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Archaeometric characterization of *Terra Sigillata Hispanica* from Granada workshops

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Terra Sigillata was a Roman pottery, which was also produced in several workshops in Hispania. Two known workshops at Granada (Cartuja and Carmen de la Muralla, or Albayzin) are known to have produced both typical *Terra Sigillata* and low-gloss coating related pottery. Potsherds from these workshops have been thoroughly studied from an archaeological approach, but not by analytical chemistry techniques. Here, we report a full characterization of nine selected samples from these workshops and also a tenth sample, *Terra Sigillata* made in the Gaul, for comparison. The pastes characterization includes elemental analysis from XRF and quantitative mineralogical analysis by Rietveld analysis of XRPD data. Cluster analysis to both types of data has been carried out. SEM-EDX and GI-XRPD have been used to characterize the slips of the pottery. The elemental analysis results for the pastes suggest that *terra sigillata* potsherds from both workshops were likely made from the same clay, different to that used to make the low-gloss coating pottery. The firing temperatures have been estimated from the phase assemblages being about 900-950ºC for the Granada *sigillata*.

Keywords: Archaeometry, Terra Sigillata Hispanica, Diffraction methods, Microscopy

Caracterización arqueométrica de Terra Sigillata Hispanica de los alfares de Granada

La *Terra Sigillata* fue una cerámica romana, que se produjo también en varios talleres en Hispania. Los talleres ubicados en Granada (Cartuja y Carmen de la Muralla, o Albayzín) produjeron *Terra Sigillata* y cerámicas de barniz mate. Ambos talleres han sido ampliamente estudiados desde un punto de vista arqueológico, pero no mediante técnicas analíticas. En este trabajo, se estudian nueve muestras de estas producciones, y una décima de *Terra Sigillata* producida en la Galia, para su comparación. La caracterización de las pastas incluye análisis elemental por XRF, y análisis mineralógico cuantitativo mediante análisis de Rietveld de los datos de XRPD. Estos dos conjuntos de datos también se han evaluados mediante análisis de cluster. Los recubrimientos superficiales se han caracterizado mediante SEM-EDX y GI-XRPD. Los análisis sugieren que ambos talleres utilizaron probablemente la misma materia prima para la *Terra Sigillata*, y otra diferente en las cerámicas de barniz mate. Se ha estimado una temperatura de cocción para la S*igillata* en el intervalo 900-950ºC.

Palabras clave: Arqueometría, Terra Sigillata Hispánica, Métodos de difracción, Microscopía

1. INTRODUCTION

Terra sigillata was a red pottery very appreciated during the first centuries of the Roman Empire and it was originated in some workshops of Etruria about the half of the first century B.C. (1). It was a fine ware easily recognized because its clay color (*terra*) and because it usually wore potter stamps (*sigillum*). The high-gloss red coating on Roman *terra sigillata* was produced by the application of a fine-textured, non-calcareous slip with high iron oxide content. For this type of pottery, a single-stage firing was used under oxidizing conditions, and the red coating consisted of fine particles of hematite in a partially vitrified matrix. The appealing *sigillata* quickly became a standard and the number of workshops increased enormously. In the first century A.D. there was a flourishing ceramic industry in Hispania (2). The most important workshops of *Terra Sigillata Hispanica* (TSH) were located at Tritium Magallum in La Rioja (3) and at Andújar, in Jaén (4) (Figure 1).

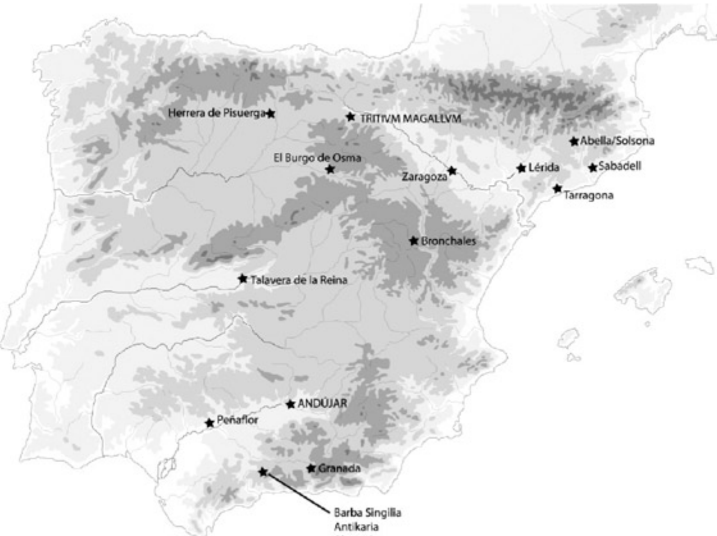

Fig. 1. The known TSH workshops from reference (2).

