CHARACTERIZATION AND ALTERATION STUDIES OF VALENCIAN SOCARRATS

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ABSTRACT: *The need for a conservation-restoration intervention on a series of socarrats originating from archaeological excavations in Manises (Valencia) and the lack of studies about them have motivated research into the materials and technologies used for their production.*
The lack of research work into the study of Valencian socarrats presently outlines certain discussion about its technique. However, its ornamental technique would be better linked with wall paintings than with ceramic ones, thus endowing these pieces exceptional singularity and difficulty for their intervention.
Sixteen socarrats stored in the Municipal Ceramics Museum in Manises have been characterised by a series of analytical techniques: OM, SEM-EDX, XRD, FTIR, VMP (optical microscopy, scanning electron microscopy and energy dispersive X-ray spectrometry, X-ray diffraction, Fourier transformed infrared spectroscopy, voltamperometry of microparticles). A fired ceramic body constitutes the support of a carbonated slaked lime layer, painted with hematite red and carbon black pigments. As a result of contamination during the burial period, to some extent calcite had converted into gypsum; pigments show a severe loss of cohesion and adhesion to the lime layer. Potential treatments to remove surface dirt must be performed after taking the situation into account. No relevant contamination of soluble salts was detected. This study was undertaken by analysing chromatic changes in a series of replicas submitted to artificial ageing.

KEYWORDS: socarrats, ceramics, wall paintings, lime, alteration, characterisation

INTRODUCTION

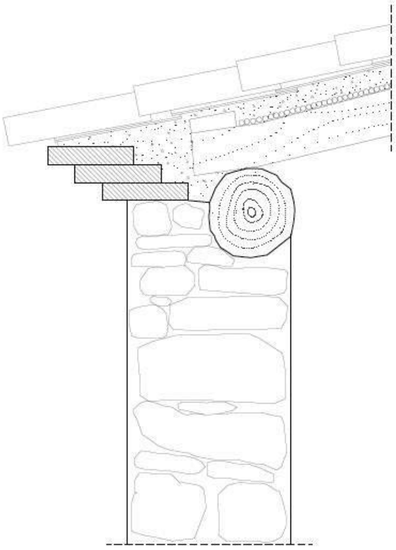
The term socarrat[1] refers to fired clay tiles covered with a white base, generally painted red and black. These were placed between beams and joists in the ceilings and eaves of of buildings. Their origin is typically Medieval, although the subsequent production of these objects is known, mainly in Valencia. There are other words to name such objects of a similar function: rajola, maó prim, atovó or cairó.

The first registered information of their existence dates back to 1604 when D. Feliciano de Figueroa, Bishop of Segorbe, referred to a group of roof and wall tiles, written and coloured with Koranic transcripts (Labarta and Barcelò, 1986: 468). Socarrats were mainly manufactured in two basic sizes: the smaller being 30 x 15 x 3 cm, and the larger measuring 40 x 30 x 3 cm (approximate measurements). The former may have been used in buildings in two main ways: to decorate eaves (the lower edges of a roof projecting over walls) that leaned on either walls or joists. They could have also been used in ornamental friezes, and on balconies and staircases (López et al., 2000: 15).

The largest tiles filled the spaces between joists on interior ceilings for both structural and decorative functions, and to support pavings or roofs. The smaller-sized pieces could have performed the same functions (Pérez Camps, 2007). *Socarrats* were frequently reused to build new walls and to level out pavings (López et al, 2000: 68). Traditionally, they are said to have come from Paterna (González Martí, 1952: 337-538), but the presence of these and of other similar objects has also been documented in Manises (Pérez Camps, 2003: 11) and in other places in Valencia, Aragon and Catalonia (Coll Conesa, 2007: 138-144).

Debate as to socarrat manufacturing is ongoing. Since they are objects with a ceramic base, controversy starts when considering the stages that occured after drying the moulded ceramic paste. González Martí and Blat Monsó are the most representative authors on this subject.

