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ARCHAOMETRIC ANALYSIS OF WALL COATINGS FROM THE CHALCOLITHIC SITE OF SU CODDU (SARDINIA, ITALY)

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ABSTRACT

This paper addresses the archaeometric analysis of several wall coatings applied to diverse architectural structures (wells, silos and domestic spaces) from the Chalcolithic site of Su Coddu/Canelles (Sardinia, Italy; c. 3400-2850 BC). The study of the samples was carried out by means of optical microscopy by thin-section analysis, micro X-Ray Diffraction and Scanning Electron Microscopy combined with Energy Dispersive X-Ray Spectroscopy. The study showed the application of successive layers of different thickness and granulometry to isolate the architectural structures. On the one hand, up to two layers made with a Tertiary fossiliferous marly clay as plaster are documented. On the other hand, the application of a very thin final layer very rich in calcite is observed in the majority of the samples studied. The analyses conducted evidence certain variability in the technological choices made by the craftpeople. However, it is also observed a clear adaptation of the properties of the studied materials to the insulating and waterproofing function that they played in such architectural structures.

KEYWORDS: thin-section analysis, SEM-EDX, micro-XRD, provenance, technology, clay coatings, limewash coatings

1. INTRODUCTION

The aim of this paper is to address some of the technological choices used in the manufacture of clay and lime coatings that were applied on the walls of diverse kinds of structures in the Chalcolithic archaeological site of Su Coddu/Canelles (Sardinia, Italy; c. 3400-2850 BC). This study continues with previous preliminary analysis conducted on coatings of other architectural structures from this site (Mameli and Melis, 2008). The final aim of our study¹ consists in identifying the kind of raw materials used to coat the structures as well as to determine their potential sources of origin. Furthermore, we will approach the compositional variability existing between the samples of the diverse structures studied and the procedures used to stick the coatings on the walls. Finally, chemical analysis of the limewash coatings were conducted by means of Scanning Electron Microscope with Energy Dispersive X-ray Spectroscopy (SEM-EDX) in order to calculate the hydraulicity index and to address some of the physical properties of the materials and their functional efficiency. Micro X-ray diffraction (XRD) was also used for determination of some crystalline components of the limewash coatings.

The use of clay in building was well established in prehistory, as demonstrated by several categories of finds. This material was used in the construction and refinement of walls, floors and ceilings; they include mud bricks, daub with plant imprints and plaster. In most cases, the raw material was unfired and any eventual firing was accidental.

In Sardinia the use of clay is well documented in both prehistory and proto-history, in particular on the floodplains in the south of the island (Campidano); in the settlement of Su Coddu the wealth of data collected during the excavations conducted by Maria Grazia Melis confirmed the use of mud bricks of varying form and dimensions. The large number of clay fragments showing plant imprints as well as fragments of wall plaster throw light on standing structures in organic materials that were mixed and coated with clay (Melis, 2010).

The paucity of systematic studies on the numerous finds belonging to these categories is partially compensated by the information provided by the representations that were sculpted, incised or painted within Neolithic rock-cut tombs, which provide insights as to architectural elements in wood and clay.

The use of wall plaster is well documented on a religious monument of enormous relevance to Sar-

dinian and Mediterranean prehistory; the terrace of the shrine at Monte d'Accoddi. In the earliest building phase (first half of the 4th millennium cal BC) the entire monument was covered in red painted wall plaster. Thin section analysis of two samples using a petrographic microscope showed the presence of Cainozoic limestone, as well as a microcrystalline calcite binder (Mannoni, 1987 and 1992). The presence of plaster fragments is also noted in the prehistoric cave at Guano: the archaeological deposit has been heavily damaged in recent times; therefore it has not been possible to establish its precise chronology or the characteristics of the structures from which the fragments result (Castaldi, 1980). Mud bricks and beaten earth floors can be found in Nuragic contexts from the Bronze and Iron Ages and they were still commonly used in traditional Sardinian dwellings of the last century.

The earliest documented use of raw earth and lime plaster is found in PPNB contexts in the near East (Beidha and Jericho; Aurenche, 1981) and from the Balkans. In the western Mediterranean the first earthen buildings are datable to the early Neolithic in France and the Iberian Peninsula (Wattez, 2003; Gómez Puche and Diez Castillo, 2005). The use of unfired clay in construction has been recognised in various contexts on the Italian peninsular starting from the Neolithic (e.g. Peinetti, 2014; Venturino Gambari et al., 2002; Fronza et al., in press; Rossi et al., 2013; Fiorentino and Muntoni, 2002). The data refers to vertical (walls) and horizontal (floors and hearths) elements of buildings. Numerous archaeometric and technological studies have been produced on these categories of material (Feneuille et al., 2016; Peinetti, 2014 and 2016, *ivi* bibliography).

