



Earthen modeling in the depths of the Sierra Mixe of Oaxaca, Mexico: investigating the production process of unfired clay reliefs and sculptures in Mesoamerica's underworld

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ABSTRACT

This paper addresses the mineralogical and chemical composition of earth pastes used in the production of selected unfired clay reliefs and sculptures modeled within a cave in today's Mixe (Ayuuk ja'ay) territory of Oaxaca, Mexico, through a multi-technical approach comprising Optical microscopy (OM), Portable X fluorescence spectrometry (p-XRF), Energy Dispersion Microanalysis by Electron Probe Micro Analyzer (EPMA), UV-fluorescence microscopy, Micro-Raman Spectroscopy, X-ray fluorescence (XRF), X-ray powder diffraction (XRPD), Differential scanning calorimetry (DSC) and thermogravimetry (TG). The analyses demonstrated that no external materials were added to the mix and that manganese deposits were exploited *in situ* and used as black pigment. In addition, by studying the differences in the natural stratigraphic structure between clay relief samples and those of the terraces on which the clay figures were shaped, we attested that such terraces are natural sediment deposits. The study also includes the first systematic radiocarbon dating and identification of charcoal fragments, thus providing an initial chronological framework for the production of unfired clay figures and the use of this Mesoamerican sacred underworld space.

1. Introduction

From everyday domestic vessels to highly ornamented polychromatic sculptures, Mesoamerican cultures have been prolific designers and producers of fired clay objects, giving rise to a fertile variety of styles across Mesoamerica's geography through time. Such expertise has demanded exploring and understanding the properties of soft and plastic raw earthen materials, the addition (and proportions) of temper to modify its consistency and enable its manipulation, shaping the resources manually or by molds, mastering the heat workability to harden and preserve those objects, and the management of slips and pigments

for decoration when necessary. Nonetheless, unfired clay artefacts have been noticed less in the archaeological record, probably due to conservation issues. In some rare contexts, such as El Zapotal, Veracruz (ca. 800–1000 CE), soft and unfired earth was deployed by the Mixtequilla cultures to model an elaborate 1.6 m sculpture of the God of the Death. This green, red, white, black and blue painted anthropomorphic figure was discovered in association with mural paintings and human remains within a monumental 76 m long earthen platform (Ceja Tenorio and Barjau, 1992: 99; De la Fuente et al., 2003: 90; Gutiérrez Solana and Hamilton, 1977: 29–30; Wyllie, 2010: 213).

Other examples come from Cacaxtla, Tlaxcala (ca. 650–950 CE),

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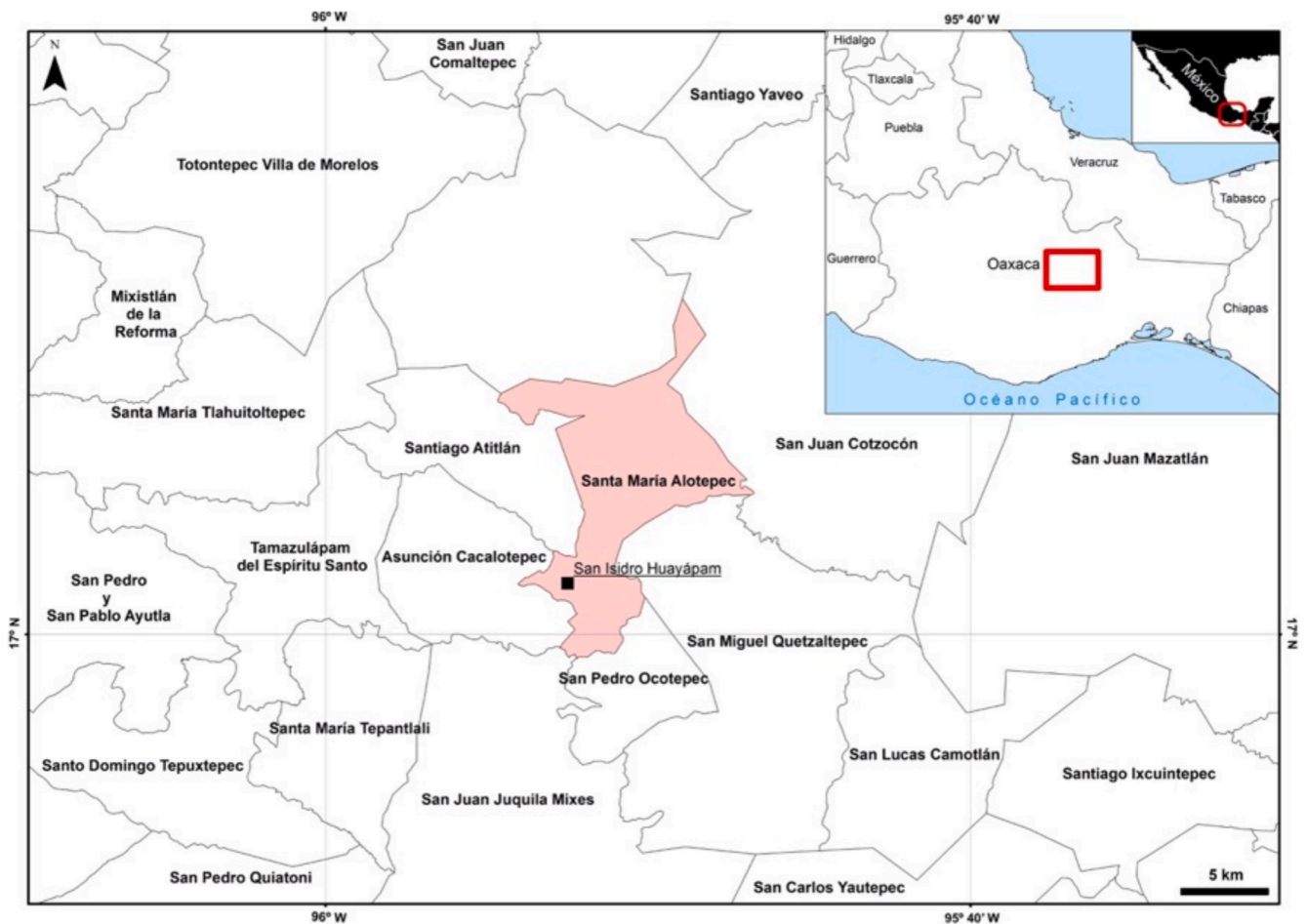


Fig. 1. Map showing the location of the study area.

where a “life-sized three-dimensional polychrome unfired clay human figure” with a rich attire (i.e., collar and twisted waistband) was discovered during rescue archaeology activities in a fine bed of sand within a pit east of Building A (Nagao, 2014: 118, Fig. 92). Near Building B, fragments of unfired clay objects in the shape of corn, shells, flowers, and a serpent had traces of blue, yellow and red pigment (Nagao, 2014: 520). Not only were three-dimensional unfired clay objects recovered, but also two unfired clay reliefs:

- The first shows the lower part of a standing human figure flanked by plants wearing sandals and a garbed attire (perhaps representing a textile). This clay relief was attached with lime plaster to an exterior pier of Building E (Brittenham, 2009: 145, Fig. 2).
- The second is a highly attired human figure in profile in a dynamic half-lotus seated position on a throne wearing a headdress, necklace and loincloth. According to Debra Nagao (2014: 521), the relief shares elements with Mayan culture (i.e., *cauac* monster). Interestingly, this dark red clay was applied on top of a mural painting flanking the door of Building A.

These examples suggest that some Mesoamerican cultures produced elaborate, large, unfired clay sculptures and reliefs for specific purposes and contexts, regardless of their fragile nature, thus suggesting deliberate purpose and meaning behind this choice. However, for shaped earthen materials not yet fired to persist, temperature and humidity must be constant; those conditions occur in closed environments such as caves (see Bourges et al., 2014; Brunet and Vidal, 1980: 99–103 for discussion on Palaeolithic caves). Yet, there is also scant evidence of unfired clay items in subterranean contexts in Mesoamerica. One is that

reported in Footprint Cave in Belize as a “unique grotesque face or mask, modeled in a clay bank along the west wall” (Graham et al., 1980: 158) of a ledge about 450 m from the entrance. This 30 cm high and 25 cm wide “mask” was described as having “humanoid” features such as prominent ridges around the eyes with a tongue hanging out from the open mouth. Most interesting is the mention of a headdress expanding outwards and upwards with a flattened top. Furthermore, the archaeologists suggested that a black stain on the ceiling on top of such a “crown” might have resulted from something being burned on the flattened surface of that figure. Thus, given the scant examples of raw earthen figures in the archaeological record, the presence of a prolific repertoire of clay reliefs and sculptures preserved within the depths of the Mixe Highlands in Oaxaca offers here the possibility to explore the procurement of materials and production strategies of unfired clay figures in sacred caves in Mesoamerica for the first time.

1.1. The study area

Located southwest of the Middle Mixe Subregion of Oaxaca, Cueva del Rey Kong-Oy (or Kondoy) lies in the land of the town of San Isidro Huayápam (*Pox am*—the place of guavas in the Ayuuk language), positioned at 1050 m above sea level (masl) (Fig. 1) and currently closed to public access. Many caves and other landscape elements in the Mixe territory are linked to the mythical character of Rey Kong-Oy for the Ayuuk ja’ay people or Mixes. Following the traditional authorities’ recommendations of the community of San Isidro Huayápam in 2018, Kong-Oy is used here instead of Cong-Hoy. Some other local people have suggested Konk-Oy as a spelling alternative, and others say that Kondoy is the most ancient version of the name. Nonetheless, this cave is also