Possibly associated to the Andújar workshop, there were another two in Granada. The first of them was placed at Cartuja and found in 1964 by M. Sotomayor. It was located in Granada city, near the river Beiro. This workshop produced *terra sigillata* and a variety of low-gloss red-brownish coating very micaeous pottery. Later, in 1982, M. Sotomayor also discovered the Carmen de la Muralla or the Albayzin workshop, in Granada city (5).

The pottery of these two factories has been thoroughly studied from an archaeological point of view; see for example (5-7). From these studies it seems that the production begun at the Albayzin and soon the factory was moved to Cartuja, maybe searching for a better provisioning of raw materials. In any case, their productions should have begun about the half of the first century A.D. and likely reached their end about the half of the second century A.D., due to the massive arrival of African pottery (6). The main production of the workshops has been mainly cups, bowls and dishes, in the same line of the other Hispanic centers (Figure 2).

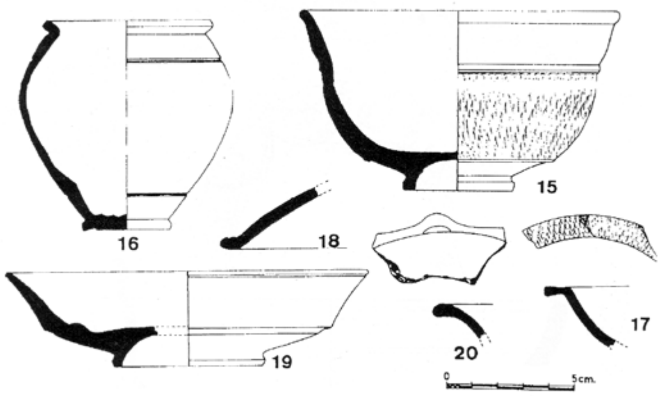

Fig. 2. Some productions from Cartuja and Albayzin workshops from reference (6).

In this work, several potsherds of TSH from Granada workshops have been analyzed to gain a deeper understanding about the nature of the raw materials and about their production technology. Furthermore, these data complement archaeological studies that may be useful in future excavations. The reported analytical results are compared to those obtained for a *terra sigillata* sample from a gallo-roman workshop (TSG).

2. MATERIALS AND EXPERIMENTAL TECHNIQUES

2.1. Samples

Nine selected potsherds from Granada workshops were studied, distributed as follows:

- Three samples of TSH from the excavations of Cartuja (TSH003-TSH005).
- Three samples of low-gloss coating pottery from the excavations of Cartuja (TSH006-TSH008).
- Three samples of TSH from the excavations of Carmen de la Muralla (TSH009-TSH011).
- A sample of gallo-roman *terra sigillata* (TSG001) has also been studied.

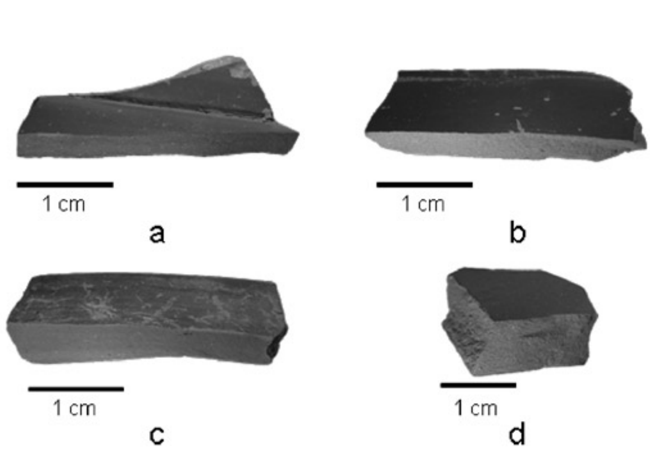

Fig. 3. Photographs of four selected potsherds (a) TSG001, (b) TSH004, (c) TSH006 and (d) TSH009.

Figure 3 displays photographs of four selected potsherds (TSG001, TSH004, TSH006 and TSH009) representative of the studied groups of samples.