Research and available data on the use of lime in the western Mediterranean basin is rarer. In Bronze Age contexts it's use has been speculated in beaten clay and lime floors at Cava d'Ispica-Baravittala, Modica (Di Stefano, 2008) and Cannatello, Agrigento (Cultraro, 2015; Mosso, 1907). The use of clay carbonates has been hypothesized in Middle and Recent Bronze Age contexts in northern Italy (Cesena-Foro Annonario; Peinetti, 2016). Lime-based plaster is noted in pre-Roman period southern France starting from the 5th century BC (d'Ovidio, 2016), data confirmed through archaeometric analyses. The practice of using lime in architectural structures appears to begin earlier in the Iberian peninsula, where in the *Levante* area it appears in an advanced phase in the 3rd millennium cal. BC (Jover-Maestre et al., 2016). In more recent times lime was employed to coat basins for the production of wine during the Iberian and Punic periods (Pérez Jordà, 2000; van Dommelen et al., 2008).

¹ Maria Grazia Melis is the author of sections 1, 2 and 6. Daniel Alberio Santacreu is the author of sections 3 to 6.

2. SAMPLES SELECTED AND ARCHAEOLOGICAL CONTEXT

We have conducted the petrological, mineralogical and chemical analysis of six samples related to wall coatings recovered from several architectural structures of the archaeological site of Su Coddu/Canelles (Fig. 1). This settlement is located in southern Sardinia, close to a large number of marine and inland water bodies. Radiocarbon dating of bone fragments recovered from diverse structures indicates that the village was occupied over a long period. The oldest part of the settlement (Su Coddu) was founded during the first half of the 4th millennium cal. BC (Ozieri I phase). The settlement gradually extended to the south during the course of the Early Copper Age (Ozieri II phase) and occupied a new large area (Canelles) of about approximately 1.5

hectares, which was still inhabited during the first centuries of the 3rd millennium cal. BC (Melis, 2013).

The samples analyzed in this paper were recovered from a sector of the southern area of the site (lotto Badas) in which 12 structures were identified (Melis, 2005). The materials selected are associated with four of these structures: structures 39 (C-3), 46 (C-6), 47 (C-1, C-2, C-4) and 48 (C-5) (Fig. 1). These structures are partially dug in the ground and have diverse shape. They have been mainly interpreted as wells and cylindrical silos (structures 39, 47 and 48). However, another sample is associated with a more complex structure (structure 46) that has a circular main room with other structures related to diverse functions, silos and ovens, attached. Organic matter imprints can be observed in many mud fragments, adobe bricks and coatings related to these structures, thus evidencing the use of both, mud and plants to construct the walls.