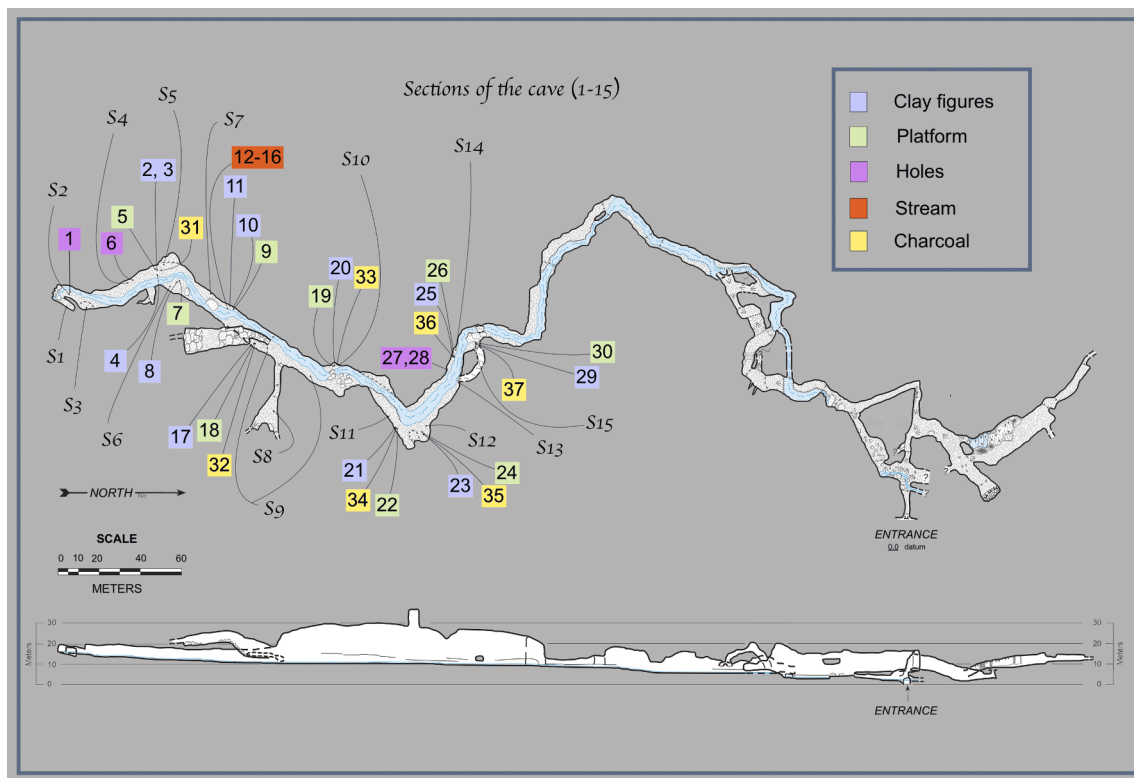


Fig. 2. The cave plan shows the sections of the cave and sampling areas where the study took place. Compass and Disto survey by Jason Ballensky, Tamara Ballensky, Phillip Rykwald, Rob Spangler and Elliot Stahl. Cartography by Bob Richards (Modified from Ballensky, 2012).

known as Cueva del Diablo, “Devil’s Cave” (see Zubieta, 2021, for further discussion regarding the current Indigenous perception and importance of the cave).

The small entrance to the cave is at the base of a Lower Cretaceous limestone wall and 60 m from a permanent river known locally as the Río Grande. Due to the topographic characteristics of the Sierra Mixe, the territory has a remarkably varied climate depending on the elevation of the terrain. While the high parts of the surrounding mountains are characterized by oak-pine forest, the entrance of the cave—positioned at 590 masl—displays an array of fruit trees (i.e., sapote, banana, mamey) due to the confluence of Semi-warm humid with rain all year and Warm humid with abundant rains in summer at the bottom of the canyon (INEGI, 2010). Caves are semi-closed systems that buffer external variations and are characterized by remarkable thermal stability, particularly in temperate zones (Badino, 2004, 2010). Temperatures inside caves are regularly highly correlated with average annual surface temperatures (Dominguez-Villar et al., 2013). Given the varied climatic conditions in the locality, it is challenging to know precisely the temperature ranges of the canyon within which the cave is situated. However, weather stations in the Mixe region, less than 50 km from the cave (some currently not operating), give us an idea of the weather’s diversity. According to the *Comisión Nacional del Agua (CONAGUA)* (n.d.) based on the data reported in 1984, San Pedro and San Pablo Ayutla (2014 masl) had an average annual high temperature of 22.7 °C and a minimum of 14.5 °C; Santiago Choapam (900 masl) of 26.3 °C and 16.4 °C; San Miguel Quetzaltepec (1211 masl) of 27.2 °C and 14.1 °C; San Lucas Camotlán (1372 masl) of 26.6 °C and 11.2 °C, and Santa María Puxmetacan (342 masl) of 26.9 °C and 17.6 °C.

The cave’s gallery that concerns us here is projected at the end of the northern branch and runs towards the south with a depth of more than 500 m. It is in this last southern gallery where, from its discovery in 2011 (Ballensky, 2012; Winter et al., 2014; Zubieta, 2021), black drawings have been recorded (e.g. negative hand stencils, heads in profile, glyphs of place and others). Red rock paintings are also visible, such as a

negative hand stencil at the end of a narrow branch and a series of dots 10 cm in diameter at the deepest part of the cave, where human bones can be seen seemingly unconnected and fragmented (Zubieta, 2021: 1182). Some rock paintings are spatially accompanied by an extensive repertoire of clay figures, but no chronological or cultural association has yet been established.

1.2. Unfired clay figures’ repertoire

Since 2018, seventy-two clay figures have been recorded by the present project; mostly reliefs protruding more than half of their volume from the floor of female and male bodies with dimensions varying from 0.7 to 2.2 m in length, generally showing accentuated genitals, and six clay sculptures in the round of animals. The term *clay figure* is used here to refer, in general terms, to the plastic art of the cave divided by Leslie F. Zubieta (2021: 1175) into:

1. Human: Mostly found in a supine or frontal seated position with the back or head reclined against the wall. Human bodies with exposed genitalia were identified as twenty-two females, seven males, eight children, sixteen of unidentified sex, one hermaphrodite, and three heads/masks.
2. Animal: Regularly in a seated, prone, or crouching position; eight felines, one frog, one monkey-like, and two reptiles (possibly chameleons).
3. Architectural features: Particularly characterized by a 3.8 m long ballcourt with round corners, one miniature ballcourt/platform sculpted as a hollow relief, and two rectangular blocks that may represent an architectonic structure.

The clay reliefs and sculptures were modeled on the surface of elevated terraces of the same material (endokarstic clays) deposited on each side of the course of a narrow underground stream, and whose elevation varies between 1 and 1.6 m above the base of the stream. For

Table 1

List of analyzed samples, providing the section from which those were extracted and the sample number. Consecutive numbering 1 to 30 allows the reader to identify their location in Fig. 2. OM: Optical Microscopy; p-XRF: Portable X-ray Fluorescence Spectrometry; EPMA: Energy Dispersion Microanalysis by Electron Probe Micro Analyzer; XRF: X-ray Fluorescence; XRPD: X-ray Powder Diffraction; DSC-TG: Differential Scanning Calorimetry and Thermogravimetry; UVFM: UV-fluorescence microscopy; Micro-Raman Spectroscopy: μ -Raman.

No.	Section	Sample	Description	%RH	T °C	Analyses
1	2	S2-H	Bottom of the hole in section 2 (no clay figures here)	80.16	25.5	XRF, XRPD, DSC-TG
2	5	S5-5E	An eroded feline head fragment with a black patina collapsed on the floor	92.33	23	OM, XRPD, XRF, DSC-TG
3		S5-5 N	Feline face with black patina. Spalling from the head.	92.33	23	OM, EPMA, μ -Raman, p-XRF
4		S5-7E	An amorphous mass of material in front of the S5-5 feline	92.33	23	OM, XRPD, XRF,
5		S5-7P	Platform sample close to clay figure S5-7	92.33	23	OM, XRPD, XRF
6		S5-H	The deepest part of a hole	92.33	23	XRF, XRPD
7	6	S6-5P	Platform sample close to clay figure S6-5	92.52	23.40	OM, XRPD, XRF, EPMA, DSC-TG, UVFM, μ -Raman, p-XRF
8		S6-4E	Possible hand-made platform supporting a female figure	92.52	23.40	OM, EPMA, UVFM, μ -Raman, p-XRF
9	7	S7-2P	Platform sample close to figure S7-2P	72.31	26.31	OM, XRPD, XRF
10		S7-3E	Fragment of eroded human figure shoulder	72.31	26.31	OM, XRPD, XRF
11		S7-5E	A spalled fragment of an arm of a female figure	72.31	26.31	OM, XRPD, XRF
12		S7-1	The upper part of the stream profile	72.31	26.31	XRF, XRPD, DSC-TG
13		S7-2	25 cm below the previous	72.31	26.31	XRF, XRPD
14		S7-3	25 cm middle part of the stream profile	72.31	26.31	XRF, XRPD
15		S7-4	25 cm below the previous	72.31	26.31	XRF, XRPD
16		S7-5	The lower part of the stream profile (underwater)	72.31	26.31	XRF, XRPD
17	9	S9-8E	Support of a female's head figure against the wall	95.08	22.96	OM, XRPD, XRF
18		S9-8P	Platform sample close to figure S6-5	95.08	22.96	OM
19	10	S10-4E	Leg of a small female figure	88.11	23.25	OM, XRPD, XRF
20		S10-4P	Platform sample close to figure S10-4	88.11	23.25	OM, XRPD, XRF, DSC-TG

Table 1 (continued)

No.	Section	Sample	Description	%RH	T °C	Analyses
21	11	S11-1E	Fragment placed on top of a modeled cube. Perhaps the body of an animal was in process.	91.18	23.24	OM, XRPD, XRF, EPMA, DSC-TG, μ -Raman
22		S11-1P	Platform sample close to figure S11-1	91.18	23.24	OM, XRPD, XRF
23	12	S12-5E	Fragment- exterior part of the calf of a human figure	90.02	23.41	OM, XRPD, XRF
24		S12-5P	Platform sample close to figure S12-5	90.02	23.41	OM, XRPD, XRF
25	14	S14-1E	Fragment- inner part of female figure's thigh	89.09	23.53	OM, XRPD, XRF, DSC-TG
26		S14-1P	Platform sample close to figure S14-1	89.09	23.53	OM, XRPD, μ -Raman, XRF, EPMA
27		S14-H1	Dirt from the bottom of the hole (60 cm deep)	89.09	23.53	XRF, XRPD
28		S14-H2	Dirt from the bottom of the hole (60 cm deep)	89.09	23.53	XRF, XRPD
29	15	S15-2E	The distal part of a highly eroded human figure	83.92	24.57	OM
30		S15-2P	Platform sample close to figure S15-2	83.92	24.57	OM

practical reasons and to organize the data and the sampling process better, the southern corridor has been divided into fifteen sections in consecutive order since 2018 (e.g., sections 1, 2, 3, and so on; see Fig. 2, Table 1). The sections correspond to different terraces that are physically separated from one another.

Some better-preserved clay figures have decorative patterns carved on the clay on specific parts of the body (e.g., possibly tattoos or representations of body painting on the chest and arms) and regalia (e.g., headdresses, sandals). Moreover, red and black traces of pigment are still visible on some human faces, thus showing that some examples were also painted in the past. The fine details of the many clay figures suggest a multifaceted logistical strategy in their production. In this regard, by meticulously observing the modeled clay reliefs and sculptures within Cueva del Rey Kong-Oy, we have learned more about the past artisans' relationship with the local resources: clay was not the only material involved in the manufacture.