According to González Martí (1952: 337-538), the dried tile would have been covered with a kaolin-based earth and painted with iron



1 Figure 1 Scheme of the usage of *socarrats* in eaves

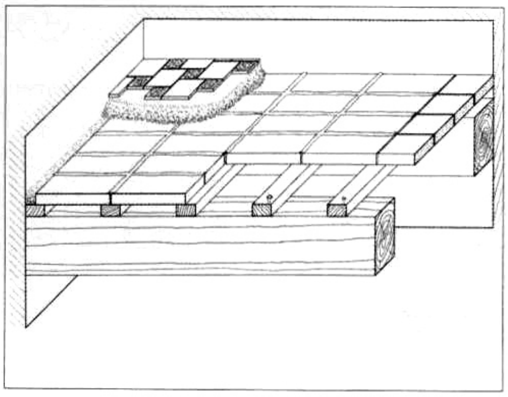


Figure 2 Scheme of the usage of *socarrats* on floors with superimposed pavement (Mileto et al., 2006: 301-303)

Stages	1	2	3	4	5	6	7
Initial T (ºC)	25	40	40	80	80	40	40
Final T (ºC)	40	40	80	80	40	40	25
Duration (h)	6	6	6	6	6	6	6

Table 1 Stages of the relative humidity and temperature ageing cycles

and manganese oxides. Then it would have been fired, resulting in a matt decoration. González Martí describes the finding of 50 socarrats and affirms that those covered with lime and smoke were fakes. After washing them with water and a scouring pad, all the decoration vanished.

The existence of a firing process and the nature of pigments has always remained unclear (Almarche Vázquez, 1924: 30-51). Before González Martí, some authors referred to the lime used to decorate socarrats, and how it could not have been fired after being applied to the tile (Ribera i Tarragó, 1889: 542-549). Some time later, Blat Monsó (1962: 527-529) and others (Llubiá, 1967: 22), (Cirici, 1977: 173-180) reinforced this statement. Two recent analysis confirm it, despite a slight misunderstanding among the various forms of lime in its cycle (Soler Ferrer, 1988: 237-249) or the results not being clearly presented (Pita and Torrijos, 1999). The most recent work shows that lime, in the form of calcium carbonate, is the white coating material of socarrats, and that almagra red, carbon black and manganese black are the pigments used to paint on it (Doménech-Carbó and Aura Castro, 2002). The authors also state that the compounds in the ceramic pastes confirm a firing temperature of at least 900ºC owing to the presence of mullite and cristobalite. Temperatures like these would burn lime and cause its decomposition in calcium oxide and carbon dioxide at 787ºC. On the other hand, some *socarrats* found while extending the Ceramics Museum in Manises show remains of graphite used as a preparatory drawing, which would have been burnt and disappeared if they had been fired (Coll Conesa and Pérez Camps, 1993: 881, 888).

There are three basic types of representations in *socarrats*: religious, magic and social. The first includes crosses and inscriptions, such as Koranic verses written on the *socarrats* of the Xara mosque in Valdigna. Fatima's hands, boats, towers, animals and chimeric figures such as Butoni, a monster of Valencian imaginary, are part of the second type. Finally the third type is depicted by heraldic symbols and decorated elements located visibly in public spaces, as well as courtesan and satiric scenes. *Socarrats* were also used for public announcements, such as the Duke of Segorbe's edict to recruit soldiers in 1513 (Coll Conesa, 2007: 138-144).