The gallo-roman *terra sigillata* potsherd was obtained from the current excavation of the Malaga Roman Theatre (8). The nine samples from Granada workshops were kindly provided by 'Museo Arqueológico y Etnológico de Granada' where the materials from the excavations of Cartuja and Carmen de la Muralla are deposited (5). Three samples from each workshop were gathered and studied as this is part of a wider on-going research project where potsherds from many other Roman kiln locations within Andalusia are being studied.

2.2. Sample preparation

The slip layers were mechanically removed from the body (pastes) by using a pincer. The small fragments of the pastes, without the slips, were then reduced to fine powder in an agate mortar. These powders were analyzed by X-ray fluorescence and diffraction (see below). The fresh broken slip pieces, obtained with the pincer, were gold metalized for the electron microscopy study (see below). Finally, appropriate (flat) surfaces of some raw potsherds were studied by grazing-incident X-ray powder diffraction without any treatment.

2.3. Analytical techniques

The samples were analyzed by the following techniques:

- X-ray fluorescence (XRF): to study the pastes.
- X-ray powder diffraction (XRPD) coupled with the Rietveld method: to study the pastes.
- Scanning electron microscopy (SEM) with energy-dispersive X-ray microanalysis (EDX): to study the slips.
- Grazing-incident X-ray powder diffraction (GI-XRPD): to study the slips.

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. This system can detect all mayor elements and traces from Na to U. A cup was prepared with a 6 mm polypropylene foil and filled up to about 10 mm height with the sample. The weight, diameter and height of the sample were used in the calculations by Omnian, in order to carry out the advanced volumetric geometry correction. The chemical data, expressed as oxides wt%, are given in Table I.

Laboratory XRPD data 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 ($CuK\alpha_1$) 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.

In addition, small slip samples from selected potsherds were metalized in a JEOL Ion Sputter JFC-1100 for about 10 minutes, to give a gold coat being ~300Å thick. Then, they were observed in a JEOL JSM-6490LV scanning electron microscope using secondary electrons. EDX measurements were carried out with the OXFORD INCA Energy 350 attachment. This unit has a Si(Li) detector with a super atmospheric thin window (SATW) capable to detect elements from Be. This equipment was used to analyze the slips of the samples.

GI-XRPD patterns for selected slips were recorded on the PANanalytical X'Pert PRO MPD diffractometer of University of Malaga. For the grazing-incident study, a hybrid monochromator, a parallel plate collimator with 0.18º of equatorial acceptance and a point proportional detector were used. The hybrid monochromator converts the divergent X-ray beam, from a line focus tube, to a quasi-parallel beam with a pure $CuK\alpha_1$ radiation component. A fixed low angle of incidence (θ=1.5º) was selected for all the samples in order not to penetrate more than ~10 μm. The patterns were recorded between 5 and 55º in 2θ scan mode with a step size of 0.03º (2θ). The X-ray tube worked at 45 kV and 40 mA. The approximately flat surfaces of the bulk potsherds were placed on the goniometer centre using the multi-purpose holder of the diffractometer which allows to align samples up to 1 kg of mass.

3. RESULTS AND DISCUSSION

3.1. Chemical composition

The chemical composition of the samples, given in Table I, was processed statistically in order to confirm their expected homogeneity. First of all, the P_2O_5 content was checked, because high P content usually indicates external contamination, i.e. lixiviation of bones if the sample was recovered from a grave (9,10). The highest P_2O_5 content is 0.23% for TSH011, so we can reasonably accept that there is not external contamination in any sample. We have transformed the compositions into log-ratios, in the following way (11-13):

$$x \in S^d \rightarrow z = \log\left(x / g(x)\right) \quad (1)$$

Where x is a vector which contains the d compositions as variables and g(x) is a vector with the geometric mean of each component in group of samples. Then a dendrogram of Euclidean distances, average linkage method, was calculated (Figure 4). P_2O_5 was excluded from the calculations to avoid possible interferences.

All analyzed pastes are Ca-rich, see Table I. For this kind of pastes, Fe cations incorporate mainly into the structures of gehlenite and pyroxenes giving a beige color rather than forming hematite of red color (14) (Figure 3).