Thanks to the natural erosion, we could see the interior of the clay figures in some instances, thus providing us with a window to better notice the production process. For example, artisans shaped a jaguar sculpture in the round (Section 10) by plastering an existing stalagmite with clay (Fig. 3). Some features were made by using stone slabs to add volume, as evidenced by the eroded headdress of a female figure (Section 9; Fig. 21). Also, rounded pebbles were collected and mixed with clay to create certain body parts, as observed from the torso of a feline sculpture in the round (Section 5) (Zubieta, 2021: 1176–1177). Therefore, our efforts have focused on the creation processes, including the many factors on which their continuous transformation depends, inside the cave (e.g., rising water, humidity and temperature) (Zubieta, 2021). In that sense, the earthen figures must be observed as *things* in constant formation rather than having a static and fixed form (see Ingold, 2012: 436–438).

Lastly, and concomitantly with looking into the production materials



Fig. 3. A jaguar sculpture was molded in clay, using an existing stalagmite as a support. (Photo by Leslie F. Zubieta Calvert, 2018; courtesy of Secretaría de Cultura- INAH.-MEX. Reproduction of image authorized by Instituto Nacional de Antropología e Historia de México. Any request to reuse this image needs to be addressed to the aforementioned authority.) (c) 2018 Zubieta INAH research permit 401.1S.3–2018/654.

and techniques, it was imperative to provide the first chronological context of the plastic art and an initial comprehensive understanding of the occupation of this exceptional Mesoamerican site and its placement in Mesoamerican history. In order to achieve this goal, some fragments of charcoal were collected near specific figures (e.g., at the base, inside visible cracks, or less than 5 cm) in most Sections, as described in detail below.

2. Materials and methods

Because of the fragile nature of the clay reliefs, a careful selection of the sampling areas was discussed in advance based on photographs and orthophotos acquired during the recording phase of this project in 2018–2019. In the latter process, each earthen figure was given a consecutive number related to the section of provenience, thus facilitating the localization of the sampling areas. In order to maintain the integrity and aesthetics of the clay figures, naturally eroded sections of specific examples were chosen for extraction, especially those that, due to gravity, collapsed or detached from the central figure. In this sense, the study aimed to collect samples from each terrace to obtain a representative sample and an overall understanding of the production process of earthen figures. Nonetheless, it was essential to work with detached pieces and guard the integrity of the most preserved examples, and thus,

the study was not exhaustive. Our interdisciplinary and multiple-technique examination aimed to analyze the composition of the pastes used in the modelling process to (1) attest the presence of allochthonous materials in the mix, (2) facilitate the future conservation of clay figures—if necessary—by understanding the composition of those pastes, and (3) corroborate if clay figures were exposed to fire in some degree due to presence of charcoal fragments on the floor of the cave.

In addition, the Temperature (T) and humidity (%RH) were obtained using a Preciva digital psychrometer thermo-hygrometer after each sample extraction in April 2021 (Table 1). Although the measurements show a slight discrepancy, the humidity and temperature were constant inside the cave. However, Section 7 showed less humidity and higher temperature, perhaps reflecting specific factors such as air circulation, stream water flow, wall temperature and terrace dimensions. The lush vegetation around the cave entrance and the low altitude of 590 m above sea level allow humid conditions outside at certain times of the day, mainly due to its location in a mountain enclave. In the exterior of the cave, close to the entrance, we recorded a temperature of 23,17 °C, and 84.54 %RH. Humidity and temperature fluctuations throughout the year require further study to understand their impact on the conservation of the clay figures.

Four kinds of samples were collected to develop a global understanding of the production process of earthen reliefs and sculptures

(Fig. 20).

- Clay figures (E) (reliefs and sculptures): Twelve samples (Nos 2, 3, 8, 10, 11, 17, 19, 21, 23, 25, 29, see Table 1) from eroded areas and fragments to determine the composition of the pastes. One (sample No. 4, see Table 1) is from an amorphous mass of clay, possibly left as a pile of raw material ready for use.
- Platforms (P): Nine samples (Nos 5, 7, 9, 18, 20, 22, 24, 26, 30, see Table 1) from the terraces were collected to determine if they were prepared or levelled as “platforms” before modeling the clay figures on top.
- Holes (H): To compare the composition of pastes and dirt at the bottom of four holes observed in different sections of the cave, presumably used to extract raw material (Nos 1, 6, 27, 28, see Table 1).
- Subterranean stream profile (R): Five 25 cm-equidistant samples were obtained from the stream profile in Section 7 to determine and compare the composition of pastes and sediments (Nos 12–16, see Table 1).

2.1. Archaeometric analyses

- **Optical Microscopy (OM).** For comparison, all thin sections of the platform and sculpture samples were studied under polarized light microscopy using a “Zeiss Axioskop 40” petrographic microscope equipped with a “Canon PowerShot A640” photo camera. The semiquantitative estimate of the percentages of the skeleton was obtained by image analysis using the “ImageJ software 1.53e”. In particular, the skeleton composed of minerals with high brightness interference colors was considered. It is important to note that this method underestimates the percentage of the skeleton because it does not consider opaque minerals and minerals with dark interference colors. However, it is a valid method for making semiquantitative comparisons between samples.
- **Portable X fluorescence spectrometry (p-XRF):** A “Bruker Tracer IV-SD” spectrometer (Bruker, Billerica, MA, USA) equipped with a Rhodium tub, was used for detailed analysis on three representative samples, S6-5P, S6-4E and S5-5 N (Table 1), to determine the chemical composition of the different layers present inside them. Two analytical conditions were used to acquire the p-XRF spectra: for major elements a voltage of 40 kV and a current of 35 μ A were used, with an acquisition time of 100 s, under vacuum conditions < 17 Torr, without the “use of filters”; for trace elements a voltage of 40 kV and a current of 11 μ A were used, with an acquisition time of 100 s, in air, using a 12 mil Al/1 mil Ti (Yellow) filter. The spectra were interpreted using the “Bruker AXS MA Artax 7.4” software. The relative intensity of the peaks is indicated by the number of strokes per second (cps).
- **Energy Dispersion Microanalysis by Electron Probe Micro Analyzer (EPMA)** “JEOL- JXA 8230” equipped with an EDS Spectrometer was used for a detailed micro-chemical study (instrumental acquisition conditions: HV: 15 keV; Probe current: 10nA; Time: 30 sec.). This analysis was performed on the thin sections of samples S6-5P, S6-4E, S5-5 N, S11-1E and S14-1P (Table 1). Uncertainties are estimated based on typical instrumental precision reported in the literature, ranging from ± 2 % relative for major oxides (Goldstein et al., 2018).
- **UV-fluorescence microscopy (UVFM):** The thin sections of samples S6-5P and S6-4E (Table 1) were examined through fluorescence to reveal the distribution of the organic matter (Teichmüller and Wolf, 1977; Stach et al., 1982; Tyson, 1995). UV-fluorescence was induced by a Hg vapor lamp linked to a “Zeiss Axioplan II imaging” microscope equipped with high-performance wide bandpass filters (BP 436/10 nm/LP 470 nm for a green light; BP 450–490 nm/LP 520 nm for yellow light).

- **Micro-Raman Spectroscopy (μ -Raman)** was used for detailed mineralogical analyses. It was performed on the thin sections of samples S6-5P, S6-4E, S5-5 N, S11-1E and S14-1P (Table 1) by a “Thermo Fisher DXR” Raman Microscope and “OMNICxi Raman Imaging software 1.0”, and employing a 532.0-nm line, at an incident power output variable ranging between 3 and 7mW. The spectra obtained were compared with those available in the Department of Biology, Ecology and Earth Sciences (DiBEST) database of the University of Calabria and the RRUFF Project database (Lafuente et al., 2015).
- **X-ray fluorescence (XRF)** was performed on all pulverized samples (platform, clay figures, holes and stream) to determine the chemical composition of major (SiO_2 , TiO_2 , Al_2O_3 , Fe_2O_3 , MnO , MgO , CaO , Na_2O , K_2O , P_2O_5) and trace elements (Ni, Cr, V, La, Ce, Co, Nb, Ba, Y, Sr, Zr, Zn, Rb, Pb). The analysis was carried out on pressed powder pellets realized using 6 g of specimen pressed on a boric acid substrate at a maximum working pressure of 25 bar. The analysis was performed by a wavelength-dispersive X-ray fluorescence spectrometer, “Rigaku Primus II”, equipped with a 4 kW Rh X-ray source. Specific slits were used per each detector, scintillation and flow counter, mounted on a goniometer with an angular range from 5° to 148° . All elements were measured at 50 kV and 60 mA. Chemical data of both major and trace elements were processed through Principal Component Analysis (PCA), using the software STATISTICA 7. The PCA plots were generated using the first two principal components, as they alone account for more than 60 % of the total variability among the samples.
- **X-ray powder diffraction (XRPD)** was performed on all pulverized samples (platform, clay figures, holes and stream, Table 1) to determine their mineralogical composition through a “Bruker D8 Advance” X-ray powder diffractometer, with Cu-K α radiation, operating at 40 kV and 20 mA. The scans were collected with a step scan of $0.02^\circ 2\theta$ and 2 s/step, in the range $3\text{--}60^\circ 2\theta$. The width of the divergence, anti-scattering and receiving slits were 0.6, 0.2, and 0.1 mm, respectively. The “EVA software program (DIFFRACplus EVA)” was used to identify the mineralogical phases in each X-ray powder spectrum by comparing experimental peaks with PDF2 reference patterns (Kabekkodu et al., 2024). In addition, XRPD analysis was repeated on seven representative samples coming from platforms (S6-5P and S10-4P), clay figures (S5-5E and S11-1E), stream (S7-1) and holes (S2-2 and S14-1), after subjecting the samples to a specific treatment to discriminate the different topologies of clay minerals. In particular, the analysis was repeated (with a scan speed of $0.2^\circ 2\theta$ in the range of $3\text{--}30^\circ 2\theta$) after saturating the samples with ethylene glycol at 60°C for 24 h.
- **Differential scanning calorimetry (DSC) and thermogravimetry (TG)** were performed on the same selected samples (S6-5P, S10-4P, S5-5E, S11-1E, S7-1, S2-2 and S14-1), to delve into their mineralogical composition. The analysis for each sample was carried out in an alumina crucible under a constant nitrogen flow of 30 mL/min through a Netzsch STA 449C Jupiter (Netzsch-Gerätebau GmbH, Selb, Germany) in a $25\text{--}1000^\circ\text{C}$ temperature range and a heating rate of $10^\circ\text{C}/\text{min}$. Derivative thermogravimetry (DTG), derivative differential scanning calorimetry (DDSC) and endothermic peaks were obtained using Netzsch Proteus thermal analysis software, version 4.7.0.
- **Radiocarbon Dating.** Many fragments of charcoal mixed with clay are visible on the cave floor in almost every section (e.g., Sections 5, 6, 9, 15), but not all were suitable for dating due to their small size and preservation. Charcoal fragments > 1 cm were collected at the base of the clay figures subjected to the above archaeometric study (S11-1_C, S15-2_C). When this was not possible, suitable charcoal was extracted less than 5 cm from clay Fig. S5-5_C and S14-1_C and 30 cm from the leg of a female figure (S9-8_C). On one occasion (S12-5_C), a piece of charcoal was removed within a visible crack in the leg of the clay relief. Some charcoal fragments near clay figures not

