coords.), Cuenca, Universidad de Castilla-La Mancha, pp. 167-208.

García Huerta, R. and Morales, F. J. (2011) El poblamiento ibérico en el Alto Guadiana, *Complutum*, vol. 22. No.1, pp. 155-176.

García Huerta, Mª. R. and Morales, F. J. (2020) El Cerro de Alarcos (Ciudad Real): Formación y desarrollo de un oppidum ibérico. 20 años de excavaciones arqueológicas en el Sector III, Oxford, Oxford University Press,

Guirao, D., García Huerta, R. Rodríguez, D., Acosta, A. and Morales, F. J. (2013) Thermal Characterization of iberian ceramics from Alarcos site, *Proceedings of the II Congreso Internacional sobre Estudios Cerámicos*, Granada.

Guirao, D., Pla, F. and Acosta, A. (2014) The archaeometric characterization of majolica ceramics from Talavera de la Reina and El Puente del Arzobispo (Toledo, Spain), *Archaeometry*, vol. 56, No. 5, pp. 746-763.

Harbottle, G. (1982) *Chemical Characterization in Archaeology*. In: contexts for Prehistoric Exchange, J. E.Ericson and T.K. Earle (eds.), , New York, Academic Press, pp. 13-51.

Hein, A., Tsolakidou, A., Iliopoulos, I., Mommsen, H., Buxeda i Garrigós, J., Montana, G., and Kilikoglou, V. (2002) Standardisation of elemental analytical techniques applied to provenance studies of archaeological ceramics: an inter laboratory calibration study, *Analyst*, Vol. 127, No. 4, pp. 542-553.

Higueras, P. (1997) El Distrito minero de Almadén. In XVII Reunión Científica de la Sociedad Española de Mineralogía. Itinerarios Geológicos, Higueras, P. and Sánchez, C., (eds), Universidad de Castilla-La Mancha, pp. 1-25.

Kretz, R., 1983, Symbols for rock-forming minerals, *American Mineralogist*, 68, 277-9.

Lemoine, C. and Picon, M. (1982) La fixation du phospore par les ceramiques lors de leur enfouissement et ses incidences analytiques, *Revue d'Archaeometrie*, vol. 6, pp. 101-12.

Liritzis, I., Laskaris, N., Vafiadou, A., Karapanagiotis, I., Volonakis, P., Papageorgopoulou, C. and Bratitsi, M. (2020) archaeometry: an overview, *Scientific Culture*, Vol. 6, No. 1, pp. 49-98. DOI: 10.5281/zenodo.3625220

Madrigal, A. (2020) Caracterización e importancia de las cerámicas de importación griegas en el oppidum de El Cerro de las Cabezas. In; *El oppidum oretano de El Cerro de las Cabezas: De yacimiento a Parque Arqueológico.* 35 años de investigaciones arqueológicas y patrimoniales, J. Blánquez (ed.), Valdepeñas, Madrid-Valdepeñas, ediciones de la Universidad Autónoma de Madrid, pp. 165-171.

Maniatis, Y., Simopoulos, A., Kostikas, A. and Perdikatsis, V. (1983) Effect of reducing atmosphere on minerals and iron oxides developed in fired clays: The Role of Ca, *Journal of the American Ceramic Society*, vol. 66, pp. 773-781.

Mata, C. and Bonet, H. (1992) *La cerámica ibérica: Ensayo de tipología*, Trabajos Varios del S.I.P., vol. 89, pp. 117-173.

Moneo, T., Vélez, J. and Pérez, J. (2001) Un santuario de Entrada Ibérico en el Cerro de las Cabezas, C*omplutum*, vol. 12, pp. 123-136.

Morales F. J. (2010) *El poblamiento de época ibérica en la provincia de Ciudad Real. Cuenca*, Servicio de Publicaciones de la Universidad de Castilla-La Mancha

Parras J., Fernández, M. and Sánchez, C. (1997) Estudio mineralógico de las cerámicas ibéricas del yacimiento de Alarcos (Ciudad Real), *Boletín de la Sociedad Española de Mineralogía*, vol. V.20-A, pp.151-152.

Peña-Pozas, J., García Heras, M. and Villegas, M.A. (2011) The archaeometric study of ceramic Materials in JCR journal and conference proceedings during the last decade (2000-2010)., *Boletín de la Sociedad Española de Cerámica y vidrio,* Vol. 50, No. 4, pp. 185-192.

Pérez, J. and Vélez J. (1994), El yacimiento protohistórico del Cerro de las Cabezas. Valdepeñas. Ciudad Real, Patrimonio Histórico-Arqueología. Castilla-La Mancha. *Arqueología en Ciudad Real. Toledo*, Servicio de Publicaciones de la Junta de Comunidades de Castilla-La Mancha, pp. 131-142.

Picon, M. (1973) *Introduction à l'étude technique des céramiques sigillées de Lezoux*, Centre de Recherches sur les techniques gréco-romaines, vol. 2, Université de Dijon.

Picon, M. (1985) Un example de pollution aux dimensions kilométriques: la fixation de barium par les cerámiques, *Revue d'Archéométrie*, vol. 9, pp. 27-29.

Rincón, J.M. (1985), Análisis mineralógico de piezas de cerámica común e ibérica de Cástulo. In, Cástulo V, J.M. Blázquez, M.P. García Gelabert and F. López Pardo (eds.), *Excavaciones arqueológicas en España*, Madrid, Ministerio de Cultura, pp. 329-337.

