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ARCHAEOMETRICAL STUDY OF SECOND IRON AGE CERAMICS FROM THE NORTHWESTERN OF THE IBERIAN PENINSULA

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ABSTRACT

This paper presents for the first time, the archaeometric characterization of the ceramics of an archaeological deposit located in the Almar river Valley (Salamanca, Spain) and the study of some soil samples from this archaeological site. Colour characterization was performed according with Munsell Soil Color Chart. X-Ray diffraction analysis (XRD) and polarizing petrographic microscopy observations were carried out to determine the mineralogical composition of the ancient ceramics. Minor and major elements were studied by inductively coupled plasma–mass spectrometry (ICP–MS). Specific surface area was measured using BET methodology. Finally, a multivariate statistical analysis was performance in order to distinguish families of ceramics as a function of their geochemical and mineralogical composition.

The chemical, mineralogical and morphological study characterization of these ceramic materials allows the establishment of three different groups related with the origin of the raw materials. Only one of the ceramic groups was manufactured in the archaeological area. However, the ceramics of the other two groups were probably brought by the ancient inhabitants to the archaeological site. Finally, it was possible determine conclusions about aspects of their manufacture, i.e. the use of non-plastic inclusions in some ceramics to improve the refractoriness of the ceramics.

KEYWORDS: Salamanca (Spain), Second Iron Age, Archaeometry, Ancient ceramics, X-Ray diffraction analysis, Mineralogical composition, Chemical analyses.

1. INTRODUCTION

Archaeometry is a multidiciplinary science which focuses on solving archaeological problems as study of the ceramics origin, study the technology used in the manufacture process, situation of the location production centers (Gómez, 1987, Butzer, 1989; García Heras and Olaetxea, 1992; Pérez Arantegui *et al*., 1996; Williams, 2005; Vigil de la Villa Mencía and García Giménez, 2005).

Nowadays, there are not doubts about the great importance of the science multidisciplinary. Sciences, such as chemistry and geology, could help to improve the understanding of several aspects in archeology. This is the aim of the following work.

There are not a lot of archaeometry studies for protohistoric ceramics in Spain, although there are some works, for example, focusing on the Iberian ceramic production (Coll Conesa, 2000), for Numantine productions founds in previous excavations (García Heras, 1998), for Iron Age Pottery in Galicia (Rey Castiñeira and Soto Arias, 2002) and for the Vaccea zone (Escudero Navarro, 1999). For the specific case of Salamanca province, there are few studies like the work in El Cuquero (Villanueva del Conde) (Ariño *et al.,* 2005). However, none of them had done studies of vettons pottery, for this reason, several authors have stated that the lack of studies of the ceramic pastes prevents the possibility to determine if they are local or foreign productions (Cabello Caja, 1991-1992; Martín Valls and Esparza Arroyo, 1992). The real importance of the present study is the common pottery, since these kinds of potteries do not give us a lot of information through visual and decorative analysis.

The present paper shows the archaeometric data of twenty ceramics specimens found in an archaeological site in the region of Salamanca, Spain (Figure 1). The ceramics samples were found in an archaeological survey due to the realization of the degree work of one of the signatories (de Soto, 2011). The 20 pottery fragments are common ceramic; it is table production, made with potter wheel, with fine modeling clays and small degreasants. Sometimes, this production can have some kind of painted decoration based on lines. The work includes a complete visual, mineralogical, textural and physic-chemical characterization of these ceramics and potential/local regional raw materials. The determination of the regional geochemical background is crucial to identify the source of the raw materials used in the manufacture of the ceramics (Dias and Prudencio, 2008). Therefore, to divide ancient pottery samples as function of origin groups, it is necessary a complete study of the clay materials present in the surrounding area.

Figure 1 Map of Spain with "La Cuesta de Santa Ana" and orthoimage of the archeological site.

La Cuesta de Santa Ana (Figure 1) is an archaeological site located in Garcihernández (Salamanca, Spain) near to the Almar and Gamo rivers (Figure 1). The settlement is located in a hill with no structures; however the ceramic material is abundant in surface. This archaeological site is dated of Second Iron Age (García Martín, 1982; Benet Jordana, 1990), perhaps the settlement was abandoned in the Roman Empire although recent research was found several shards that show the possibility of a later habitat –for example a shard of *terra sigillata* (Ariño 2006). After that, a chapel was built from which the hill takes his name.