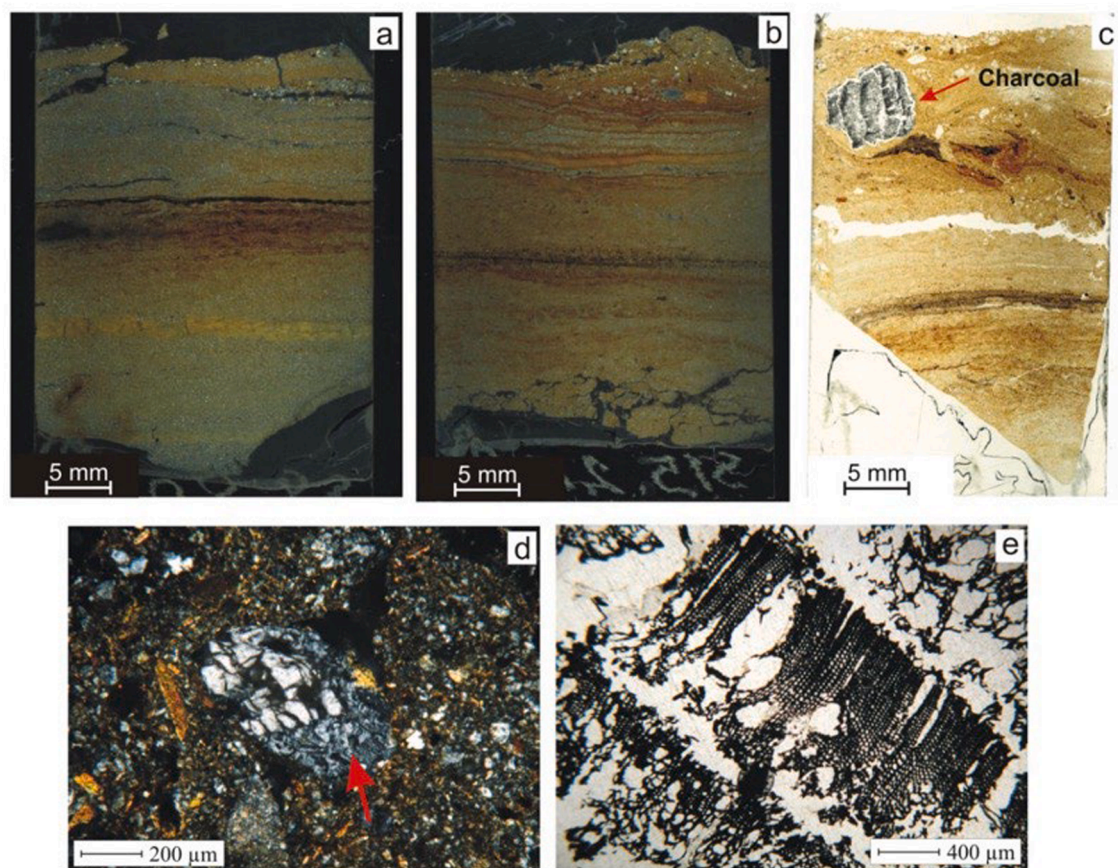


Fig. 4. Flatbed scanner images of sample S9-8P (a) and S15-2P (b) under crossed nicols. c) Flatbed scanner images of sample S6-5P under parallel nicols. d) Microphotograph with particular of polycrystalline quartz in sample S6-5P, under crossed nicols. e) Microphotograph with particular charcoal fragment in sample S6-5P, under parallel nicols.

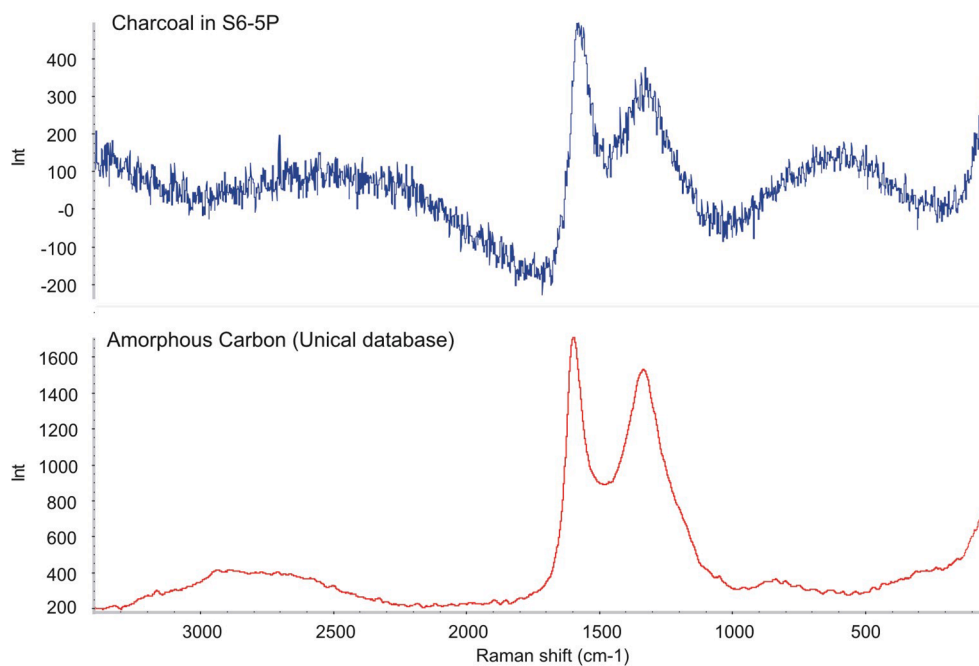


Fig. 5. Raman spectra of the charcoal fragments in sample S6-5P and the amorphous carbon in the Unical database.

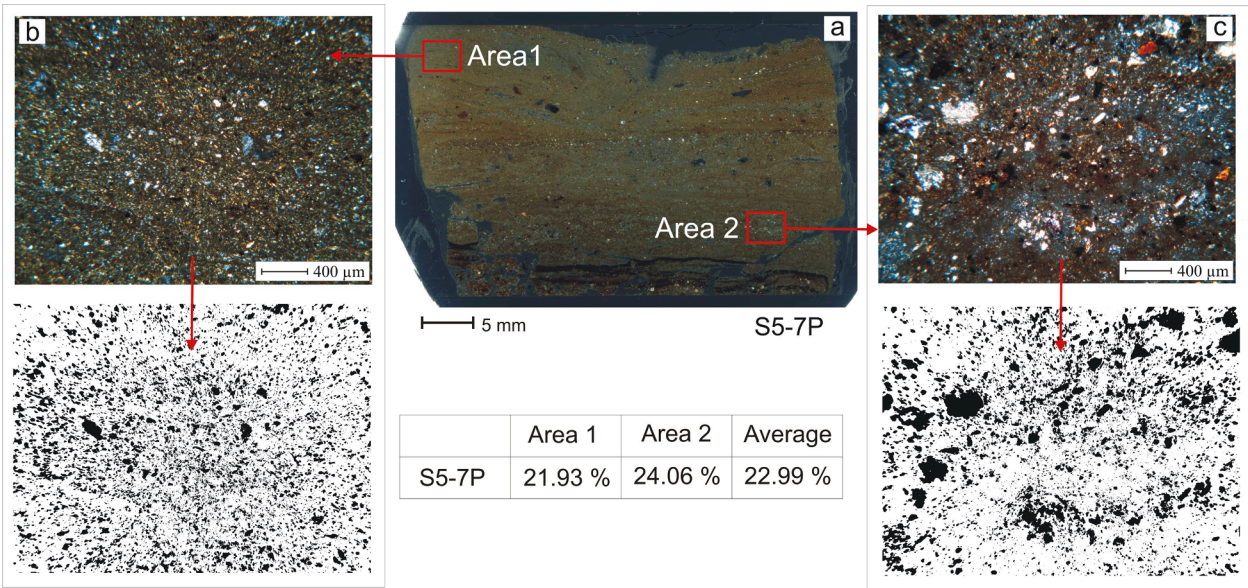


Fig. 6. Example of image analysis performed by ImageJ software. a) Flatbed scanner images of sample S5-7P under crossed nicols with the identification of the two analyzed areas. b) Microphotograph of Area 1 under crossed nicols (above) and subjected to threshold (down). c) Microphotograph of Area 2 under crossed nicols (above) and subjected to threshold (down). Skeleton percentages were calculated using ImageJ software.

Table 2

Minero-petrographic features of the samples taken from the Platform [Bt: biotite, Cal: calcite, Kfs: k-feldspar, Pl: plagioclase, Ms: muscovite, Om: opaque minerals; Qtz: quartz; tr: traces] with average percentage of the skeleton calculated by ImageJ software. The mineralogical phases were determined by combining XRPD data with OM data.

Sample	Mineralogical phases	Other	% of skeleton
S5-7P	Qtz, Pl, Kfs, Bt, Ms, Om	Charcoal fragments, polycrystalline quartz (tr)	22.99
S6-5P	Qtz, Pl, Kfs, Bt, Ms, Om, Cal (tr)	Charcoal, polycrystalline quartz (tr)	21.64
S7-2P	Qtz, Pl, Kfs, Bt, Ms, Om	Charcoal, polycrystalline quartz (tr)	28.65
S9-8P	Qtz, Pl, Kfs, Bt, Ms, Om	Charcoal (tr), polycrystalline quartz (tr)	22.02
S10-4P	Qtz, Pl, Kfs, Bt, Ms, Om	Charcoal, argillite (tr)	29.21
S11-1P	Qtz, Pl, Kfs, Bt, Ms, Om	Argillite (tr)	20.08
S12-5P	Qtz, Pl, Kfs, Bt, Ms, Om, Cal	Charcoal, polycrystalline quartz (tr)	22.17
S14-1P	Qtz, Pl, Kfs, Bt, Ms, Om, Cal (tr)	Charcoal	20.32
S15-2P	Qtz, Pl, Kfs, Bt, Ms, Om, Cal	Charcoal, polycrystalline quartz (tr)	20.64
Average % skeleton			23.08

subjected to the archaeometric study were also selected because of their large size, placement and preservation (i.e., S10-2_C; Fig. 22; Table 6). The samples were treated with acid-base-acid (1 N HCl and 1 N NaOH, 75 °C) before combustion at the W.M. Keck Carbon Cycle Accelerator Mass Spectrometer (Keck-CCAMS), University of California, Irvine. Likewise, sample preparation backgrounds were subtracted based on the measurements of ¹⁴C-free wood. All results were corrected for isotopic fractionation according to the conventions of Stuiver and Polach (1977: 355), with δ13C values measured in graphite prepared using the AMS (accelerator mass spectrometry) spectrometer. Radiocarbon dating results were calibrated using the online program OxCal. v4.4 Ramsey (2021) and the IntCal13 atmospheric curve (Reimer et al., 2020; Table 6)

Table 3

Minero-petrographic features of the samples taken from the Clay figures [Bt: biotite, Kfs: k-feldspar; Pl: plagioclase, Ms: muscovite, Om: opaque minerals; Qtz: quartz; tr: traces] with average percentage of the skeleton calculated by ImageJ software. The mineralogical phases were determined by combining XRPD data with OM data.