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

Sample	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	K_2O	TiO_2	Na_2O	P_2O_5	MnO	BaO	SrO	ZrO_2	SO_3	Cr_2O_3	NiO	CuO	ZnO	Ga_2O_3	Rb_2O	PbO	CeO_2
TSG001	55.05	22.00	14.75	6.34	-	-	-	0.51	0.53	-	-	-	0.05	0.07	-	0.01	-	0.03	-	-	-	-
TSH003	50.14	22.16	11.43	7.64	3.56	3.20	0.81	0.49	0.18	0.11	0.07	0.04	0.04	0.02	0.02	0.01	0.01	0.02	0.01	0.02	0.01	-
TSH004	49.41	21.61	12.72	7.59	3.61	3.10	0.86	0.56	0.15	0.10	0.07	0.04	0.04	0.01	0.02	0.01	0.01	0.02	0.01	0.02	0.01	0.03
TSH005	48.50	21.57	13.63	7.58	3.72	3.06	0.82	0.57	0.15	0.11	0.06	0.04	0.02	0.02	0.02	0.01	0.01	0.02	-	0.02	0.01	-
TSH006	47.47	21.31	14.51	8.03	3.32	3.48	0.85	0.44	0.18	0.12	0.07	0.06	0.03	0.03	0.02	0.01	0.01	0.02	0.01	0.02	0.01	-
TSH007	47.77	21.14	15.14	7.67	3.13	3.35	0.83	0.39	0.19	0.11	0.06	0.05	0.03	0.04	0.01	0.01	0.01	0.02	0.01	0.02	0.01	-
TSH008	49.15	22.25	12.16	7.90	3.10	3.55	0.86	0.46	0.20	0.10	0.07	0.05	0.03	0.03	0.02	0.01	0.01	0.02	0.01	0.02	0.01	-
TSH009	48.99	21.91	12.79	7.73	3.54	3.04	0.81	0.68	0.15	0.11	0.05	0.04	0.02	0.03	0.02	0.01	0.00	0.02	0.01	0.02	0.01	0.03
TSH010	48.79	21.75	12.96	7.61	4.04	3.07	0.78	0.49	0.15	0.11	0.06	0.03	0.03	0.03	0.02	0.01	0.01	0.02	-	0.02	0.01	-
TSH011	48.92	21.90	12.81	7.66	3.57	3.17	0.80	0.51	0.23	0.12	0.06	0.04	0.03	0.02	0.02	0.01	0.01	0.02	-	0.02	0.01	0.06

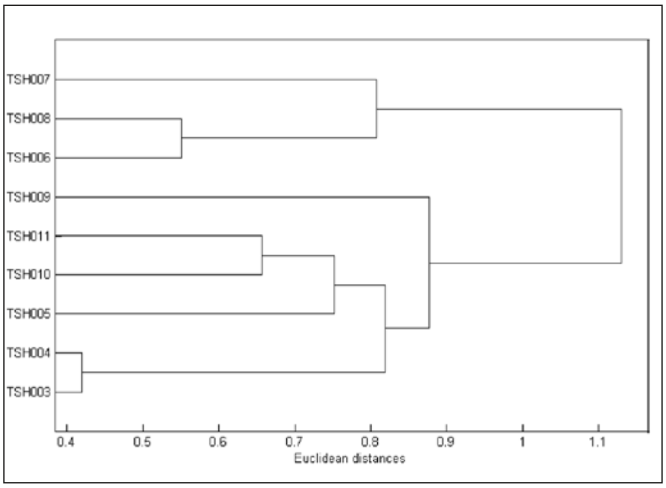

Fig. 4. Dendrogram of Euclidean distances, average linkage method, calculated using elemental analysis from XRF.

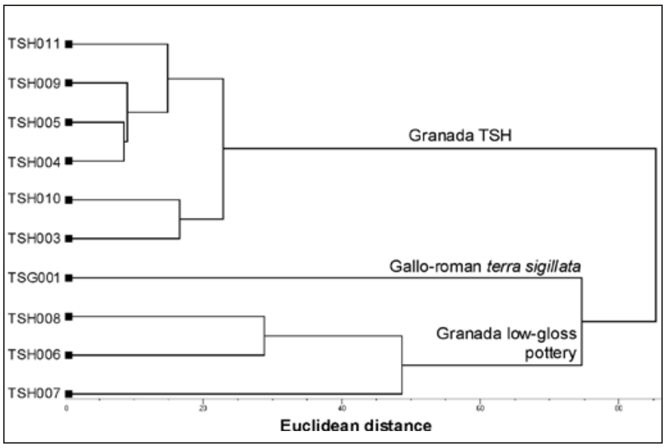

Fig. 5. Dendrogram of Euclidean distances, average linkage method, calculated using XRPD raw patterns.