This archaeological site can be classified in the *Vettones culture*, a village who lived in the provinces of Ávila- Salamanca Zamora- Toledo-Cáceres (Spain) and Portugal during the Second Iron Age (Salinas, 2001). Its main feature is the location of settlements in high places, the so-called *oppida.* The settlement used to be located near cities, roads and rivers, like Salamanca or Ledesma city. In other occasions, the position suggests the exploitation of mineral resources or agricultural exploitation (Tajo river and Ávila) (Salinas, 2001).

It is frequent to find evidences which show that *oppida* presented defensive locations such as high areas or natural defense areas. The forts of the eastern side of the Tormes river, where La Cuesta de Santa Ana is located, do not have walls because they might be dismantle due to the agricultural work or they might be built with perishable material (Martín Valls, 1998).

Other Vetton expressions were the animal sculptures, called *verracos*. It is not clear the role of the *verracos*, but some researches pointed out that they could be animal protector, milestones, pasture marks or commemorative stones (Álvarez Sanchis, 1999).

The ceramic production of the vettons people are known to the excavations in the *oppida* and necropolis. It is characteristic of this period, the pottery with comb decorations, potter wheel ceramics and painted pottery (Álvarez Sanchis, 1999). Nevertheless, the analyses are stylistic and decorative, not having done materials analysis. We do not desire detract the traditional studies, but we considered necessary the X-Ray analysis and also the chemical to try to clarify the manufacture of the ceramic.

The Salamanca Province is geologically situated within the Central Iberian Zone, the innermost part of the Hercynian Cordillera System (Sociedad Geológica de España e Instituto Geológico y Minero de España, 2004). The archaeological settlement is sited on a thick sequence of Tertiary to Quaternary sediments where siliceous sedimentary rocks are presented. The materials are levels of clays, mudstones, arkoses, sandstones, conglomerates and limestones, with intercalations of palaeochannels of sands and gravels. (García-Sánchez and Álvarez-Ayuso, 2003; Sociedad Geológica de España e Instituto Geológico y Minero de España, 2004).

2. MATERIALS AND METHODS

Twenty ancient pottery samples from the archaeological site were characterized in terms of colour, texture, mineralogical composition and chemical composition. Four clays from the archaeological area were obtained and analyzed in order to gather ceramics as a function of the origin of the raw materials and also, to verified the possible local manufacturing.

The pottery samples were named by the label "GA" to indicate the location of the archaeological site (Garcihernández) and a number. However, the raw materials were labeled with "CL" and a number (Table 1).

The following determinations have been performed to all samples:

1. Visual examination by the colour study in accordance with Munsell Soil Color Chart (Munsell, 1975).

2. Mineralogical analysis by X-Ray diffraction (XDR) using a SIEMENS D-500 with a Cu anode, operate at 30 mA and 40 kV, using divergence and reception slits of 2 mm and 0,6 mm respectively. Peaks were identified following the criteria propose by Schultz (1964) and Brindley and Brown (1984). The estimated peaks for the semiquantitative analysis are: smectite 14.4Å; illite 9.9Å; kaolinite 7.14Å; sheet silicates or phyllosilicates 4.49Å; opal 4.11Å; quartz 4.26Å; feldspar-K 3.30 – 3.24Å; feldspar-Ca-Na or plagioclase 3.22 –

3.18Å; calcite 3.30Å; amphibole 2.29Å and dolomite 2.88Å (Islam and Lotse, 1986). The identification of clay minerals to determine the qualitative and the quantitative composition are according to McManus (1988) for diffractograms of air-dried oriented aggregates, glycolated with ethylene glycol and oven dried at 550ºC.