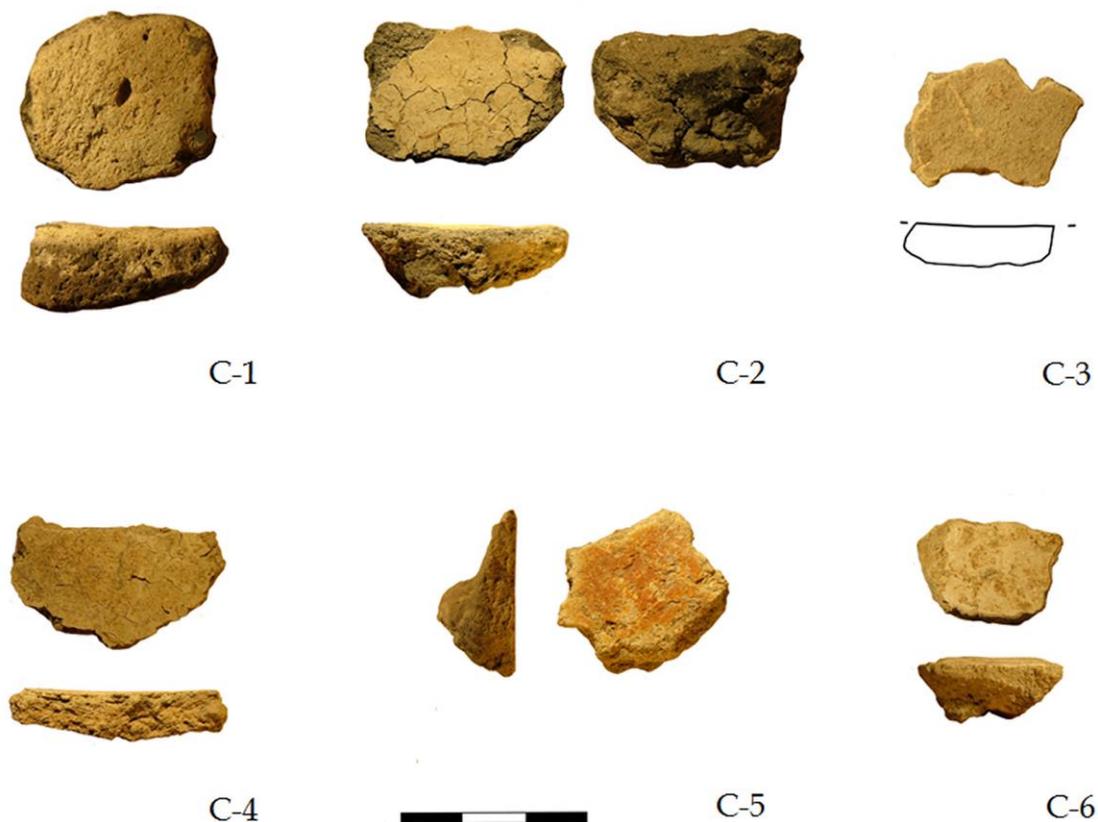


Figure 1. Photograph of the wall-coating samples analyzed recovered from the site of Su Coddu/Canelles.

3. GEOLOGICAL SETTING

According to Carmignani et al. (1996) and Barca et al. (2005), Su Coddu/Canelles is located in an environment with Quaternary alluvial terraced deposits with abundant microfossils and granulometric het-

erogeneity. These deposits consist of coarse gravel, sand, aeolian sandstones, conglomerates, biocalcarenites and muds. The detrital materials originate in the granitic and metamorphic formation of the Sarrabus. This formation yields to a flat region on the west side of the study area, whereas the mountain

formations are located in the east. Alluvial and coluvial deposits with coastal gravel, sand, silt, sandy and silty dark clays and mud rich in organic matter with fragments of marine and lagoonal shells have been documented in the south of Selargius.

The presence of substantial Lower and Middle Miocene deposits of marine origin with a contribution of continental materials was observed approximately 2.5 km to the east, north and west of the archaeological site. However, these gray marl clays with sand related to the Argille di Fangario deposit are also found beneath the Quaternary alluvial deposits in the vicinity of Su Coddu/Canelles. A deposit (Marne di Gesturi) formed by yellow marls with sand and silt, sandstones, conglomerates, limestone and calcarenites is also observed in this area.

At a greater distance, different types of non-calcareous deposits have been documented. On the one hand, there are Paleozoic metamorphic deposits approximately 7 km to the northwest. The Upper Ordovician - Lower Carboniferous formation of Pala Manna, which is formed of meta-sandstones, meta-siltites, quartzite, phyllite, meta-conglomerates, meta-volcanic rocks, metapelites and clays, should also be noted. Within this formation, Carboniferous-Permian materials with acidic rhyolites and to a lesser extent dacites also appear. On the other hand, there are Hercynian plutonic intrusive rocks associated with the unit of San Gregorio approximately 15 km to the west. Several porphyritic microgranites and, primarily, biotitic leucogranites and granodiorite outcrops form this geological unit. In addition, the previously noted Carboniferous-Permian acidic rhyolites and dacites also occasionally appear in this formation.

4. ANALYTICAL METHODS

The clay coatings studied have usually shown three layers with a clear graduation of grain-size from base to uppermost surface: 1) Coarse coat: a rough layer of clay which is applied on the smooth surface of a wall; 2) Fine coat: a series of muddy layers very similar to the previous layer but finer textured; 3) Patina: a submillimetric layer made of lime mortar and very fine sand.