Sample	Mineralogical phases	Other	% of skeleton
S5-5 N	Qtz, Pl, Kfs, Bt, Ms, Om	Charcoal (tr)	19.29
S5-5E	Qtz, Pl, Kfs, Bt, Ms, Om	Charcoal, argillite (tr)	20.28
S5-7E	Qtz, Pl, Kfs, Bt, Ms, Om	Polycrystalline quartz (tr)	19.16
S6-4E	Qtz, Pl, Kfs, Bt, Ms, Om	Charcoal (tr)	18.79
S7-3E	Qtz, Pl, Kfs, Bt, Ms, Om		19.13
S7-5E	Qtz, Pl, Kfs, Bt, Ms, Om	Charcoal, polycrystalline quartz (tr)	20.48
S9-8E	Qtz, Pl, Kfs, Bt, Ms, Om	Charcoal, polycrystalline quartz (tr), argillite (tr)	20.90
S10-4E	Qtz, Pl, Kfs, Bt, Ms, Om	Charcoal	23.99
S11-1E	Qtz, Pl, Kfs, Bt, Ms, Om		18.74
S12-5E	Qtz, Pl, Kfs, Bt, Ms, Om	Charcoal	18.38
S14-1E	Qtz, Pl, Kfs, Bt, Ms, Om	Polycrystalline quartz (tr)	19.84
S15-2E	Qtz, Pl, Kfs, Bt, Ms, Om	Polycrystalline quartz (tr)	21.71
Average % skeleton			20.06

3. Results

3.1. Petrographic features, analogies and differences among samples

The platform samples show an inhomogeneous aspect due to the high stratification of different bands with variable color, from black to red and yellow (Fig. 4. a, b, c), which correspond to the natural structure of the clayey endokarstic deposits. The skeleton (=non-clayey fraction) is unevenly distributed; thus, it is more concentrated in some areas and

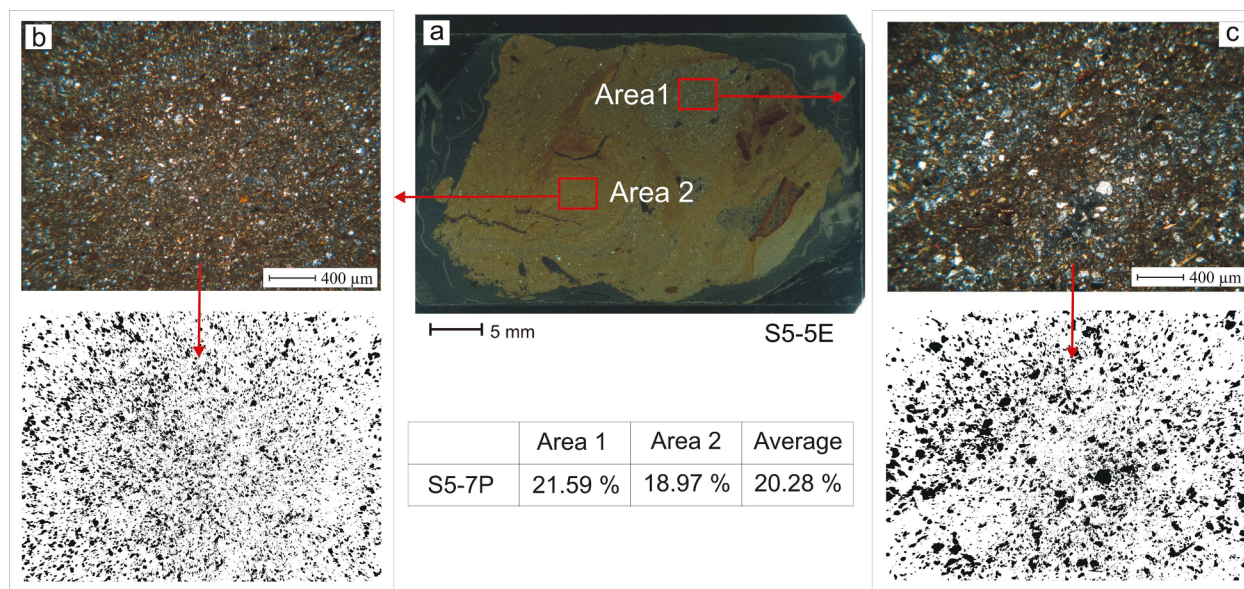


Fig. 7. Example of Image analysis performed by ImageJ software. A) Flatbed scanner images of sample S5-5E under crossed nicols with the identification of the two analyzed areas. B) Microphotograph of Area 2 under crossed nicols (above) and subject to the threshold (under). C) Microphotograph of Area 1 under crossed nicols (above) and subject to threshold (under). Table with the skeleton percentage calculated by the software.

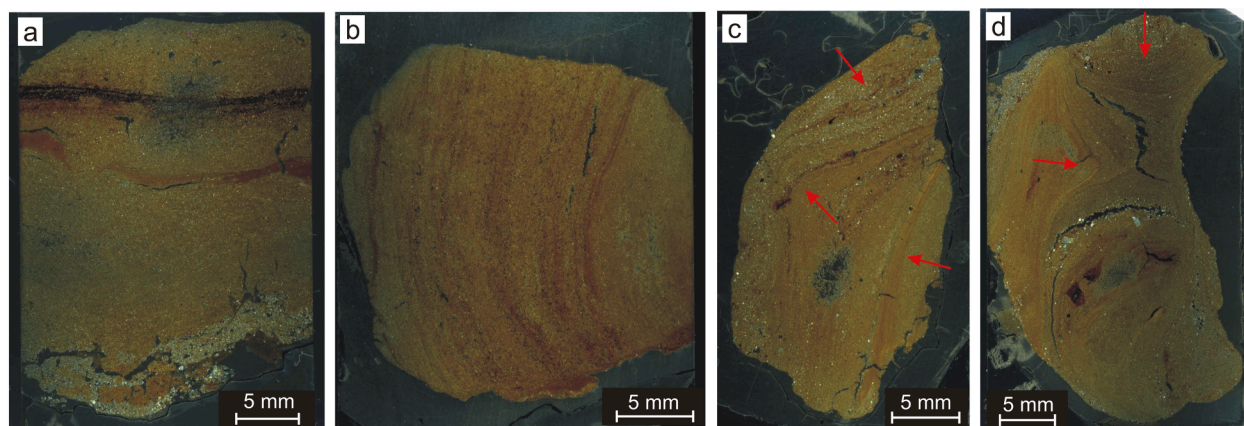


Fig. 8. Flatbed scanner images under crossed nicols. a) Sample S6-4E, belonging to the first group. b) Sample S7-3E belongs to the first group. c) Sample S7-5E belongs to the second group. d) Sample S9-8E (Fig. 21) belongs to the third group. The red arrows indicate the pressure direction.

less in others (Fig. 4. a, b), showing different grain sizes depending on the location (Fig. 6. b, c). However, the average grain size is generally very fine sand, $63 \mu\text{m} - 125 \mu\text{m}$ (Wentworth, 1922: 388, Fig. 3). In samples S6-5P, S12-5P, S14-1P, and S15-2P, the mineralogical phases identified were quartz, feldspar, mica (muscovite, biotite), plagioclase, opaque minerals and traces of calcite. Some traces of polycrystalline quartz and argillite fragments, whose size is mostly very fine, were visible in some samples (Fig. 4. d). All samples, except S11-1P, contain charcoal fragments (Table 2); in particular, a fragment of 8.00 mm is evident in sample S6-5P (Fig. 4. c, e). The presence of charcoal was confirmed by Micro-Raman Spectroscopy (Fig. 5). ImageJ software allowed us to estimate the percentage of the skeleton in two representative areas for each sample and calculate the average between those percentages (Fig. 6). Data in Table 2 indicate that the skeleton percentage is variable from 21.64 % to 29.21 %.

The clay figure samples have many analogies with those from the platform: the skeleton (=non-clayey fraction) is unevenly distributed with different grain sizes, usually very fine sand (Wentworth, 1922). The mineralogical phases identified are quartz, feldspar, mica (muscovite, biotite) and opaque minerals (Table 3). Fragments of charcoal, traces of

polycrystalline quartz, and argillite fragments were visible in some samples (Table 3). ImageJ software (Fig. 7) highlighted a skeleton percentage variable between 18.38 % and 23.99 % (Table 3). It is possible to note very similar values: 23.08 % (for the platform) and 20.06 % (for the clay figures); thus, we can suggest that the same clay was used to manufacture the latter.

3.2. Modeling the clay: Working pressure

The clay figure samples (E) have the same visible stratification as the platform samples (P), which have different bands of variable color: black, red, and yellow. Still, all samples of the earthen figures have indications of clay modeling, but these signs do not override the natural stratigraphic structure of the clay deposits *in situ*. Even though the natural stratification is well preserved, it has been modified in different ways and various directions depending on the degree of modeling of the clay.

By comparing different samples from the clay figures, we propose three degrees of modelling depending on the bending of the layers:

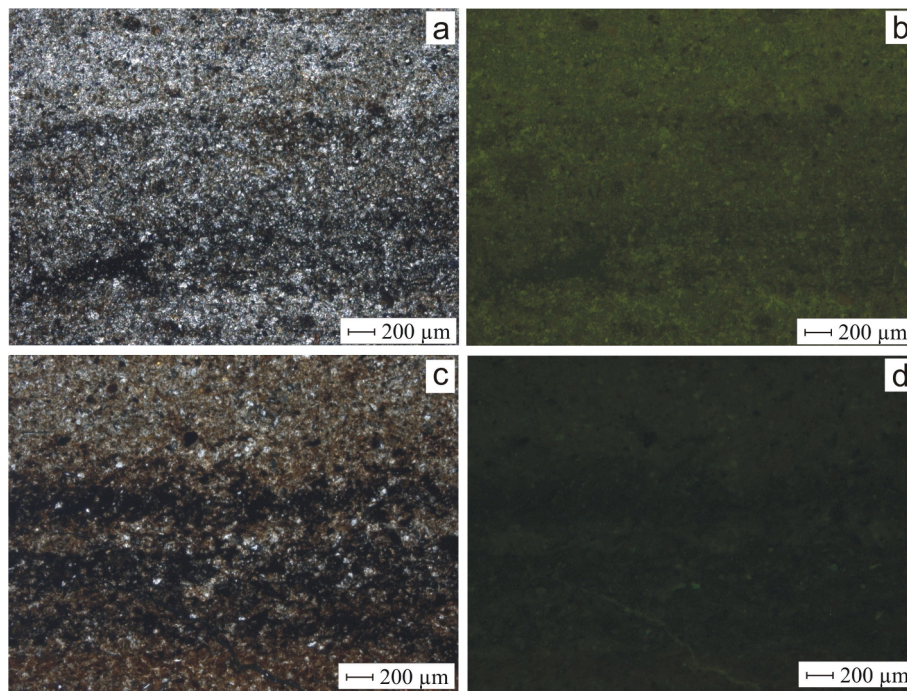


Fig. 9. Microphotograph of the black layer in sample S6-5P under crossed nicols (a) and same area under UV-excitation (b); microphotograph of the black layer in sample S6-4E under crossed nicols; (c) same image under UV-excitation; (d) analyses under UV-excitation revealed the lack of epifluorescence in both the areas analyzed due to the absence of organic matter.