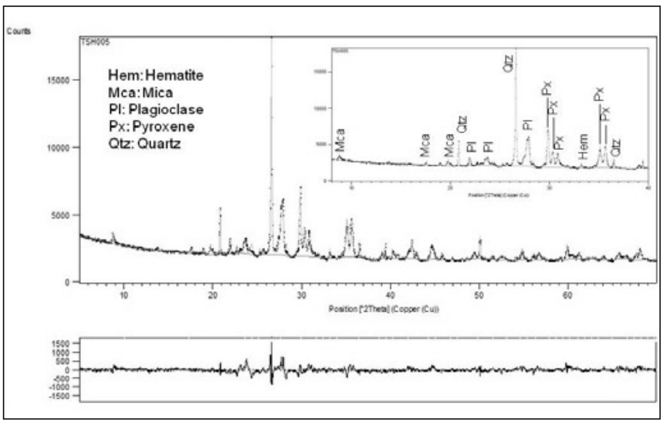

Fig. 6. Observed (dots), calculated (full line) and difference (bottom) powder patterns for TSH005. The inset shows a selected region low-angle region highlighting the main peaks for a given phase.

The most important result of this analysis is that low-gloss coating pottery (TSH006-TSH008) form a chemically independent group from those which includes all the *sigillata* from both workshops (Figure 4). The *sigillata* needed clay with a very high quality, which should be treated before their use. Conversely, low-gloss coating pottery did not required high-quality clays. Then, it is possible that craftmen selected a very specific clay vein to make *sigillata*, different from that used for the rest of the production. This behavior is known in other TSH workshops (15). There are archaeological evidences that *sigillata* production began at Albayzin, and soon was moved to Cartuja (6). So, this switch was probably associated to the finding of an especially good clay vein, and also to a better water supply from Beiro river (5). Furthermore, the provenance determination of pottery may be attained from analytical techniques (16).

3.2. Mineralogical composition

Elemental / chemical analysis of the samples is not sufficient to study the technology of pottery fabrication. For instance, similar elemental compositions may give rise to different potteries as the mineralogical phase assemblage depends upon the firing temperature.

XRPD is a key technique to investigate the mineralogical phase assemblage within the potsherds. There are different analysis levels that may give rise to complementary information. Firstly, the cluster-type analysis of the raw patterns, see for instance (17), may allow grouping the samples based on the main characteristics of the raw patterns. Secondly, the different phases may be identified by comparing the diffraction peaks present in the patterns with those included in Powder Diffraction File (PDF, www.icdd.com). Thirdly, and once the crystalline phases have been identified (and structural descriptions are available), the crystalline phase contents may be quantified using the Rietveld method (18,19). This method is being recognized as a powerful tool in some archaeometric studies like those characterizing stones and ceramics (20-23). Finally, the Rietveld method also allows quantifying the non diffracting content, for instance the vitreous part, by addition of a suitable crystalline standard (24).

Therefore, the XRPD patterns have been analyzed in different ways. Initially, a cluster analysis have been carried out where a dendrogram (Figure 5) was calculated using Euclidean distances and average linkage method (included within the HighScore Plus software) using the raw XRPD patterns for all potsherds. It is noteworthy that again the three low-gloss coating samples group together in a cluster different from the Granada TSH cluster. As expected, the TSG pattern does not group in any cluster.

Latter, the phases present in all XRPD patterns were identified by comparison with the individual powder patterns present in Powder Diffraction File 2, 2004-version (PDF2-2004). The comparison algorithm is that included in the HighScore Plus software.

Quantitative Rietveld analyses of the patterns for all pastes have also been carried out. Rietveld analysis cannot distinguish between very similar minerals, like related plagioclase feldspars, if very-high resolution data are not employed (for instance synchrotron radiation). Hence, the results are usually expressed as totals for the mineral groups, as reported in (9). The mineralogical data for the pastes are given in Table II. It must be underlined that preferred orientation was employed when appropriate (for instance, mica). Figure 6 displays, as an example, the fitted powder pattern for TSH005 where the main peaks arising from a given phase are labeled.