Rodríguez-Corral, J. (2017) Entangled worlds: materiality, archaeometry and mediterranean atlantic identities in western Iberia. *Mediterranean Archaeology and Archaeometry*, Vol. 17, No 1, pp. 159-178. DOI: 10.5281/zenodo.290668

Soria, L. and Córdoba, M.A. (1994) Análisis Mineralógicos de piezas cerámicas ibéricas procedentes de El Castellón, (Hellín-Albatana, Albacete), Al-basit, vol. 35, pp. 5-49.

Tarradell, M. and Sanmartí, E. (1980) L'état actuel des études sur la cerámique ibérique. In: *Annals Littéraires de l'Université de Besançon*, vol. 242, pp. 301-330.

Torres, T., Vélez, J., Fernández, D., Pérez, J. and Menchén, G. (2014) El sistema defensivo del cerro de las cabezas. Valdepeñas (Ciudad Real): nuevas aportaciones al estudio de las fortificaciones de la zona oretana. In Perspectivas y novedades de la historia military: una aproximación global, E. Martínez and J. Cantera (eds.), Madrid, Ministerio de Defensa, pp.265-278.

Tsantini E., Buxeda i Garrigós, J. and Gurt i Esparraguera, J.M. (2003) Estudi Arqueomètric de les àmfores ibériques del Puig Castellar a Santa Coloma de Gramanet. In: *XIII Coloquio Internacional de Arqueologia de Puigcerdà*, Barcelona, pp. 853-863.

Tsantini E., Buxeda i Garrigós, J., Madrid, M., Gurt i Esparraguera, J.M. and Miñarro i Casas, M. (2005) Amphora production in the pre-Roman northeast of the Iberian Peninsula and evidence of trade with the Balearic Islands, *Trabalhos de Arqueologia,* vol. 42, pp. 279-286.

Tsantini, E. (2007) *Estudi de la producción i la distribució d'àmfores ibèriques en el NE peninsular a travès de la seva caracterització arqueométrica*, PhD Thesis, Universitat de Barcelona, Barcelona.

Vélez, J. and Pérez, A, J. (1996) Estudios sobre la Protohistoria de Valdepeñas y su Comarca, *Cuadernos de Estudios Manchegos,* vol. 22, pp. 9-38.

Vélez, J. and Pérez, J. (2000) El Cerro de las Cabezas (Valdepeñas). Una ciudad ibérica para un Parque Arqueológico, El Patrimonio Arqueológico de Ciudad Real. Métodos de trabajo y actuaciones recientes, Valdepeñas, Sevicio de Publicaciones de la UNED, pp. 137-152.

Vélez, J., Pérez, J. and Carmona, M. (2004) Conjunto arqueológico Cerro de las Cabezas. Puesta en valor de la ciudad ibérica, *Revista de Arqueología*, vol. 279, Madrid, pp. 38-47.

Vélez, J. and Pérez, J. (2010) El Oppidum Ibérico del Cerro de las Cabezas. Estructuras de culto, (Valdepeñas, C. Real), *Anejos de Archivo Español de Arqueología*, vol. 55., Mérida, Consejo Superior de Investigaciones Científicas, pp. 387-406.

Xanthopoulou, V, Iliopoulos, Liritzis, I (2021). Mineralogical and microstructure analysis for characterization and provenance of ceramic artifacts from late Helladic Kastrouli settlement, Delphi (central Greece). *Geosciences,* Vol. 11, Nº 36, https://doi.org/10.3390/geosciences11010036

Weigand, P.C., Harbottle, G., Sayre E.V. (1977) Turquoise sources and source analysis: Mesoamerica and the southwest USA. In: *Exchange systems in Prehistory,* T.K. Earle and J.E. Ericson (eds.), New York, Academic Press, pp. 15-34.

Whitbread, I. K. (1989), A proposal for the systematic description of thin sections towards the study of ancient ceramic technology. In: *Archaeometry: proceedings of the 25th International Symposium,* Y. Maniatis (ed.), Amsterdam, pp. 127-138.

APPENDIX

Table A1. Reference patterns used for majority and minority elements

Nomenclature.	Type	Reference	Origin
GB07103	Granite	NCSDC73301	China
GB07104	Andesite	NCSDC73302	China
GB07105	Basalto	NCSDC73303	China
GB07106	Sandstone	NCSDC73304	China
GB07107	Shale	NCSDC73305	China
GB07109	Syenite	NCSDC71301	China
VS-2123-81	Urtita		Russia
VS-3485-86	Sedimentary Rock		Russia
BCS-313-1	Sand		England

Table A2: Reference Patterns used for trace elements

Nomenclature	Type	Reference	Origin
GB07103	Granite	NCSDC73301	China
GB07104	Andesite	NCSDC73302	China
GB07105	Basalto	NCSDC73303	China
GB07106	Sandstone	NCSDC73304	China
GB07107	Shale	NCSDC73305	China
GB07109	Syenite	NCSDC71301	China
VS-2123-81	Urtita		Russia
VS-3485-86	Sedimentary Rock		Russia
BCS-313-1	Sand		England
GB07108	Limestone	NCSDC73306	China
GB07114	Dolomite	NCSDC71306	China
VS-3192-89	Limestone		China
VS-3139-89	Limestone		China