The samples were impregnated with epoxy resin and sawn. Thin sections were prepared for petrographic and textural characterization by optical microscopy. The optical examination of thin sections was performed using a petrographic microscope Leica DM2500P, which incorporates a micrometer. The lenses ranged from x16 to x400 magnifications. Photomicrographs of the samples were taken with a Leica DFC295 digital camera. The quantity of each compound was established using comparative charts (Matthew et al., 1991). Descriptions of the thin sec-

tions were made following the procedure developed and detailed by I. Whitbread (1995). In addition, textural concentration features (TCF) were characterized considering the observations made by I. Whitbread (1986). In addition to the conventional petrological analysis, we also conducted micropaleontological analysis of constituent calcareous microfossils by means of thin section.

In addition to thin section analysis, polished cross-sections of five samples were mounted in resin blocks in order to characterize the chemical composition of the patina layer by means of SEM-EDX using a Hitachi S-3400-N scanning electron microscope equipped with a liquid-nitrogen-cooled detector system Bruker RXEDS using 15 Kv of tension. The detection limit of the instrument was $> 0.3 \text{ wt}\%$. All quantitative results (Table 1) are reported as weight per cent oxides with oxygen determined by stoichiometry, the reported values are the average of between two and three analyses spread over the lime layer. The final aim of this analysis is to record the concentrations of the major and minor chemical elements in order to calculate the hydraulicity index (i) of the material (Válek et al., 2012) by means of the following formula (Boyton, 1966):

$$i = \frac{\%Al_2O_3 + \%Fe_2O_3 + \%SiO_2}{\%CaO + \%MgO}$$

This equation allows us to know the amount of clay present in a limestone with respect to its quantity of lime (CaO), thus allowing us to calculate the hydraulicity index of the lime mortar.

The determination of some crystalline components of the patina layer was conducted by means of micro X-ray diffraction (XRD). The instrument used to perform such non-destructive analysis was a Bruker D8 Advance equipped with a silicon strip detector and a vertical XYZ sample stage. The instrument was equipped with an optical video recording microscope and laser alignment device to allow accurate alignment and recording of the area from which diffraction patterns were recorded. Diffraction data was recorded with Cu K α radiation, tube running at 40 kV and 40 mA. The incident beam was collimated to a diameter of 500 μm . Measures were taken from 3 to 70° using a collection time of 384 s. We used X-powder software (Martín, 2004) to evaluate the crystalline phases against intensity and spacing tables from the data bank drawn from the Joint Committee of Powder Diffraction Standards (JCPDS 2003).

5. RESULTS

All the samples studied have been classified in the same petrographic fabric, since no differences in the clay materials used to coat the walls of the different structures studied could be observed.

5.1. Coarse coat

The few voids observed in the microstructure of this layer (5-15%) are predominantly macro and meso-vughs and common macro-vesicles. The pores are normally open-spaced and oriented along the surface margin of the wall, whereas the non-plastic inclusions (15 to 30%) are open to single-spaced and non-oriented. The groundmass is dominant, light/dark brown to yellow/yellowish black (PPL, x400) and light/dark brown or black (XPL, x400). This is a crystallitic b-fabric with a calcareous matrix and an optically active micromass. Regarding the micropaleontological identification (Albero et al., 2016), we highlight the presence of bivalve fragments, planktonic (e.g., *Globigerina praebulloides*, *Praeorbulinasicana*, *Praeorbulinaglomerosa*, *Globigerina* sp., *Globorotaliasp.*) and benthonic (e.g., *Lenticulina*) foraminifera and sponge spicules.

The inclusions have a bimodal to polymodal grain-size distribution with poorly sorted sub-rounded to rounded coarse inclusions and rock fragments (very coarse to fine sand) set in a finer-grained groundmass with sub-rounded or rounded inclusions. The fine fraction is dominant with a modal grain size of 0.12 mm in the long dimension (c:f_{10μ}:v = 22:68:10 to 33:60:7). Monocrystalline quartz is dominant in the coarse fraction, although K feldspar is also commonly observed. Plagioclases, calcimudstone, chert, sandstone/metasandstone, biotite flakes, pure amorphous nodules and polycrystalline quartz are also present in the sections. In addition, shale, siltstones, augite, felsic and basic igneous rock fragments and volcanic rock fragments are rarely present in certain samples. A number of elongated pseudomorphic amorphous depletion features related to organic fibers could also be identified. Finally, TCF related to argillaceous rock fragments could be rarely documented in few samples. These TCF are sharp or clear rounded with equant to prolate shape, high optical density and features discordant with the matrix and finer-textured. The groundmass is optically active to moderately optically active and brown (PPL, x400) to dark brown or black (XPL, x400).