	Munsell Color		
	Outer surface	Inner surface	Redox condition of the melting process
GA_1	5YR 6/6	5YR 6/3	Oxidation
GA_2	7.5YR 7/2	7.5YR 7/2	Oxidation
GA_3	7.5YR 6/2	7.5YR 5/2	Reduction
GA_4	5YR 6/8	5YR 6/4	Oxidation
GA_5	5YR 7/4	7.5YR 7/2	Oxidation
GA_6	7.5YR 7/6	5YR 7/4	Oxidation
GA_7	10R 6/1	10YR 7/2	Reduction
GA_8	10YR 6/4	7.5YR 6/4	Irregular
GA_9	7.5YR N4	7.5YR 5/6	Reduction
GA_10	7.5YR 7/2	7.5YR 7/4	Reduction
GA_11	5YR 6/3	5YR 7/4	Oxidation
GA_12	5YR 6/4	5YR 7/3	Oxidation
GA_13	5YR 6/1	5YR 6/3	Reduction
GA_14	7.5YR 7/6	7.5YR 7/4	Oxidation
GA_15	2.5YR 6/6	2.5YR 6/6	Oxidation
GA_16	2.5YR 6/2	5YR 6/2	Irregular
GA_17	10R 5/6	10YR 6/3	Irregular
GA_18	5YR 6/6	5YR 6/4	Irregular
GA_19	10YR 7/1	10YR 7/1	Reduction
GA_20	5YR 5/8	5YR 6/2	Oxidation

Table 1 Colour study and redox condition of the melting process.

3. Textural analysis by the study of transversal thin sections of the ceramics (20–25 µm) in a Petrographic Polarization Orto Plan Pol Leitz Microscope, using both white and crossed polarized light with a X 64 magnification.

4. Chemical analyses of major and minor elements by inductively coupled plasma–mass spectrometry (ICP–MS) in an Elan 6000 Perkin-Elmer Sciex. ICP-MS. The previous dissolution of samples was carried out using methodology proposed by García Giménez *et al.* (2005).

5. Specific surface area study. N2 BET surface was determined in a GEMINI V micrometrics® porosimeter after degassing under N2 flow during 18 h at 90ºC (UNE-22-164/94).

6. Statistical study of the data by SPSS program version 18 and Vegana version 1.14 (De Caceres, 2003) using the concentration of minor and major elements and mineralogical composition as variables of the twenty ceramics specimens and four raw materials. Texture, colour and BET specific surface area were not taken into account in the statistical analysis, due to the fact that these properties do not give useful information under an origin point of view (Feliu *et al.*, 2004). However, these data could give information about the manufacturing process.

3. RESULTS AND DISCUSSION

3.1 Colour study of the pottery samples

Table 1 is related with the colour of the pottery samples in both sections, inner and outer surfaces. There are differences between samples and the different sections of the same sample (outer and inner surface). However, the main pottery fragments are located in the same "Hue Colour Chart" (Red-Yellow).

The colour variation observed is attributed to temperature gradients, time of the process and varying redox conditions during the melting process (Pérez Arantegui *et al*., 1996; Mirti, 1998; Mirti and Davit, 2004). According to the colour study, three groups could be drawn: red samples, gray samples and mixed samples (Table 1). The first group corresponds with samples melted under oxidizing conditions (in this environment, the oxidation of iron oxides give a reddish colour to the sample). The second group relates with the reduction of iron oxides. Finally, the last group corresponds with samples in an irregular melting process.

3.2 Mineralogical study

Mineralogical results have shown than all the samples have quartz (Qtz), phyllosilicates (Py), K-feldspar (Fks) and plagioclase (Pl) as main minerals and calcite (Cal), dolomite (Dol), opal and amphibole as accessories minerals (Table 2). Among the phyllosilicates, are recognized micas in different concentration and clay minerals which are identified mainly illite and smectite. When to compare the ceramic samples (GA) compositions with the raw materials (CL) collected in the surroundings, kaolinite appears in CL samples but is absent in GA samples. The kaolinite absence in the pottery materials is because this clay mineral disappears when the firing temperature exceeds 550ºC (Grim, 1968; Kakalis *et al.*, 2001). Not only, the kaolinite content gives information about the manufacture process, the calcite content gives information about the firing processes. The presence of primary calcite provides a temperature of melting smaller than 850ºC due to carbonates disappear during a firing exposure over 850ºC (Schüller, 1984). As a consequence, samples were manufactured under a range of temperature between 550ºC to 850ºC. As has been observed the temperatures are similar to other cases such as Iberic or Numantine pottery (Soria *et al.*, 1994; García Heras, 1998; Coll Conesa, 2000).