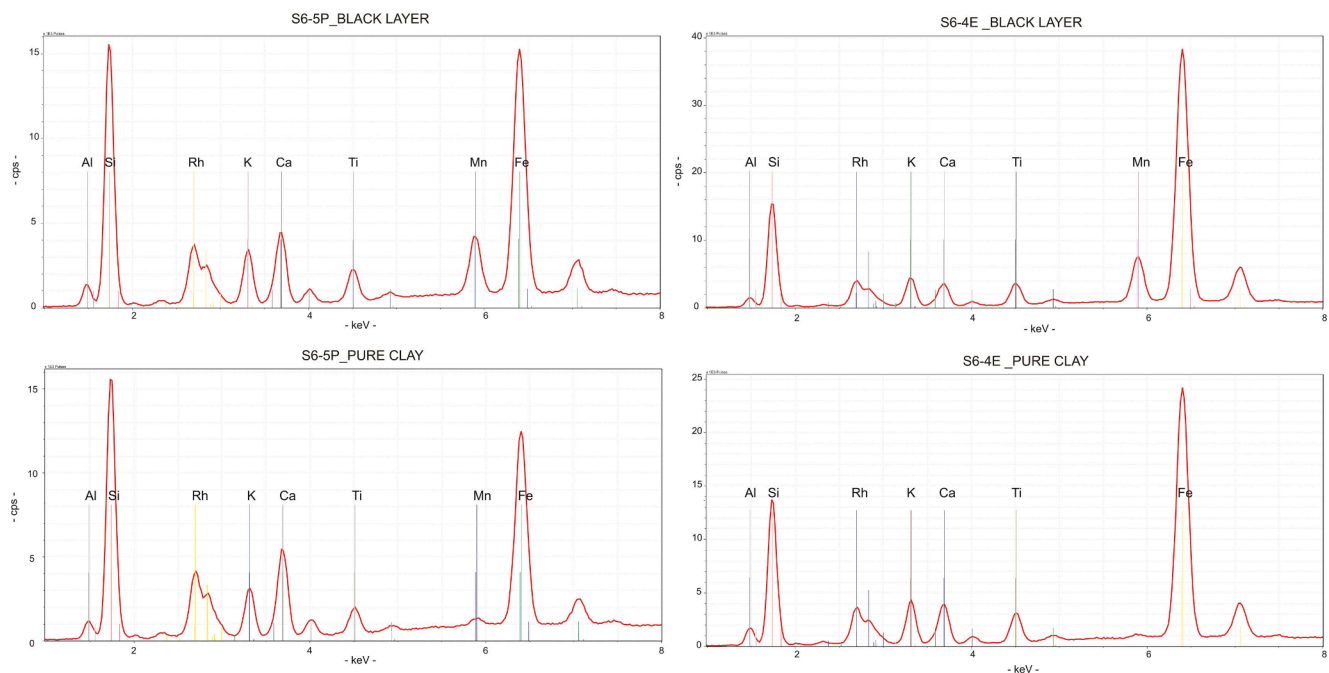


Fig. 10. p-XRF spectra of the black layer and the pure clay present in samples S6-5P (on the left) and S6-4E (on the right). Please note that (the rhodium signal (Rh) is due to the X-ray tube).

Low modeling (layers are vaguely folded): Samples S6-4E (Fig. 8a), S7-3E (Fig. 8b), and S5-7E show a parallel but slightly curved stratification to indicate a minor pressure in the modeling phase of the clay. It is worth noting that Sample S6-4E was taken from the side of a female figure modeled on a pile of mud at the end of the terrace in Section 6, so it corresponds to the support of that figure. Sample S5-7E, on the other side, corresponds to an amorphous clay mass.

Medium modeling (moderately folded layers): Samples S15-2_E, S7-5_E (Fig. 8c), S12-5_E and S11-1_E show a much more modified stratification.

High modeling (the parallelism between the layers disappears, and numerous bending cores are observed): Samples S5-5E, S9-8E (Fig. 8d), S10 4_E and S14-1_E that have a plastically bent stratification to form more or less acute angles depending on the greater or lesser pressure exerted to give to the sculpture the desired shape.

Table 4

EDS microanalysis of the pure clay and colored layers in the samples, expressed in %wt (udl: under detection limit). Uncertainties are estimated based on typical instrumental precision reported in the literature, ranging from ± 2 % relative for major oxides (Goldstein et al., 2018).

%wt	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅
S6-4E_pure clay	70.84	0.77	15.71	4.77	0.21	1.32	1.68	1.21	2.43	1.05
S6-4E_black layer	65.52	0.66	15.37	5.38	5.01	1.23	1.88	1.73	2.50	0.72
S6-4E_red layer	62.19	0.98	22.50	6.88	0.06	1.47	1.27	0.77	2.77	1.10
S6-5P_pure clay	69.93	0.59	15.72	5.28	0.04	1.38	1.95	1.54	2.58	1.00
S6-5P_black layer	69.49	0.55	13.89	4.62	3.89	1.08	1.60	1.41	2.44	1.02
S6-5P_red layer	68.25	1.11	15.29	7.63	0.21	1.37	1.35	1.03	2.49	1.27

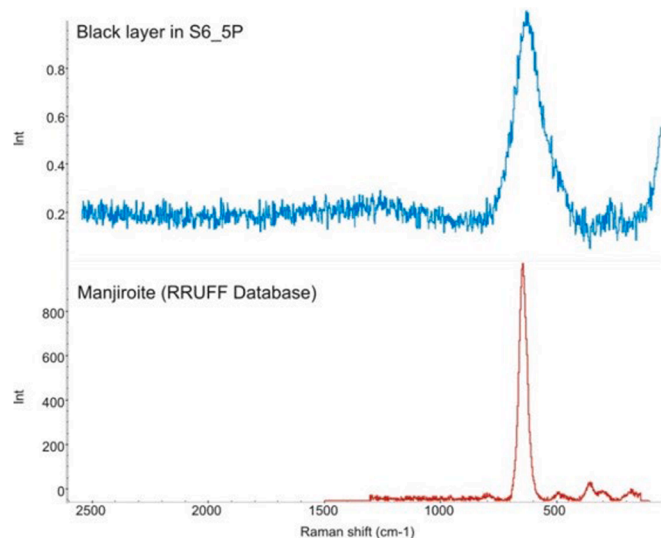


Fig. 11. Raman spectrum of the black layer in sample S6-5P compared with the spectrum of “Manjiroite” of the RRUFF database.

3.3. Composition of black and red layers within the samples

UV-fluorescence microscopy was used on two representative samples, S6-5P and S6-4E, to study the nature of the black layers visible in the samples. The lack of epifluorescence revealed no organic matter in the analyzed areas (Fig. 9). The samples were subjected to portable X fluorescence spectrometry (p-XRF) to compare the pure clay and black layer composition. The only difference was the higher amount of Manganese (Mn) in the black layers. Elements such as Aluminum (Al), Silica (Si), Potassium (K), Calcium (Ca), Titanium (Ti) and Iron (Fe) are present in the clay composition and visible in both pure clay and black layers (Fig. 10).

To obtain a quantitative estimate of the manganese content in the black layers and determine the clay's chemical composition, EDS analysis by Electron Probe Micro Analyzer (EPMA) was performed on the same two samples (S6-5P and S6-4E). In addition, the layers with a red color were studied to verify the differences in composition between the black and pure clay. On each layer, two areas of 500x500 μm^2 were analyzed, and average values were obtained (Table 4). The study revealed that manganese oxide (MnO) content in pure clay and red layers is very low. In particular, in the pure clay, it is of 0.21 %wt and 0.04 %wt, while in the red layers, it is 0.06 %wt and 0.21 %wt. On the contrary, in the black layer of samples S6-4E and S6-5P, the MnO content is 5.01 %wt and 3.89 %wt, respectively. As shown by p-XRF analyses and confirmed by EDS, iron (Fe) is present in the pure clay and the black layers. However, in the red layers, the iron oxide (Fe₂O₃) is much higher than the others (6.88 %wt in S6-4E and 7.63 %wt in S6-5P); thus, the red coloration of the layers is linked to a higher content of iron oxide.

Micro-Raman Spectroscopy was used to study the mineralogical composition of the black and red layers in the samples. The black layers of samples S6-5P and S6-4E highlighted the presence of “manjiroite”

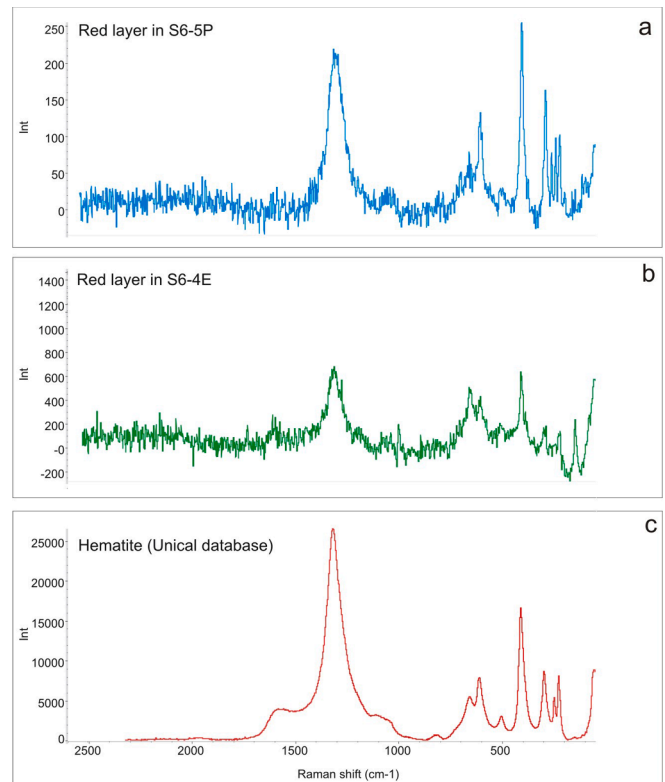


Fig. 12. Raman spectra of the red layers present in samples S6-5P (a) and S6-4E (b) and comparison with the Hematite spectrum (c) of the Unical database.