EXPERIMENTAL

Instruments and material

To perform this research, we used the following material and instruments:

- Cross section samples were included in a polyester resin (Kit Struers Serifix®), and polished with Struers Knuth Rotor 2®, and water sandpaper 220, 500, 2400 e 4000 mesh. The process was controlled with a Leica GZ 6® microscope.
- The optical microscopy examination was performed with a Leica DM RXP® equipment and a Leica MPS 60 photographic system.
- The characterisation of the ceramic body, pictorial layers and pigments was carried out with a Jeol JSM 6300 electronic

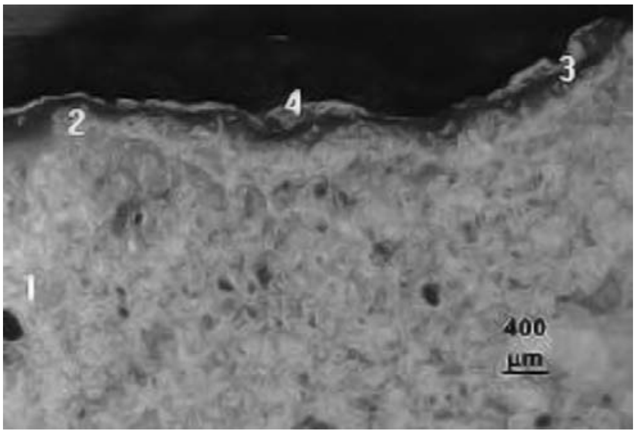


Figure 4 Cross section of *socarrat* 10. BSE (back scattered electrons)

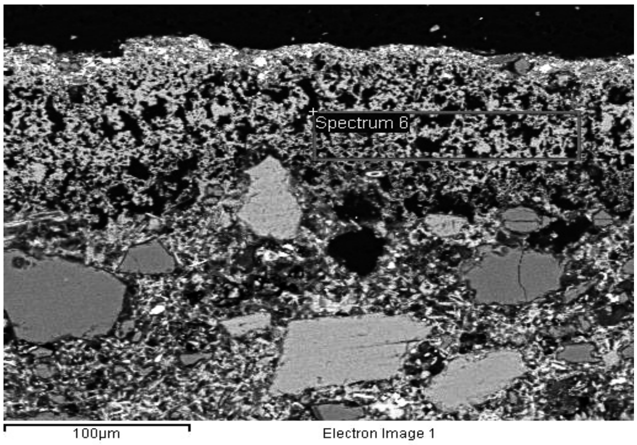


Figure 6 Correlation of the weight percentage of silica vs calcium oxide in the ceramic body of the analyzed *socarrats*