The quantitative Rietveld phase analysis reported in Table II allows understanding the cluster grouping observed in Figure 5. Low-gloss coating samples group together mainly because the relatively high calcite and quartz contents and their relatively low contents of pyroxenes and plagioclases. The obtained crystalline phase contents, Table II, allow estimating the firing temperatures that will be discussed in section 3.4. Furthermore, it must be noted that this methodology quantifies the crystalline hematite fraction. Meanwhile, XRF measures the overall iron content of the samples being expressed as Fe_2O_3. Therefore, direct comparisons between the iron oxide contents from both techniques are not straightforward.

It must be noted that the presence of 5.2 wt% of calcite within the crystalline fraction of TSG001 is clearly of secondary origin (from the burial conditions). The absence of mica and gehlenite indicates a firing temperature higher than 1000 ºC, which will impose total reaction of primary calcite.

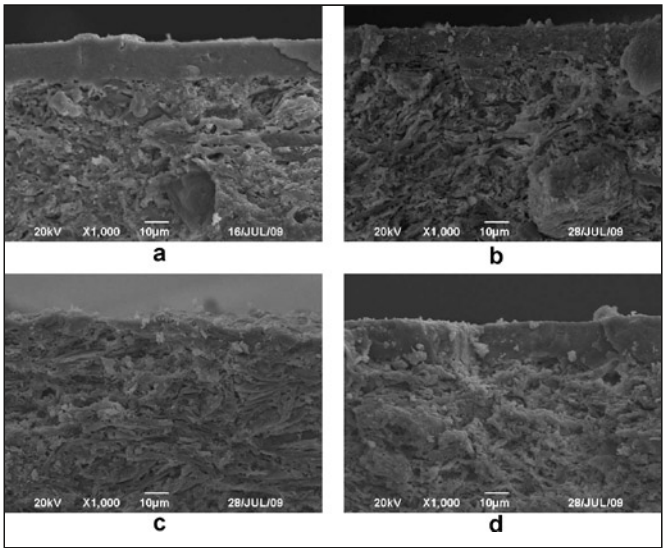

Fig. 7. SEM images of the slips for (a) TSG001, (b) TSH004. (c) TSH006 and (d) TSH009.

3.3. Slips characterization

Small samples of TSG001, TSH004, TSH006 and TSH009 potsherds (see Figure 3) were selected, as representatives of their corresponding groups, and coated with gold, see experimental section. The microstructure of the slips together with its elemental analysis is a very important complementary way to learn about the technology used in *sigillata* ceramics (25,26,27,28). Usually the slips were made of clay with very high content of flux materials, such as K_2O, to allow their adherence to the paste and keeping the appealing high-gloss.

Figure 7 shows the secondary-electron SEM images for the four selected potsherds. It is readily evident that the slip layer for the gallo-roman sample is much well formed that the remaining slips. It is worth noting the absence of a well defined glaze layer for the low-gloss coating Granada pottery. The slip layers for the Granada TSH are well developed but they lack the density of the gallo-roman counterpart. Furthermore, differences in the microstructure of the body of the ceramics are also evident. Concerning the pastes, none of the Granada samples are as well sintered as the Gallic or Italian *sigillata* (25,26). Finally, the high mica content for TSH006 is also visible in the microphotography as sets of layers stacking.

The slip elemental compositions are given in Table III. As expected, high contents of K_2O and Fe_2O_3 were found for all *sigillata* samples. The high content of CaO in the low-gloss ceramic indicates that the clay used to prepare its coating was not as carefully selected/prepared as that used for the *sigillata*. Furthermore, the low K_2O content in the low-gloss ceramic

TABLE II. MINERALOGICAL COMPOSITION (WT%) OF THE PASTES OF THE STUDIED SAMPLES FROM THE RIETVELD ANALYSIS.