Similar mineralogical composition was determined by Ariño *et al.*, (2005) from the samples found in La Armuña (Salamanca). These authors concluded that the pottery samples were manufactured in the surrounding area because the geological features are in accordance with the mineralogical data of the samples.

3.3 Textural study

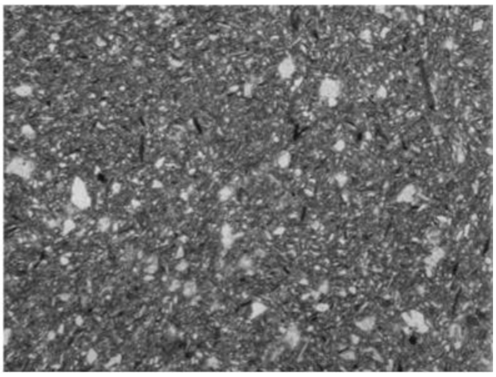
According to the textural analysis, the ceramic samples present two textural groups: fine (Figure 2A) and coarse texture (Figure 2B).

	Py(%)	Qtz (%)	Fks (%)	Pl (%)	Cal (%)	Dol (%)	Others (%)
GA_1	45 (40 illite, 5 mica)	43	2	8	2	-	-
GA_2	45 (40 ilite, 5 mica)	38	6	3	4	4	-
GA_3	28 (22 illite, 6 smectite	30	30	12	-	-	-
GA_4	61 (52 illite, 9 mica)	27	6	-	3	3	-
GA_5	62 (49 illite, 13 mica)	31	2	5	<1	<1	-
GA_6	57 (50 illite, 7 mica)	28	7	8	<1	<1	-
GA_7	37 (8 illite, 29 mica)	52	6	<1	<1	5	-
GA_8	44 (36 illite, 8 mica)	24	26	6	<1	<1	-
GA_9	17 (8 illite, 9 mica)	21	3	55	4	-	-
GA_10	26 (18 illite, 8 mica)	31	31	9	3	-	-
GA_11	50 (41 illite, 9 mica)	37	3	5	3	2	-
GA_12	61 (48 illite, 13 mica)	30	4	-	2	3	-
GA_13	52 (48 illite, 4 mica)	31	5	3	6	3	-
GA_14	52 (45 illite, 7 mica)	24	4	20	-	-	-
GA_15	40 (32 illite, 8 mica)	39	18	-	<1	<1	Opal (3)
GA_16	4 (4 mica)	92	-	2	-	<1	Opal (2)
GA_17	25 (12 illite, 13 mica)	50	2	14	<1	-	Amphibole (9)
GA_18	62 (51 illite, 11 mica)	17	10	11	-	-	-
GA_19	50 (39 illite, 11 mica)	34	5	11	<1	<1	-
GA_20	72 (59 illite, 23 mica)	23	-	5	<1	<1	-
CL_1	1 (1 kaolinite)	99	-	-	-	-	-
CL_2	17 (12 kaolinite, 2 smectite, 3 illite)	77	1	4	1	traces	Opal(<1)
CL_3	<1 (kaolinite)	99	-	-	-	-	-
CL_4	27 (15 kaolinite, 13 illite)	73	<1	<1	-	-	Opal (<1)

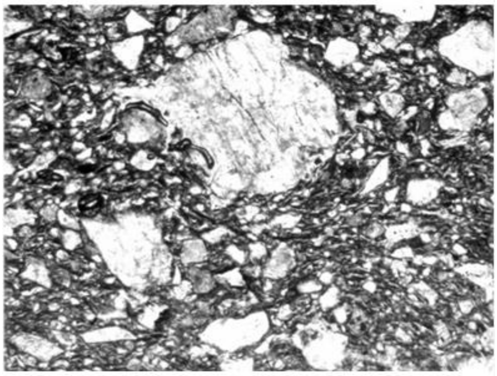
Table 2 Mineralogical composition by XRD for all samples. Py: Phyllosilicates; Qtz: quartz, Fks: K-feldspars, Pl: plagioclase, Cal: calcite, Dol: dolomite.