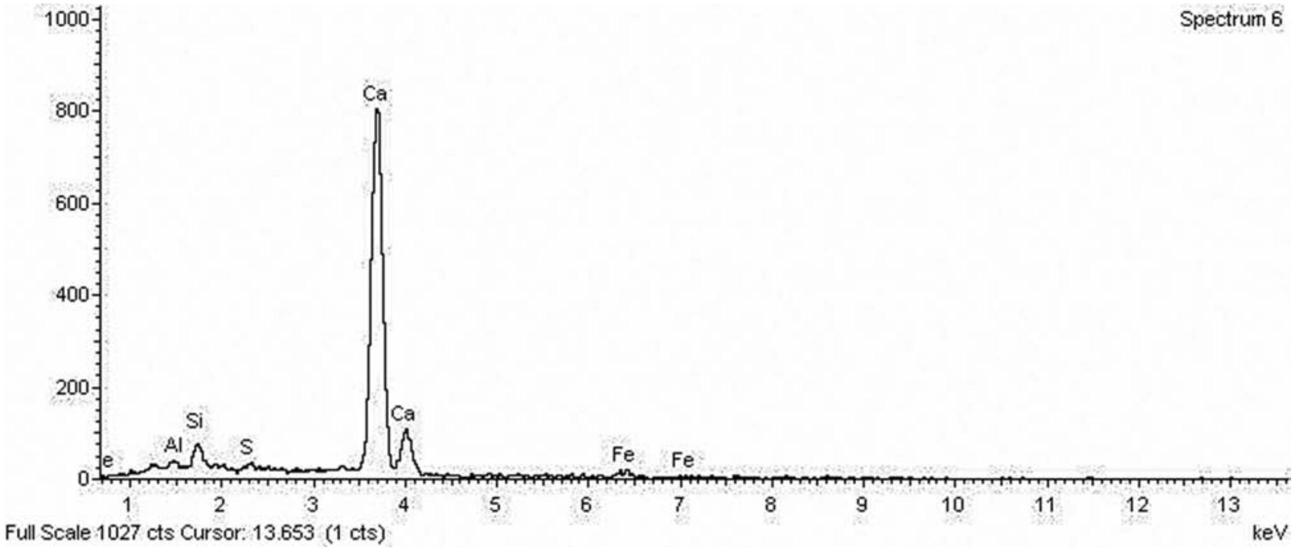


Figure 5 X-ray spectrum obtained in the indicated area of the last figure, with presence of calcium, iron, aluminium, silicon, and sulphur

microscope with a Link-Oxford Isis X-ray microprobe, operating at 20 kV voltage with a beam of 2.10-9 Å at a working distance of 15 mm. Samples were carbon-coated. Three different areas were analysed in order to obtain quantitative results.

- The white layer was characterised with a Vertex® 70v infrared spectroscope, equipped with a coated and thermally stabilised detector by FR-DGTS (fast recovery deuterated triglycine sulphate). Number of scans: 32, resolution: 4 cm-1. Analysis software is OPUS®.

- Crystallographic identification was carried out following the powder method with the X-ray difractometer Philips PW 1830 DMP 2000, with a copper anticathode. Identification was done with the software and data from the International Center for Diffraction Data, USA.

- Voltamperometric analysis were done at 298 K after the immersion of the modified electrodes in deareated 0.20 M HCl, using a CH I420 equipment. A conventional three-electrode arrangement was used with a PIGE (paraffin-impregnated graphite electrode) working electrode, a Pt-wire auxiliary electrode and an AgCl (3M NaCl)/Ag reference one.

- All the weight measurements were taken with a Precisa XT 120 A analytical balance

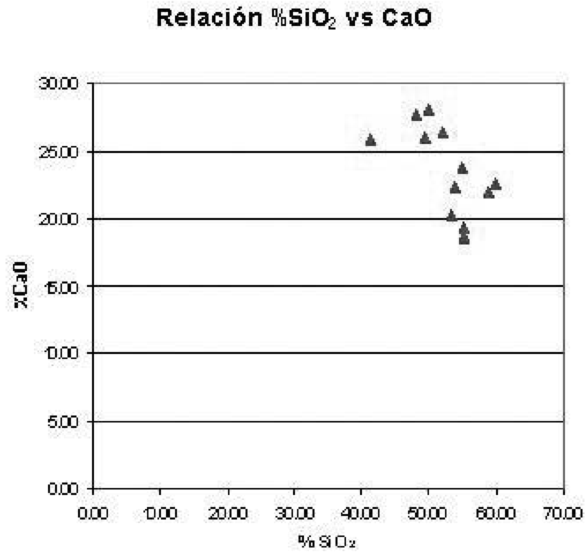
- Replicas were prepared with the 5 x 5 x 1 ceramic tiles, grassello of slaked lime CTS, pH 12-13.5, d = 1.59 g/cm3, red pigment 'Rosso di Marte' PR101 Maimeri, carbon black pigment Kremer (PBk10.77265), manganese black pigment Kremer PBk14.77728. Five series were produced: NA (unaged), HT (humidity and temperature), UV (ultraviolet), SO (SO2), MP (multi processes). Each series included a white sample (covered with lime), and three others covered with lime and each of the aforementioned pigments. Reserve replicas were prepared following the ME process.

- The chromatic change of replicas has been determined with a Minolta® CM-2600 D, small analysis area, illuminant D65, observer at 10º, CIELAB measuring system. Each artificially aged simple chromatic change was measured at four points, three times each. The final result is an average of these measurements.