(Fig. 11), a hydroxide containing manganese, potassium and sodium [(Na, K)(Mn⁺⁺⁺⁺, Mn⁺⁺)₈O₁₆*n(H₂O)]; while in the red layers showed the presence of hematite (an iron oxide [Fe₂O₃]) (Fig. 12).

3.4. Black patina on the exterior surface of samples

Some rare examples of human clay figures show red and black colors decorating the face (e.g., a female figure in Section 9; Fig. 21), and some feline clay figures in Section 5 show a black patina covering their heads, thus raising the question of whether those colors were applied at the end of the production process or if they were part of the mixture. Due to conservation limitations, only three samples of the black patina were collected and studied: S5-5 N, S11-1E, and S14-1P (Table 1).

The study of the superficial black patina in sample S5-5 N was performed using p-XRF (Fig. 13) and revealed a high content of manganese (Mn). The EDS analysis carried out by Electron Probe (Fig. 14) detected a content of manganese oxide (MnO) of 5.81 %wt in the black patina, while in the pure clay, its content is under the detection limit (Table 5). As mentioned above, Micro-Raman Spectroscopy revealed the presence of “manjiroite” in the black layers of samples S6-5P and S6-4E (Fig. 15). Consequently, it is possible to affirm that the black patina visible on the surface of sample S5-5 N contains manganese.

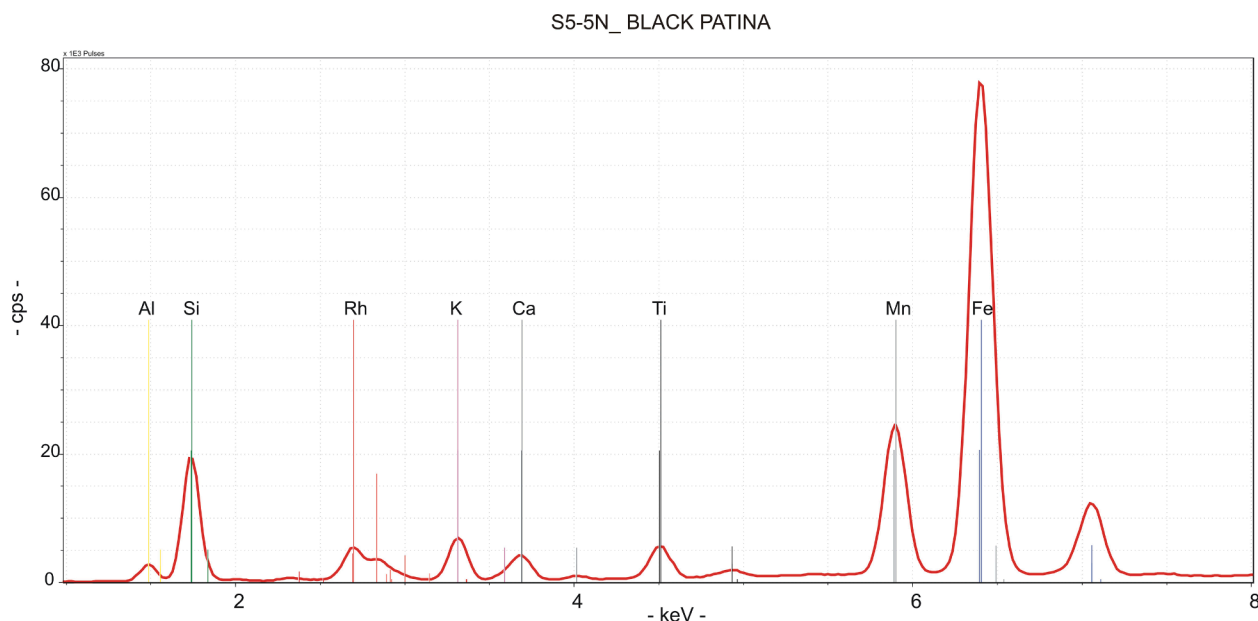


Fig. 13. P-xrf spectra of the superficial black patina present in sample s5-5 N.

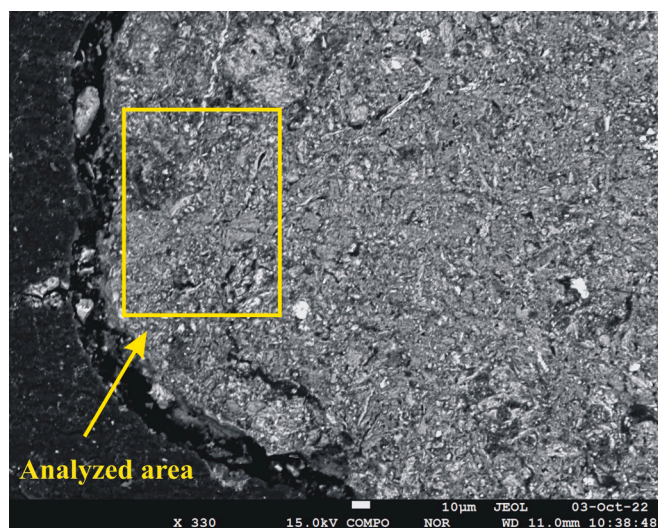


Fig. 14. Image obtained by Electron Probe with the area analyzed in sample S5-5 N (corresponding to the black patina).

Although thinner and more fragmented, a similar black patina is visible on the exterior surface of samples S11-1E and S14-1P. Through EDS microanalysis of S11-1E, we identified a manganese oxide content of 7.50 %wt and 5.10 %wt for S14-1P inside the black patina, while manganese content is under the detection limit in the pure clay (Table 5). Such values fit with the data obtained for sample S5-5 N. In

parallel, Micro-Raman Spectroscopy confirmed the presence of “manjiroite” in these samples (Fig. 16).

By observing the superficial black patina of samples S5-5_N, S11-1_E y S14-1_P through optical microscopy, it is possible to note a very thin, homogeneous and compacted stratum (Fig. 17a) which differs from the less homogeneous and compacted natural layers of the samples (Fig. 17b). The morphology of the black superficial patina can be explained as the artificial application of the manganese layer after the completion of the clay figure. This conclusion is supported by the fact that the black patina in the external part of the sample does not have polycrystalline quartz fragments (Fig. 17a) that are present in the natural black layers of the samples (Fig. 17b). Moreover, the black surface patina has a constant thickness. As discussed below, the manganese used to apply the black finish was likely procured from the natural deposits observed *in situ*, given the presence of “manjiroite.”

3.5. Mineralogical and chemical analysis

XRPD was performed on all samples to study the mineralogical composition. In addition, on some selected samples (S6-5P, S10-4P, S5-5E, S11-1E, S7-1, S2-2 and S14-1), XRPD analysis was performed after saturating the samples with ethylene glycol and thermogravimetric analysis (DSC-TG), to clarify the nature of some minerals present inside the samples. The main minerals found are quartz, plagioclase and k-feldspar. The thermogravimetric analysis and the treatment with glycol allowed the identification of phlogopite (instead of biotite and muscovite, observed by optical microscopy), the presence of kaolinite and vermiculite as clay minerals and the presence of todorokite-ranciéite which are manganese oxides; the latter a product of weathering of manganese deposits in limestone caves (see Anthony et al., n.d.):

Table 5

EDS microanalysis of the pure clay and the black patina in the samples, expressed in %wt (udl: under detection limit). Uncertainties are estimated based on typical instrumental precision reported in the literature, ranging from ± 2 % relative for major oxides (Goldstein et al., 2018).

%wt	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅
S5-5N_pure clay	61.24	0.66	24.67	4.46	udl	1.47	2.09	2.83	2.22	0.37
S5-5N_black patina	58.79	0.55	20.12	6.05	5.81	1.48	1.37	2.19	3.02	0.63
S11-1E_pure clay	62.00	0.80	21.00	7.80	udl	2.00	2.10	1.10	3.20	udl
S11-1E_black patina	60.60	0.20	18.20	5.80	7.50	1.40	2.40	1.00	2.70	0.10
S14-1P_pure clay	71.50	0.80	17.20	4.30	udl	1.70	1.80	udl	2.60	udl
S14-1P_black patina	68.20	0.10	14.60	3.80	5.10	1.60	1.50	2.30	2.50	0.30

Table 6

List of collected charcoal concerning clay reliefs subjected to archaeometry analysis. Please note sample *254903 for a comparative chronological framework of plastic art production (see Fig. 2).

No.	UCIAMS	Sample	¹⁴ C age (BP)	±	Cal BCE/CE	Charcoal fragment location
31	254,897	S5-5_C	1265	15	675 777	Under the feline's head, on the floor.
32	254,901	S9-8_C	1235	15	702 878	30 cm from the right ankle of a female figure.
33	*254903	S10-2_C	2060	15	−151 8	Directly underneath the “monkey-like” sculpture.
34	254,904	S11-1_C	1275	15	675 774	Collected at the base of a modeled cube.
35	254,905	S12-5_C	1395	15	607 663	Inside a crack of the leg of a human figure.
36	254,906	S14-1_C	1245	15	682 872	On the floor between the legs of a female figure, next to the right heel.
37	254,907	S15-2_C	1205	15	782 881	Underneath the distal section (foot) of the left leg.

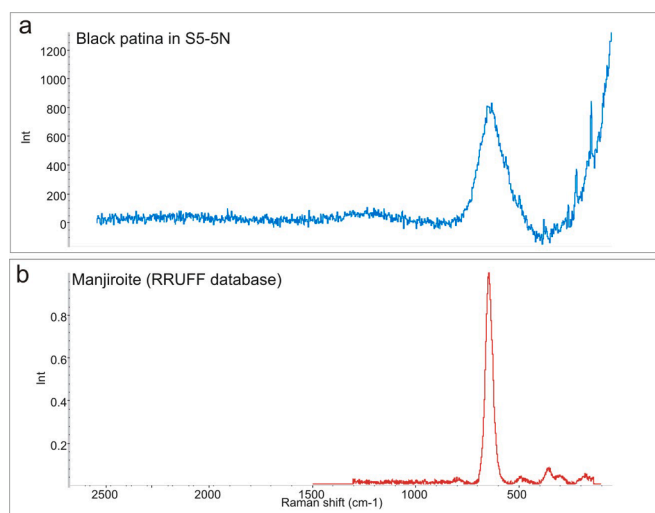


Fig. 15. Raman spectrum of the black patina in sample S5-5 N (a) and comparison with the spectrum of “Manjiroite” present in the RRUFF database.

anciéite). In addition, traces of calcite, also visible by optical microscopy, were identified in sample S12_5P.