5.2. Fine coat

The micromass and porous microstructure of this layer are highly similar to those of the previous layer although in this case the voids are open to single-spaced and poorly oriented along the surface mar-

gins of the wall. Several pores have completely alloctonous secondary calcite. The variations between the samples occur in terms of microstructure and the number of non-plastic inclusions. The non-plastic inclusions have the same characteristics as those in the coarse coat with respect to their orientation, size and shape. There are fewer inclusions (10-15%) in this layer, and the inclusions have a bimodal grain-size distribution with sub-angular to rounded coarse inclusions and rock fragments set in a finer-grained groundmass (c:f_{10μ}:v = 15:75:10 to 10:60:30). The mineralogical composition is analogous to that observed in the previously discussed layer. However, the previously observed rare to absent basic igneous rock fragments were not identified in this layer. In one case, there is a sharp to merging well-rounded TCF (i.e., a clay pellet) of up to 3.2 mm length with a prolate shape and neutral to high optical density with features concordant with the matrix.

5.3. Patina

This layer was preserved in five of the six samples selected. There are rare voids in the microstructure of this layer that comprise dominant open-spaced micro-vughs and micro-vesicles. In most cases completely alloctonous secondary micritic calcite fill the pores. The non-plastic inclusions (< 5%) are open to double-spaced and poorly oriented in parallel along the surface margins of the wall. The groundmass is predominant and homogeneous throughout the samples. The variations are in terms of microstructure and the amount of non-plastic inclusions (ranging from 1 to 5%) the color is light brown/yellow (PPL, x400) to yellowish-brown (XPL, x400). The micromass is optically active, representing a crystallitic b-fabric with a calcareous matrix. Foraminifera could not be identified in this layer.

The inclusions have a unimodal grain size distribution with scarce poorly sorted sub-angular to rounded fine inclusions and rare rock fragments (< 0.5 to 0.15 mm; medium to fine sand) set in a finer calcareous groundmass. The fine fraction is dominant in the sample (c:f_{10μ}:v = 0:98:2 to 5:90:5) with a modal grain size of 0.06 mm long dimension. The petrologic composition is well related to the observed in the previous layers but in a much finer silty fraction. The inclusions are composed by monocrystalline quartz, K feldspar and plagioclase, mica laths (biotite, muscovite and rarely chlorite) and few to predominant calcimudstone. Also very rare to absent sedimentary rock fragments (such as siltstone, chert and claystone) could be observed.

The micro-XRD analysis of the patina indicates that calcite is the major component in this layer, though quartz, anhydrite and traces of albite feldspar are also present (Fig.4). As stated before, the

chemical composition of the patina was analyzed by means of SEM-EDX in order to characterize the concentrations of the major and minor elements. Results obtained through this method show a significant concentration of CaO (average = 44.8%) in this layer (Table 1), which is in agreement with the XRD analysis.

The high amount of CaO recorded, the presence of a matrix with abundant micritic calcite in thin section and the dominant calcite peaks observed in the XRD diffractograms point to the use of lime to make this layer. The absence of calcium oxide in the micro-XRD analyzes may be consequence of recarbonation processes of the lime after its application (or perhaps during its deposition). Furthermore, the absence of clay minerals in the XRD diffractograms suggests that this material suffered some kind of heating process. As is common in prehistoric mortars, lime would have been present in a variable and scarce quantity, mixed together with other elements. In any case, we must be aware that the identification of lime in prehistoric coatings is always a tricky issue (see Jover Maestre et al., 2016). The development of further analyses by means of other analytical techniques (e.g. Fourier Transformed Infrared Spectroscopy, Thermogravimetry and Thermal Differential Analysis) is, therefore, advisable to confirm the use of this material in the production of this patina.

The data obtained from SEM-EDX analysis was also used to calculate the hydraulicity index (i) of the material. According to this index, the higher is the i value, the greater is the chemical resistance and the capacity of the material to harden in a humid or water-saturated environment. This index, which is related to the purity of the limestone used in the production of the lime, should be below 1.2 (Piovesan et al., 2009). The hydraulicity index values obtained (Table 1) show that all the samples analyzed (except C-3) have an i that is between the range $0.7 < i < 1.1$. These values evidence that all the samples had a high hydraulic property and were, therefore, perfectly functional.