- Replicas were submitted to humidity and temperature ageing in a Dycometal DI-100®. The 48-hour ageing cycles were performed over a 24-day period. Relative humidity (RH) was

	Main d (Å)	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Quartz	3,34 4,26 1,817	VI	VI	VI	VI	VI	VI	VI	VI	VI	VI	VI	VI	VI	VI	VI	VI
Calcite	3,35 3,86 2,285	M	I	M	I	VI	VI	M	VI	VI	M	M	VI	VI	VI	VI	M
Gehlenite	2,85 2,404 3,07	I	W	M	M	VI	M	M	M	M	M	M	W	M	M	VI	M
Diopside	2,991 2,952 2,893	W	-	W	W	W	-	-	W	M	-	-	-	M	W	-	-
Wollastonite	3,510 1,826	-	W	W	W	W	W	W	W	M	W	-	W	-	-	-	-

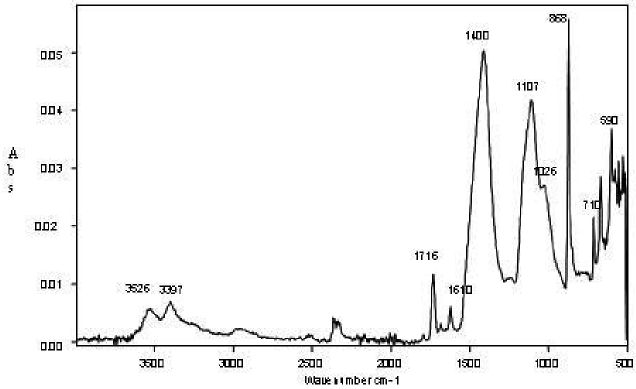
Table 2 Qualitative evaluation of the spectral intensity of the peaks corresponding to crystalographic species of the ceramic body. (VI = very intense, I = intense, M = moderate, W = weak)



Bands (cm^{-1})	Identification
3526, 3397	O-H tension
1716	C=O (ester) tension
1610	O-H deformation
1400	CO_3^{2-} tension
1026	Si-O-S tension
868	CO_3^{2-} tension
710	CO_3^{2-} tension
590	SO_4^{2-} tension

Figure 7 IR spectrum obtained through analysis of the white layer of *socarrat* 2.

Table 3 FTIR Table



	White				Mn Black				C Black				Almagra Red			
	ΔL*	Δa*	Δb*	ΔE	ΔL*	Δa*	Δb*	ΔE	ΔL*	Δa*	Δb*	ΔE	ΔL*	Δa*	Δb*	ΔE
NA	-0.51	0.08	0.47	0.70	20.89	-1.84	-5.25	21.62	7.02	-0.22	-0.62	7.06	8.40	-13.34	-27.88	32.03
HT	-0.48	0.04	0.61	0.77	18.54	-1.68	-4.23	19.09	6.90	-0.24	-0.45	6.92	6.40	-13.67	-27.65	31.50
SO	-1.31	-0.07	2.31	2.66	25.21	-1.65	-3.16	25.46	1.88	-0.11	0.68	2.00	8.49	-14.67	-27.72	32.50
UV	-0.32	0.09	0.27	0.43	20.02	-1.80	-4.55	20.61	7.91	-0.26	-0.61	7.94	8.66	-12.69	-28.16	32.08
MP	0.55	0.03	1.27	1.38	27.39	-1.80	-3.34	27.65	1.52	-0.13	0.16	1.54	8.96	-14.46	-27.97	32.74

Table4. Variations of the chromatic coordinates L*, a*, b* and E in replicas submitted to artificial ageing. NA = non aged, HT = humidity and temperature, UV = ultraviolet, SO = SO2, MP = multiple processes

maintained in the 70±10% interval. The process followed the subsequent stages:

- UV accelerated ageing was performed with Dycometal QUV-Basic®, with lamps UVB-313EL. The test took 9 hours.