The fine texture is characteristic for ceramics with a homogeneous groundmass, in which small and sub to well-rounded grains of framework silicates (quartz, feldspars and plagioclase) are included into a matrix of phyllosilicates (Figure 2A).

The coarse fabric is characteristic for ceramics with a poor matrix of sheet silicates where abundant non-plastic inclusions were found (Figure 2B).



A



B

Figure 2 Microphotograph of thin-sections (x 230). A: Fine texture. B: Coarse texture.

Thin-sections show that the most abundant non-plastic inclusions were quartz, feldspars and plagioclase (abundant minerals in the archaeological site) and small fragments of granitic rocks. Therefore, these inclusions are clearly related to the geological environment area (Sociedad Geológica de España e Instituto Geológico y Minero de España, 2004). The grains are big angular to sub-angular grains compare with the fine-grained minerals from the raw material (Figure 2A). Quartz, feldspars and plagioclases were intentionally added to improve the refractoriness of the samples (Hein *et al*. 2007).

3.4 *Chemical study*

Inductively-coupled plasma spectrometry is one of the most important chemical techniques for the characterization of solid materials in recent studies and is becoming more popular in archaeological studies, due to provide information of a huge number of elements (William, 2005). Chemical analysis is ideal to provide a concentration fingerprint of the pottery sample (Marengo *et al*., 2005; William, 2005).

According to their chemical features the pottery samples, there are a high variability of Al, Fe, B and Ba concentrations and a medium variability of V, Li, Cr, As, La, Pb, Zn, Rb and Sr concentrations. These results could be shown in a box and whiskers graph of oxide components (Figure 3) and in the tables 3 and 4.

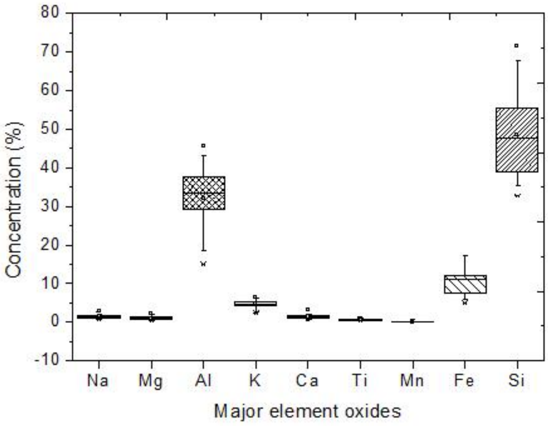


Figure 3 Box and whiskers graph of the major elements of the samples.

3.5 *Specific surface area study*

There is one article that includes nitrogen porosimeter as a method of characterizing ancient pottery samples (Zouridakis and Tzevelekos, 1999) in order to discover the origin of the ceramics. For this reason, we tried to find a relationship between ceramic samples and clay samples using a GEMINI V micrometrics® porosimeter. The results (Table 5) have shown that the values of BET specific surface area in raw materials are higher than values of the parameter in ceramic samples. Thus, a relationship between both ceramic and clay samples, was not found. Therefore, we did not use the BET specific surface area data to verify the possible local manufacturing.

During manufactured process at high temperatures, the ceramic begin to melt and the porous media are smaller than the porous size of the raw material.

	Minimum value	Maximum value	Mean for all samples	RSD*
Phyllosilicates (μgg¹)	4.00	72.00	44.50	17.33
Quartz (μgg⁻¹)	17.00	92.00	35.10	16.17
K-feldspar (μgg⁻¹)	2.00	31.00	9.44	9.77
Calcite (μgg⁻¹)	2.00	6.00	3.37	1.30
Plagioclase (μgg⁻¹)	2.00	55.00	11.06	12.62
Dolomite (μgg⁻¹)	2.00	5.00	3.33	1.03
Na_2O (%)	0.63	2.78	1.46	0.55
MgO (%)	0.35	2.18	1.07	0.53
Al_2O_3 (%)	15.09	15,7	1.99	0.81
K_2O (%)	2.29	6.33	4.68	1.05
CaO (%)	0.57	3.27	1.43	0.60
TiO_2 (%)	0.35	0.93	0.56	0.14
MnO (%)	0.02	0.13	0.05	0.02
Fe_2O_3 (%)	4.93	7.43	5.14	1.89
SiO_2 (%)	62.69	71.45	68.62	4,22

Table 3 Main descriptive statistic of the mineralogical and main chemical dates of the ceramics and raw materials. RSD* = relative standard deviation for all samples expressed in percentage.