Sample	Quartz	Mica[1]	Pyroxenes[2]	Plagioclases[3]	Gehlenite	Hematite	Calcite	K-feldspars[4]	Rwp[5]	FT (ºC)[6]
TSG001	14.9(2)	-	12.4(7)	37.0(1)	-	3.5(2)	5.2(4)	27.0(9)	6.22	>1000ºC
TSH003	18.9(2)	22.2(9)	13.6(6)	20.5(5)	22.0(5)	2.3(2)	0.4(2)	-	7.06	<900ºC
TSH004	19.1(2)	13.8(7)	28.4(6)	37.7(4)	-	1.0(1)	-	-	5.43	~950ºC
TSH005	16.9(2)	10.9(6)	36.5(6)	34.7(9)	-	1.0(1)	-	-	4.92	~950ºC
TSH006	20.6(2)	19.8(8)	38.6(9)	9.2(6)	2.5(3)	2.0(4)	7.2(5)	-	5.62	<900ºC
TSH007	24.9(2)	45.5(5)	10(1)	-	-	0.8(2)	16.7(4)	2.0(4)	5.67	~900 ºC
TSH008	21.8(3)	30.2(1)	5(1)	14.2(9)	19.4(3)	3.7(3)	5.8(9)	-	6.64	<900ºC
TSH009	18.8(2)	10.5(7)	31.0(6)	34.2(9)	-	2.3(2)	-	3.1(8)	5.31	~950ºC
TSH010	18.3(3)	25.7(2)	30(1)	24.4(9)	-	1.4(3)	-	-	7.76	900-950ºC
TSH011	18.4(2)	17.5(8)	29.2(6)	32.5(5)	-	2.3(2)	-	-	5.29	~950ºC

[1] Mica is accounted for as sericite. [2] Pyroxenes are accounted for as diopside and augite. [3] Plagioclases are accounted for as albite and anorthite (or bytownite if appropriate). [4] Potassium-feldspars are accounted for as orthoclase (and microcline in one case). [5] R_{wp} is a disagreement factor which gives an indication of the quality of the fit (17). [6] FT stands for the estimated firing temperature assuming similar residence times in the kilns.

result in a poor developed slip (Figure 7c) which justifies its low-gloss appearance despite its high Fe_2O_3 content. It is worth noting that TSG001 has an Al_2O_3/SiO_2 ratio much smaller that those of the Granada samples, which is likely due to the use of a different type of clay. In Figure 7c, it can also be seen the laminar arrangement of the mica microcrystals being parallel to the slip surface. It is a clear consequence of the use of potter's lathe and moulds in the manufacture process of these wares.

The mineralogical composition of the crystalline phases of the slips has been studied by GI-XRPD, see Figure 8, for those samples studied by electron microscopy. All slips display the diffraction peaks of hematite, as expected. However, it is must be noted that the powder pattern for the low-gloss coating contains the diffraction peaks of quartz and mica but not those of plagioclases and diopside. In any case, the intensity of the plagioclase diffraction peaks in TSG001 is much higher than in the remaining Granada *sigillatas*. Furthermore, some peaks from new phases likely arising from partial decomposition of the outmost slip layer, like zeolite, are evident for some samples (Figure 8). Gypsum may be of secondary origin (a contamination from the burial) or a decomposition product of the slip.

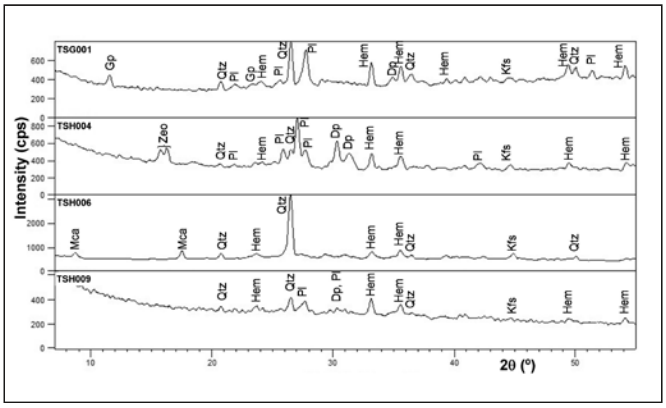

Fig. 8. GI-XRPD patterns for the slips from top to bottom, TSG001, TSH004, TSH006 and TSH009, with the peaks from the main crystalline phases labelled: Dp (Diopside), Gp (Gypsum), Hem (Hematite), Kfs (K-Feldspars), Mca (Mica), Pl (Plagioclase), Qtz (Quartz), Zeo (Zeolite).

3.4. Compositional and technological implications

Taking together the similar chemical analysis of the *sigillata* pastes from the two Granada workshops, see Table I, the similar crystalline phase assemblage, see Table II, and some archaeological findings; it is reasonable to deduce that that the type of clay used in both workshops, for the fabrication of *sigillata*, was the same.