- SO2-accelerated ageing was performed in a Dycometal VCK-300® prepared for the DIN-50.108 test (Kesternich test). Samples were submitted to four consecutive cycles: the first and third lasting 8 hours in a closed chamber with an atmosphere saturated in SO2 (constant concentration of 2g/l), while the second and third cycles lasted 8 hours in an open chamber. T and RH were maintained at 40ºC ± 1ºC and 100%, respectively.

Damage and alteration

In this group of fragments from the 16 excavated socarrats, the alteration and damage forms were all quite similar to each other. These were:

- Fractures. The fragments presented fractures caused by disintegration of the building structures, earth pressure or by building operations (in some casesthe context they were found in was not an archaeological one).

- Fissures. The white layer presented fissures, caused by hydro-thermal changes and material expansion (mechanical tension and increased porosity)

- Encrustations. The white layer and the ceramic body presented deposits of earth, mortars, lime and cements that were hard to remove

- Abrasion, losses in pictorial layers. Abrasion, lack of cohesion and adhesion of the black or red pigments and of the white layer implied a loss of pictorial material.

- Chromatic changes. The ceramic body and pictorial layers coming into contact with foreign materials caused staining. A fine white veil covered almost every fragment.

Replica preparation

Several tests were carried out and the main difficulties observed were the appearance of bubbles and fissures. The drying process was satisfactory and the pictorial layers were uniform. Some lime and pigments concentrations dissolved in water were tested. Optimal concentrations were obtained by adding 100 mL of lime water to 650 g of slaked lime (4.33 g of slaked lime per mL of water). The red and black paints were prepared by adding 150 mL of lime water to 0.1 g of almagra red, 0.35 g of manganese black and 0.15g of carbon black. The methodology used for replica preparation was the following:

- Place the ceramic tiles in deionised water for 24 hours to eliminate the air deposited in its pores. Use a cloth to remove any excess water before applying the slaked lime.

- Apply the slaked lime with a brush and obtain an irregular surface. After the loss of superficial gloss, allow the drying process to continue for two more hours.

- Apply the paints with a painting brush and wait until they have dried

RESULTS AND DISCUSSION

Optical microscopy

The ceramic body of the fragments of the 16 socarrats was strong but coarse. Porosity was variable, as were the colours which ranged from ochre yellow to earth. All of the pieces had been buried for a significant period: 12 of them were excavated in Manises in the last decade of the 20th century, while the other four were accidentally found during renovation works of the city's buildings. All of them presented quartz grains that were particularly evident of optical behaviour. The white layer presented variable porosity in each cross section sample. The pictorial layers presented an irregular surface and porosity, probably due to alteration. Contact with earth produced some deposits.

A cross-sectional examination provided a better understanding of the decoration of the socarrats. The ceramic body was covered with a white layer that was later painted in red or black. Some preparatory drawing had been likely applied on the white layer, and lime water would have been used as a medium for the red and black paints. Although it was possible to observe a partial or total penetration of the paint into the white layer, it was not possible to assume whether this represented an a fresco or an a secco process. The heterometric granulometry of the red pigment was characteristic of red earth ones.

Scanning electron microscopy – energy dispersive x-ray spectrometry

SEM–EDX was used to characterise the morphology and elemental composition of the cross sections previously observed by OM, and to quantify the components of the ceramic body.

The analysis of the morphology of the ceramic body confirmed its good conservation state. We were able to see a sinterized texture with variable porosity. The ceramic paste would have been of good quality and the firing process well executed. This also made the penetration and degradation by soluble salts somewhat more difficult.

The white layer was quite porous and X-ray spectrometry confirmed the presence of calcium in it. Its origin would have been carbonated lime. Silicon and aluminium would have been the result of contamination after the white layer came into contact with the ceramic body. Sulphur may have been present in the gypsum, which is a typical alteration product of carbonated compounds.

The intention of analysing the black paint was to understand the nature of the black pigment: either carbon- or manganese-based. The X-ray spectra obtained confirmed that there was no manganese in the black pigment, while the analysis of the red pigment confirmed the presence of iron.