	Minimum value (μgg⁻¹)	Maximum value (μgg⁻¹)	Average (μgg⁻¹)
Li	28	97	58±22
Be	2	6	4±1
B	n.d.	966	268±255
Sc	n.d.	26	10±6
V	8	250	107±60
Cr	28	126	71±23
Co	6	21	11±4
Ni	3	42	23±13
Cu	13	58	30±10
Zn	35	233	105±52
Ga	14	33	26±5
As	n.d.	80	21±22
Rb	18	232	112±57
Sr	65	206	125±42
Y	4	23	12±4
Zr	10	47	25±12
Nb	6	21	12±3
Sn	n.d..	8	4±2
Cs	1	14	7±4
Ba	399	964	567±139
La	8	59	25±14
Ce	15	94	47±24
Pr	2	11	66±3
Nd	7	41	22±9
Sm	1	8	4±2
Eu	n.d.	2	1±1
Gd	1	7	4±1
Dy	1	4	3±1
Ho	n.d..	1	n.d.±1.
Er	1	2	1±1
Yb	1	2	1±1
Hf	1	2	1±1
Ta	1	2	1±1
W	2	4	3±1
Tl	n.d.	1	1±1
Pb	16	56	29±11
U	n.d..	11	6±4

Table 4 Main descriptive statistic of the trace elements concentration of the ceramics and raw materials. Nd= non detected (below detection limit).

3.6 Statistical study

Tables 3 and 4 represent the main descriptive statistics for chemical concentrations in archaeological samples. The results show that standard deviation values are significant higher for Al, Fe, B and Ba, than for the other elements. The same results could be shown with a box and whiskers graph (Figure 3). Therefore, we consider that the ceramic are homogeneous under a chemical point of view.

Sample	Specific surface BET (m²/g)
GA_1	2.,38
GA_2	4.06
GA_3	4.15
GA_4	3.66
GA_5	15.71
GA_6	7.08
GA_7	3.3
GA_8	12.89
GA_9	3.99
GA_10	2.94
GA_11	3.42
GA_12	14.73
GA_13	4.29
GA_14	6.11
GA_15	3.08
GA_16	1.68
GA_17	22.23
GA_18	12.85
GA_19	5.69
GA_20	5.32
CL_1	20.4
CL_2	30.54
CL_3	15.67
CL_4	34.78

Table 5 BET Specific Surface Area (m²/g) of the samples

The second step was to obtain the cluster dendogram for the samples (ceramics and raw materials) taking into account mineralogical and chemical variables. Despite the fact that mineralogical concentration data are semiquantitative and chemical data are quantitative, the statistic study has considered both in order to determine the relationship between mineralogical and chemical composition. Clustering analysis is a statistical technique that allows the study the relationships between variables and recognizes the existence of groups (Marengo *et al*., 2005). The most outstanding result is that samples could be divided in three groups (Figure 4) and the raw materials are included in the third group. Therefore, the ceramics from called group three where manufactured in the archaeological site of "La Cuesta de Santa Ana", is worth mentioning that some raw materials have opal, like some ceramics and this mineral is characteristic of some typical formations of the study area. However, ceramics from the first and second group were transported to the archaeological site by the ancient inhabitants, and come from different sources of materials provisioning, one of which contains amphibole (table 2), this mineral are typical from volcanic areas.

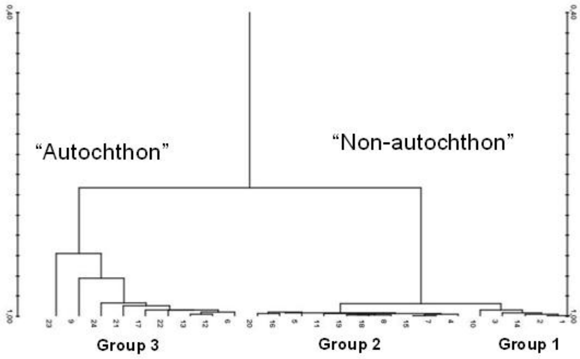


Figure 4 Cluster dendogram of the samples.