In this case, cluster-analysis of raw powder diffraction data, Figure 5, allows grouping the pottery from its precedence and technology used. We are currently expanding this work by studying/characterizing more Roman potsherds found in Andalusia. The final objective of this study is to determine if this type of cluster-analysis can reveals the origin of a 'given/problematic' potsherd by comparing within a large database. This ongoing work will be reported elsewhere.

The firing temperature, FT, of sigillata can be estimated by using XRPD data (29). We can also estimate FT, within a range, from the phase composition determined in the quantitative Rietveld analysis for the pastes. This is based in the known temperature stability range of the raw phases and also in the formation/decomposition temperatures for the synthetic phases. So, we briefly discuss the key phases and the information that can be deduced. Mica is a key phase as it is a raw material that fully reacts above 1000-1050 ºC depending upon the raw phase composition. Full absence of this clay indicates a firing temperature higher than 1000ºC. Generally speaking, high mica contents indicate low firing temperatures. It must be noted that the observed mica is dehydroxilated. On the other hand, the phases formed at high temperatures are gehlenite, pyroxenes, plagioclases and potassium-feldspars. It must be noted that feldspars (and another silicates) may be added as tempers in the raw mixes but in connection with the clays may give valuable information. Gehlenite is also very indicative as it is formed at high temperature but it reacts/decomposes over 900 ºC (9). Therefore, its presence limits the higher temperature that the analyzed pottery has been heated. Pyroxenes and plagioclases are also indicatives as its ratio evolved with temperature, the amount of plagioclases being larger as temperature increases. Finally, calcite is a bit problematic phase. Primary calcite on heating decomposes, releasing out CO_2, and giving free CaO which usually reacts with the decomposing clays and quartz, resulting in gehlenite, pyroxenes, and feldspars. At temperatures above 850-900 ºC, calcite is not longer stable. However, the so-called secondary calcite can be found in potsherds arising from the burial.

Taken all together, Table II gives the estimated firing temperatures for the different Granada workshops. On average these *sigillata* are fired at temperatures close to 950 ºC. Conversely, the low-gloss coating pottery from these workshops were fired to somewhat lower temperatures, slightly below 900 ºC. The

Table III. Normalized chemical composition of selected slips from SEM-EDX (oxides wt%).

Sample	Al_2O_3	SiO_2	Fe_2O_3	K_2O	Na_2O	CaO	MgO	TiO_2
TSG001	32.82	40.46	13.98	9.70	0.74	1.21	0.43	0.65
TSH004	41.82	25.77	17.06	10.19	1.77	1.34	1.50	0.55
TSH006	30.71	24.34	15.35	5.90	0.58	22.09	0.73	0.28
TSH009	35.74	31.75	10.85	15.82	2.81	1.93	0.79	0.32

phase assemblage for the gallo-roman sigillata indicates a firing temperature higher than 1000 ºC

The firing temperatures estimated from the crystalline phase contents, Table II, are in good agreement with those that can be deduced from the microstructures of the slips and pastes given in Figure 7. Low-gloss coating Granada pottery, see Figure 7c, has pastes with high mica contents which is consistent with firing temperatures lower than 900 ºC. Additionally, these potsherds have not a well defined slip due to a low firing temperature and mainly because the lack of alkaline fraction, see Table III, which can not produce the glaze. On the other hand, TSG001 has the best developed dense slip with a paste that it also seems to contain a vitreous fraction, see Figure 7a. These findings indicate a firing temperature higher than 1000 ºC, in agreement with the powder diffraction study. Finally, Granada *sigillata* samples were fired at intermediate temperatures, close to 950 ºC, given dense slips but relatively porous pastes, see Figure 7b and 7d.

4. CONCLUSIONS

Terra sigillata potsherds from two workshops in Granada have been characterized by chemical analysis (XRF) and mineralogical analysis (XRPD coupled with the Rietveld method). XRF data suggest that both workshops used the same clay for the *sigillata*. Cluster-analysis of XRPD data allowed grouping the samples from their sources. The firing temperatures were estimated from the crystalline phase assemblage being about 900-950ºC for the Granada *sigillata*. Low-gloss coating Granada pottery has also been studied and its firing temperature was lower than 900ºC. Furthermore, the clay selection was less fine and they lack of the high alkaline content that would produce a dense slip. This study has been completed by comparing the results to those obtained for a gallo-roman *terra sigillata* which had a denser paste and a very well defined slip layer.

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