The percentages of silica and of some other oxides have been correlated. We verified that there were quite high calcite contents (this situation has been previously described (Aura Castro, 1996: 104). Of the set of samples, the potassium oxide contents were somewhat heterogeneous. On the other hand, the iron oxide contents were very similar. The alumina contents formed a homogeneous group, except for one sample with a higher alumina content. The standard deviation values associated with this measure were quite high. Therefore, we may assume that this sample presented local alumina enrichment.

X-ray diffraction

X-ray diffraction is a technique that allows the identification of the crystalline structures by measuring the constructive interferences of radiation with crystalline planes of compounds. By correlating this data with the temperature formation of each species, we obtained data about the firing temperature of the samples. Here, the aim was to determine the firing temperatures of the ceramic body

There was a considerable amount of quartz in the ceramic body. Calcite, diopside, gehlenite and wollastonite were also detected. These circumstance led us to assume that these ceramics had been fired at temperatures of around 1000ºC.

Fourier transformed infrared spectrometry

The Fourier transformed infrared spectrometry technique is based on the detection of the energy transitions generated by dipolar moment variations. SEM-EDX indicated the presence of calcium in the white layer. The aim of this analysis was to understand what kind of calcium compound was present in the white layer of the *socarrats*.

Carbonates and sulphates were detected. All the spectra presented a clear carbonate band which led us to assume that calcite was the main compound present in the white layer. However, sulphates were also present and their presence is generally associated with calcite to gypsum alteration. Silicate detection may be attributed to the contamination of the white layer by either earth or the ceramic body. Some residual silica may have also been present in lime before application. The consolidation of socarrats is documented, which is why the carbonyl (ester) groups were detected (Blanes, 2003).

Square wave microparticles voltamperometry

In previous works, this technique has been used to identify earth pigments in pictorial samples (Doménech et al, 2001: 1764), (Grygar et al, 2002: 1100-1107), (Doménech et al, 2007). The present work has revealed information about the crystalline structure and the oxidation state of the species composing the black and red paints.

The presence of Fe in the X-ray spectra of the red paint showed that the pigment could be iron oxide. This result has been confirmed in voltagrams obtained from microsamples of the red paint, where a strong hematite signal was detected. The majority of the samples presented a voltametric profile, including a large peak at -300 mV and another one at -525 mV, which may be attributed to the presence of hydrated hematite and crystalline hematite, respectively. This means that the sample had not been submitted to significant heating. At 500ºC, hematite is transformed into maghemite, while crystalline hematite partially transforms magnetite at 600ºC (Yariv, 1980: 37-61). Sample 4 showed a particularly evident sign of crystalline hematite. In this case, a different pigment preparation or a less aggressive alteration could have applied.

The total lack of signs of characteristic elements in the X-ray spectra of black pigments led us to confirm the presence of carbon black. This result has been confirmed by voltamperometry in which no trace of Fe or Mn blacks was found.

Artificial ageing

Replicas of the socarrats were artificially aged in order to understand the main alteration agents of these works. The data obtained from the colorimetric measures before and after ageing are presented in the following table:

Firstly, among all the considered accelerated ageing processes Firstly, of all the considered accelerated ageing processes, there was a general evolution leading to lighter ($\Delta L^*>0$), and greener ($\Delta a^*<0$) and bluer ($\Delta b^*<0$) colours. Then, the submission to all the ageing processes (MP) produced the most significant chromatic changes in almost every case. However, neither the former consideration for the white series nor the latter for the C black one is valid.

Little change in the white series was observed. Some yellowing appeared ($\Delta b^*>0$), but more yellowing was observed with SO. Yet, the colorimetric equipment had a low resolution to the values in the extremes of the L* coordinates, which may have caused the modification in the results.

The Mn black series produced the most noticeable changes, particularly with the SO process. UV produced a greater change than HT in this case. Globally, C black revealed a less significant change than Mn black. This series was the least susceptible to SO and the most photosensitive (UV). The low value obtained with MP must relate to the formation of unstable reaction products and to the hypothetical attenuation of chromatic changes by the successive processes.