The results show the desire and success of the inhabitants of the site for waterproofing these structures. Thus, they produced a material that favored the absorption and evaporation of moisture, providing thermal insulation to the structures and reducing the risk of surface condensation. In addition, the significant amount of silicates and aluminates observed in the microanalyses suggests that the craftpeople used argillaceous limestone containing some clay in order to create a lime with hydraulic properties. This kind of hydraulic lime has the ability to set in wet conditions, but it is also a substance that hardens very fast (between 5 and 15 minutes).

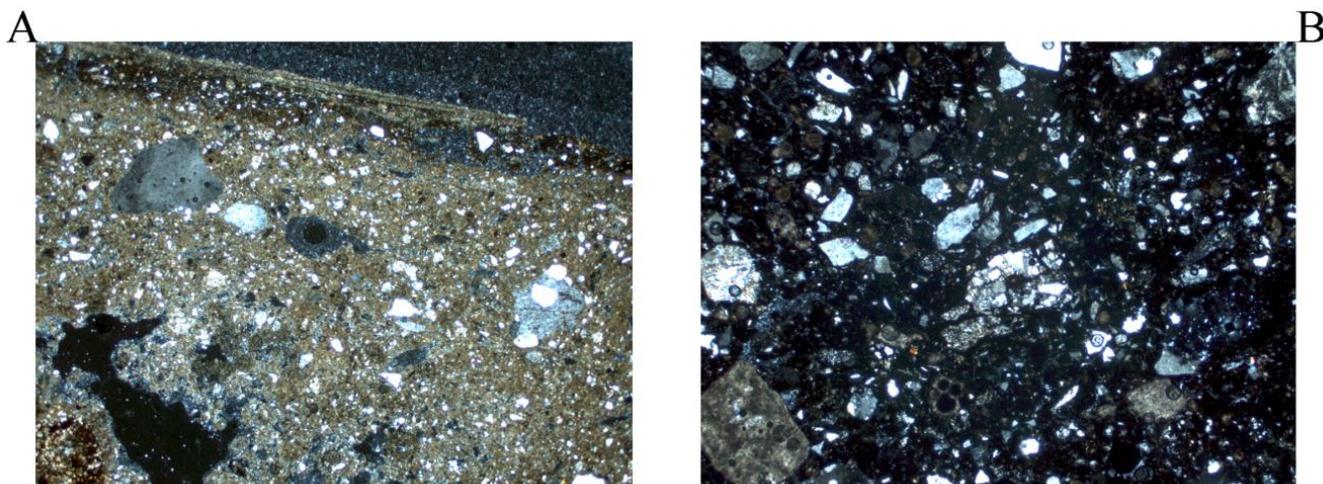


Figure 2. A) Thin-section micrograph of the three layers identified in a clay coat; Note the thin-textured paste of the fine coat and the patina layers and the different color of the fine coat layer (image width = 8.3 mm; XPL); B) Thin-section micrograph of the coarse coat layer showing the presence of calcimudstone and planktonic foraminifera (image width = 3.3 mm; XPL).