The differentiation between "autochthon" and "non-autochthon" ceramics is again revealed in the results of a Canonical Discriminant Analysis (Figure 5). The values of the canonical coefficients 1 and 2 are given in Table 6.

Variables	F 1	F 2
Phyllosilicates	4.250	8.057
Quartz	0.433	8.385
K-Feldspar	3.719	3.432
Plagioclase	1.809	5.770
Calcite	4.684	3.364
Dolomite	-0.978	-0.140
Na_2O	1.211	1.721
MgO	-5.008	-4.297
Al_2O_3	5.845	-5.367
K_2O	3.588	1.090
CaO	-5.927	1.061
TiO_2	7.822	-5.518
MnO	-0.953	-1.237
Fe_2O_3	-4.560	3.688
Li	3.282	0.670
Be	-5.326	0.696
B	3.716	-1.004
Sc	0.249	3.289
V	2.395	3.992
Cr	0.817	1.887
Zn	-5.455	0.069

Table 6 Values of the canonical coefficients obtained with a Canonical Discriminant Analysis.

3. CONCLUSIONS

This study is a pioneer in ceramic characterization collected from the Vetton area (Spain region).

This study we are able to complete several points mentioned below related to the production techniques and the provenience of the potteries. In spite of the fact that the little amount of pottery samples, the study is a great step to the knowledge of the Vettones ceramic productions, just as the possible sources of supply of raw materials.

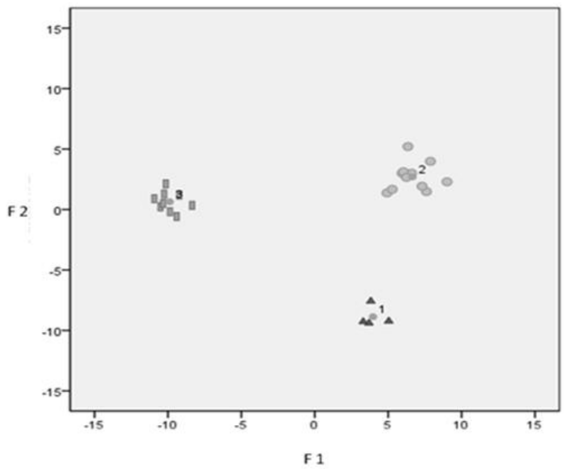


Figure 5 Graph of the Canonical Discriminant Analysis of the ceramic and clay samples.

4.1 Conclusions in relation with the manufacture process

- Two kind of manufacture process were carried out. Addition of non-plastic inclusions (coarse texture) into the raw material to improve the refractoriness of the ceramic and finest ceramic without non-plastic inclusions (fine texture).
- Coarse texture coincides with the group characterized as the archaeological site.
- The temperature of the manufacture process was in the range between 550ºC – 850ºC.
- Two different redox environments were taking place in the melting process, samples melted under oxidizing conditions (most important) and samples melted under reducing conditions.

4.2 Conclusions in relation with the origin of the ceramics

- Three clay types were used for the production of the samples but only one is from the archaeological site. The other two raw materials, used in the manufacture of the ceramics, were from a near area of the archaeological site and another group with amphibole farthest. Therefore, ceramic of group three has an autochthon origin, in contrast with ceramics of group 1 and 2, which have and non- autochthon origin.

- Chemical values are the main variables in a statistic study in order to gather ceramics as a function of the origin of the raw materials and also, to verified the possible local manufacturing. Chemical analysis is ideal to provide a concentration fingerprint of the pottery sample.
- BET specific surface, colour, texture and melting condition give not information about the origin of the ceramics, but they give us another kind of relevant information that we must combine with the precedent analysis. If we study all the techniques, we realized that the habitants of La Cuesta de Santa Ana did not prefer one firing technique over another, all their pottery are not decorated (only a few shards of pottery have paint decoration) and they made their pottery with different kind of material, but the nearest material was the most used.

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