The almagra red series underwent the equivalent changes in each process, which made us assume that this pigment was highly susceptible to all the degradation agents.

We ought to add that the NA samples had been exposed to environmental conditions to obtain a better understanding of the chromatic changes which may occur naturally. It is necessary to increase the total exposure time in each artificial ageing process because, in each case, the attained variations were similar to those reached naturally.

CONCLUSION

This study intends to characterise the various pictorial layers of the socarrats, these being the ceramic support, the pictorial base and the coloured layers. On the other hand, replicas have been produced and submitted to artificial ageing to obtain better knowledge of the main alteration agents of the *socarrats*.

The characterisation of the socarrats has proved to be important to not only perform good conservation planning, but to also clear the debate about these works in ceramic technology theories. By correlating the data obtained by analysing this group of fragments, we can affirm that the ceramic body has been fired at a temperature of around 1000ºC, the paint layers had not been fired, and the white, red and black layers are constituted by carbonated lime, almagra red and carbon black, respectively.

Artificial ageing leads mainly to lighter colours in the pictorial layers, and causes loss of cohesion and adhesion between the black and red paint layers and the white one. We assume that this information is of greater importance when dealing with excavated *socarrats*.

ACKNOWLEDGEMENTS

The authors gratefully thank Josep Pérez Camps (Ceramics Museum Museo de Cerámica de Manises), María Luisa Martínez-Bazán (Laboratorio de óptica y colorimetría), Camilla Mileto and Maria Diodato (Unidad de investigación, restauración y diffusion del patrimonio arquitectónico. Loggia) and Francisco Roig (owner of a traditional alquería in Burjasot).

NOTES

[1]A Valencian word with two meanings: socarrat: n (1) (CERAM) Ordinary tile made of fired clay to cover the space between joists on the roof (2) (GASTR) Inferior portion of the paella's rice, a bit burnt, but edible (Lacreu, 1996:1893)

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Versión española

TITULO: ***Estudios de alteraciones y caracterización de los socarrats valencianos.***

RESUMEN: *La necesidad de desarrollar un procedimiento metodológico de restauración sobre un conjunto de socarrats procedentes de colecciones privadas y excavaciones arqueológicas llevadas a cabo en Manises, junto con la escasez de estudios relacionados con este tema, han determinado llevar a cabo una investigación sobre sus materiales y tecnología de producción.*
Los socarrats son piezas cerámicas poco estudiadas y sobre las que existe gran controversia a la hora de definir su técnica, parece ser que su método decorativo estaría más vinculado con el de la pintura mural que con el cerámico propiamente dicho siendo por ello, cerámicas de singularidad excepcional y que plantean gran dificultad a la hora de ser intervenidas.
La investigación se ha llevado a cabo sobre dieciséis socarrats valencianos procedentes del Museo Municipal de Cerámica de Manises mediante diversas técnicas analíticas: microscopia óptica, microscopia electrónica de barrido y microanálisis por dispersión de energías de rayos X, espectrometría de infrarrojo por transformada de Fourier y voltamperometría de micropartículas. El cuerpo cerámico constituye el soporte de la capa blanca de cal carbonatada sobre la que se aplicó la decoración a base de óxidos de hierro y negro carbón. El deterioro de la policromía es muy notable con pérdida de cohesión y adhesión de los pigmentos. Además, y como consecuencia de los factores ambientales, se ha detectado la conversión de calcita en yeso. El contenido en sales solubles que se ha hallado es bajo y sus tratamientos de remoción junto con los de la suciedad superficial deben de tener en cuenta este dato. El estudio se completó analizando el cambio cromático experimentado en una serie de probetas reproducidas con la técnica de los originales socarrats tras ser sometidas a procesos de envejecimiento artificial.

PALABRAS CLAVES: *socarrats, cerámicas, pintura mural, cal, alteración, caracterización*