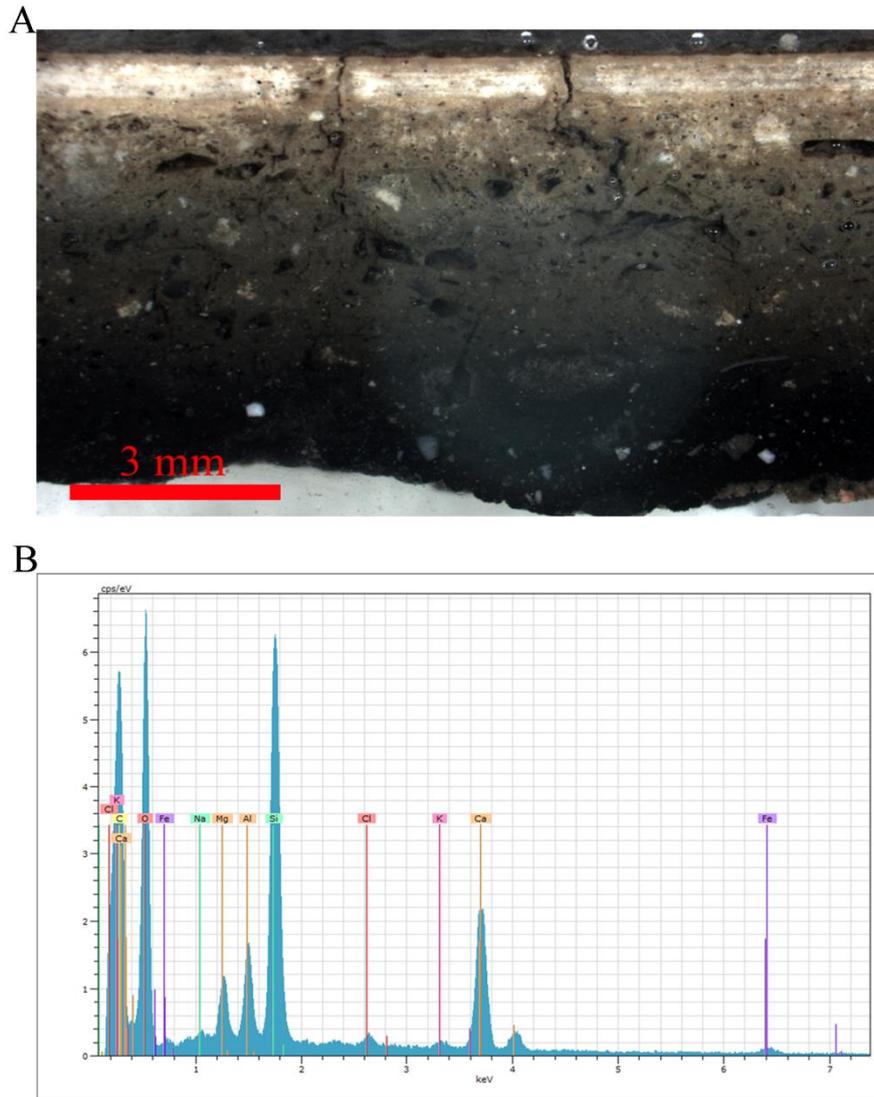


Figure 3. A) Photomicrograph of a polished cross-section taken with binocular microscope showing at low magnifications the features of the milk lime layer and the clay coating. B) SEM-EDX spectra showing the elements identified in the microanalysis conducted on the patina layer.

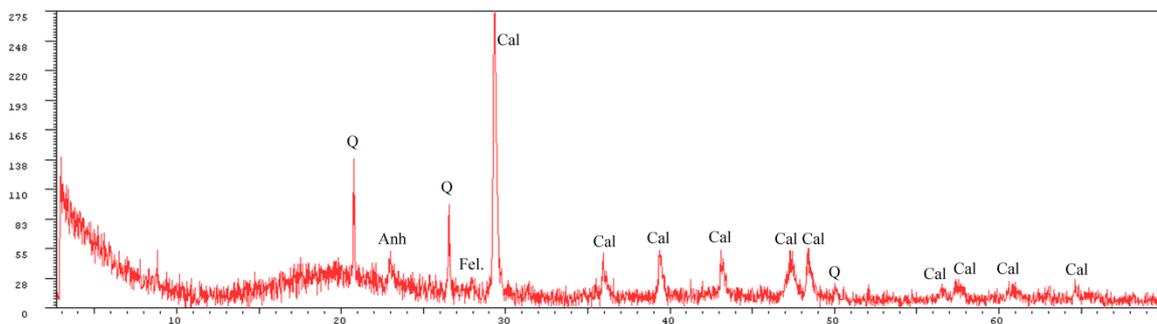


Figure 4. Crystalline phases detected in the patina layer by means of micro-XRD analysis: Cal = calcite, Q = quartz, Anh = anhydrite, Fel = Feldspar (albite).

Table 1: Results of the microanalysis conducted by means of SEM-EDX showing the hydraulicity index (i) and the chemical composition (wt% normalized to 100%) of the patina layer identified in the wall-coatings studied (n.d. = non-detected):

Sample	Structure	Al ₂ O ₃	SiO ₂	CaO	Fe ₂ O ₃	Na ₂ O	MgO	SO ₃	Cl	K ₂ O	i
C-2	47	6,4	31,1	51,4	4,5	0,9	3,1	2,1	n.d.	0,5	0,8
C-3	39	8,9	47,9	28,6	4,8	1,2	6,9	n.d.	0,7	1,0	1,7
C-4	47	7,2	35,9	46,5	3,4	2,0	2,7	n.d.	0,6	1,7	0,9
C-5	48	6,9	30,2	53,5	4,6	1,4	1,5	n.d.	0,6	1,3	0,8
C-6	46	7,4	33,9	43,8	6,1	2,3	1,6	1,1	1,1	2,7	1,0

6. CONCLUSIONS

6.1. Technological aspects

The analysis of the samples studied demonstrates that the wall coatings were applied by using several layers (between two and three). First, a thick and coarse-textured clay-coating rich in foraminifera (among other microfossils) and organic matter was applied over the walls. The presence of organic matter in the paste can be inferred by the blackish color of most of the samples (except C-4 and to lesser extent C-5) and the presence of organic matter fibers in several samples (e.g. C-1, C-3 and C-6).

The second layer (fine coat) is quite variable as regards its thickness (0.6-4.9 mm) and coloration. The petrological composition of this clay-coating is quite similar to the latter, but finer-textured and with only very rare organic matter fibers. These features generate a proper base layer on which subsequently apply the milk lime coating (patina). Another option observed in some of the samples (C-4 and C-5) recovered from the silos (structures 47 and 48) consists in applying the limewash directly on the coarse coat, without the need of producing the finer layer. Finally, in the sample C-1 only the two clay-coatings (Coarse and Fine coats) were preserved, there are no signals of the patina layer in the thin sections studied from this sample.

The patina consists in a very thin (between 0.1 and 0.7 mm of thickness) refined calcareous layer that has very few inclusions mainly sorted in a silt fraction and rare medium to fine sand. However, the patina of the sample C-4 is less refined and has a larger amount of calcareous inclusions sorted in the silt fraction and a higher porosity degree. In addition, the sample C-5 stands out due to the presence of some inclusions in the medium and coarse fraction. At Su Coddu/Canelles this limewash was added in the outer layer of the coats with the intention of

creating a homogeneous and solid surface, protecting the interiors of habitations from high external temperatures. In the case of its use on the interiors of silos it probably had an isolating and sterilising function. The application of such limewash coatings would have required a deep understanding of certain technical aspects concerning the properties of the materials. This knowledge probably had its roots in the local Final Neolithic (first half of the 4th millennium cal BC): in the cultural sphere of the Ozieri I *facies*, when it is common to find a limestone paste, applied before firing, in incised and impressed pottery. But above all it is Monte d'Accoddi the site which offers the most significant evidence of the former use of plaster in this period.

As it can be seen, although the same marly clays and calcareous materials were used to apply the coatings over the walls of the structures, there was certain variability in the technological choices used. This diversity is in agreement with the petrological analysis conducted on the pottery recovered from this archaeological site (Albero et al., 2016).

6.2. Provenance of the raw materials

The petrological and paleontological analysis of the clay coatings of Su Coddu/Canelles enables us to determine the origin of the raw materials used in the production. All of the studied samples are characterized by a crystallitic b-fabric with an optically active groundmass. The samples have a calcareous matrix rich in foraminifera as well as abundant silt and fine sand that include sedimentary, granitic and to lesser extent metamorphic rock fragments. Thus, ancient craftspeople used a clay deposit of marine origin with alluvial sediments that primarily consisted of quartz-feldspar sandbars derived from the erosion of felsic igneous rocks and clasts of metamorphic rocks with a low degree of metamorphism. These alluvial materials were redeposited in the marine clays. In

addition, it was well documented the presence of volcanic ash in the sections that are related to the presence of pyroclastic facies associated with volcanic eruption events. Finally, the occurrence of the foraminiferal taxa reported is consistent with the microfossil record of the Miocene deposits of Marne di Gesturi and Argille di Fangario Formations.

In sum, the features of the marly clays used to produce the artifacts are in agreement with the composition of the Miocene deposits associated with the Marne di Gesturi, which appears in a significant area of the surrounding territory of the site (< 1 km), or the Formazione di Fangario. The latter deposit is well documented in the vicinity of the archaeological

site, both in the west and under the Quaternary alluvial sediments (Mameli and Melis, 2008). The exploitation of these clay deposits by the individuals who inhabited the area would have been far easy thanks to the presence of a stream located in the east side of the site as well as a small swamp (disappeared in Modern times) placed in the west side. These deposits have abundant planktonic foraminifera, pelagic fauna, mollusks, echinoids, benthonic fauna and corals. Additionally, they possess pyroclastic and epiclastic facies that consist of interbedded conglomerates and sands in a clay matrix with clasts of volcanic rocks formed by pumice, sanidine crystals, plagioclase, biotite, quartz and volcanic glass.

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