As observed by the Derivative thermogravimetry (DTG) shown in Fig. 18, the composition of the samples is the same, with each peak corresponding to the presence of different minerals. In particular: 1) range 110 ÷ 160 °C: vermiculite; 2) range 220 ÷ 320 °C: phlogopite; 3) range 420 ÷ 540 °C: kaolinite; and 4) range 680 ÷ 840 °C: todorokite-ranciéite.

XRF was used to determine the chemical composition of major and trace elements of all pulverized samples (see Appendix A: supplementary data). In order to highlight similarities and differences among the samples, the chemical data were subjected to Statistical Multivariate Analysis, particularly to a Principal Component Analysis (PCA), by considering major (Fig. 19a) and trace elements (Fig. 19b). Through this

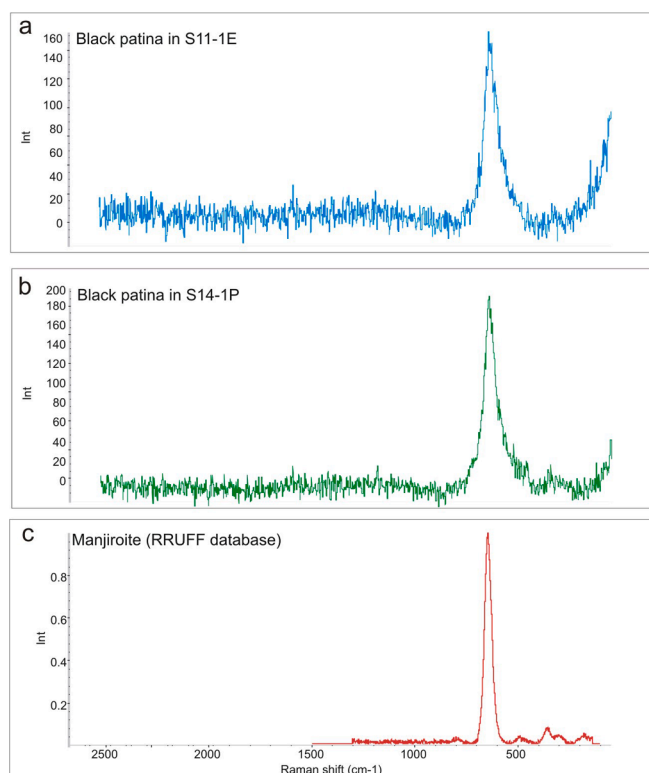


Fig. 16. Raman spectra of the black patina in sample S11-1E (a), sample S14-1P (b), and the spectrum of “Manjiroite” in the RRUFF database.

comparison, we can note that the compositional fields of the platform, clay figures, holes and stream partially overlap, thus indicating that the chemical composition of the samples is very similar. The only sample that slightly deviates from both diagrams is S7-5, which corresponds to the last level of the stream (underwater); this difference is due to the fact that, among the samples collected from the stream, this particular sample has the highest SiO₂ content. The trace elements, present in smaller amounts, are more discriminating. The chemical composition of the clay figures is very similar to those of the platform and S7-1 (sampled in the superficial part of the platform). In contrast, the other samples (holes and the stream) show a slightly different chemical composition.

3.6. Dating the clay figures and their context

Radiocarbon dates provide a noteworthy chronological framework bracket between 607–881 CE (with a 95.4 % probability; Table 6). While acknowledging here the complexity of cultural periodization as a fluid construction, this temporal range falls into what is generally known as the Late Classic Period in Mesoamerica by some researchers and is used here in a chronological sense (Coe, 1962: 15; López Austin and López Luján, 2000: 22, Cuadro 5; Sanders and Price, 1968: 16, 30; Willey, 1962: 86, Fig. 2). While samples S5-5_C and S14-1_C were collected less than 5 cm from their respective clay figures, S9-8_C was collected 30 cm from the leg of a female figure (Table 6); thus, these dates may be related to two connected activities: the age of production or use of that space. However, we have no certainty of the temporal distance between those events. On the other hand, S12-5_C was obtained from a crack in the clay figure, and S11-1_C and S15-2_C underneath the base of those figures, so it is more likely that these latter dates are linked directly to the production process of the figures, as discussed below. Although most collected samples fall into the Late Classic Period, S10-2_C, extracted from a chunk of charcoal placed underneath the foot of the “monkey-like” sculpture (not subjected to the archaeometric analyses discussed before; Fig. 22), dates from 151 BCE to 8 CE. This date coincides with

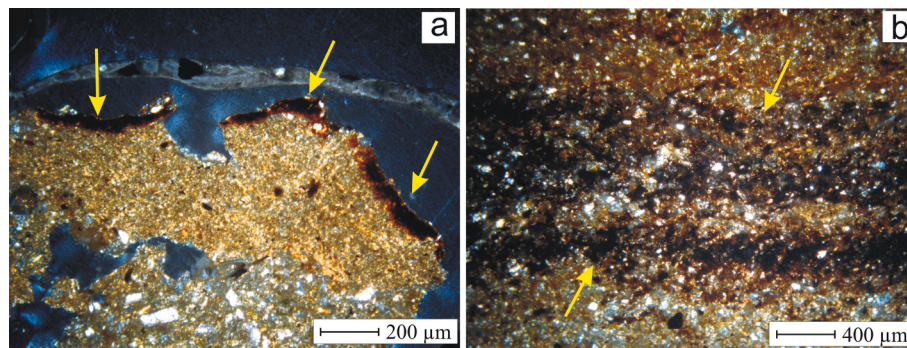


Fig. 17. Microphotograph on a thin section by optical microscopy under crossed nicols. a) Black patina of sample S5-5 N. b) Black layer of sample S6-4E.

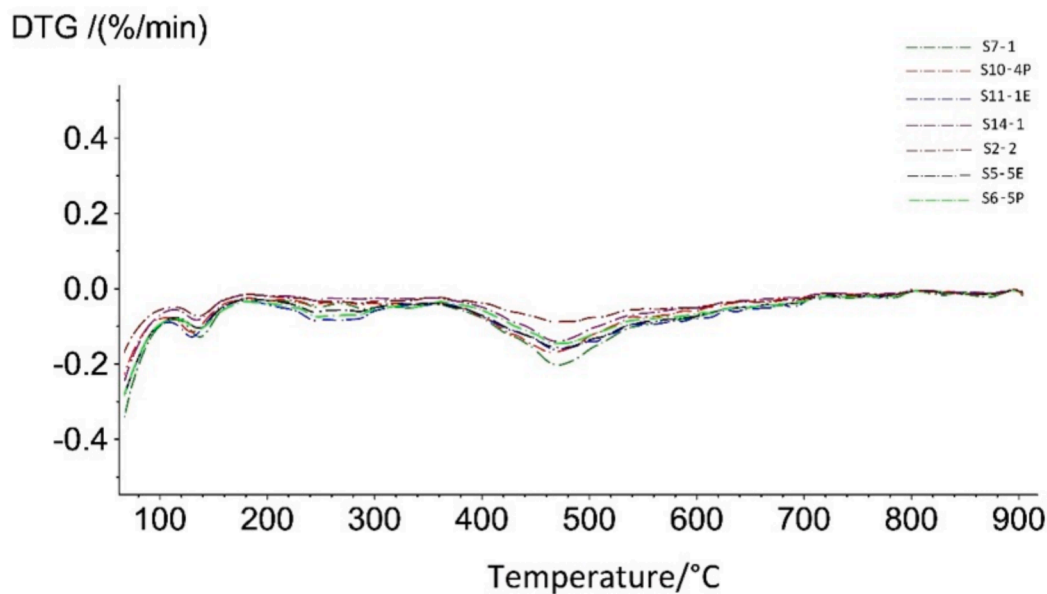


Fig. 18. Comparison between the derivative thermogravimetry of the samples highlighted the difference in temperature range linked to the minerals.

that calibrated 197 to 47 BCE (Beta-527199. Radiocarbon age 2100 ± 30 BP = 150 ± 30 BCE) published by Marcus Winter (2020: 172) from an exposed area of the sediment of the ball court in Section 7.

Some remnants of the dated charcoal were suitable for anthracological analysis following Lawrence Leney and Richard Casteel's procedure (1975) at the Laboratorio de Paleoetnobotánica y Paleoambiente, Instituto de Investigaciones Antropológicas, Universidad Nacional Autónoma de México in Mexico City (Adriano Morán, 2022). However, not all the charcoal pieces associated with the sampled clay reliefs could be adequately identified. Due to the high degree of carbonization, only some tracheids were observed in S14-1-C and S15-2-C, thus leaving the species determination at the Gymnospermae group level. Nonetheless, the charcoal fragment recovered and dated underneath the “monkey-like” sculpture (S10-2_C) was 3–7 mm long and identified as *Pinus* sp. (Adriano Morán, 2022), hence providing us with a general botanical identification of the dated charcoal and a preliminary understanding of the types of wood used in the cave.

4. Discussion

The intact stratification of the platform samples (P) indicates that the terraces are natural deposits and not the result of human activity. In this sense, (P) and (E) samples share many similarities: they have the same stratification with different color band variables, the same skeleton typology and a very similar skeleton percentage. The red layers visible

inside (P) and (E) samples show higher iron oxide (hematite; Fig. 12) content than layers of pure clay. As confirmed by UV-fluorescence microscopy, the black layers visible inside the platform and the clay figure samples are not due to organic matter or the result of fire degradation since no burning or fired clay can be seen in the samples. Moreover, the multi-technique analysis on those black layers revealed the presence of manganese oxide and hydroxide, such as “manjiroite” (manganese hydroxide), identified by Micro-Raman Spectroscopy. Consequently, these are natural layers with a higher concentration of manganese.

On the other hand, the external black patina of samples S5-5 N, S11-1E and S14-1P shows the same composition of the black layer visible inside the samples, with manganese oxide and hydroxide (as manjiroite). Nonetheless, the patina is more homogeneous, compact, and without quartz micro-fragments than the natural black layers inside the samples. Thus, it is likely that manganese was procured from specific natural endokarstic clay outcrops *in situ*, where artisans found it in high concentrations, ground it into powder, and applied it as black pigment.

To define the provenience of clay deposits used in the production of the reliefs and sculptures, XRPD revealed that the mineralogical composition of the samples from the platform, clay figures, holes and those from the stream's profile is very homogeneous, consisting of quartz, plagioclase, potassium feldspar, mica (phlogopite and muscovite), kaolinite, vermiculite and todorokite-ranciéite in all samples. The statistical processing (PCA) of the chemical data obtained by XRF revealed that the composition fields of the samples from platforms (P),

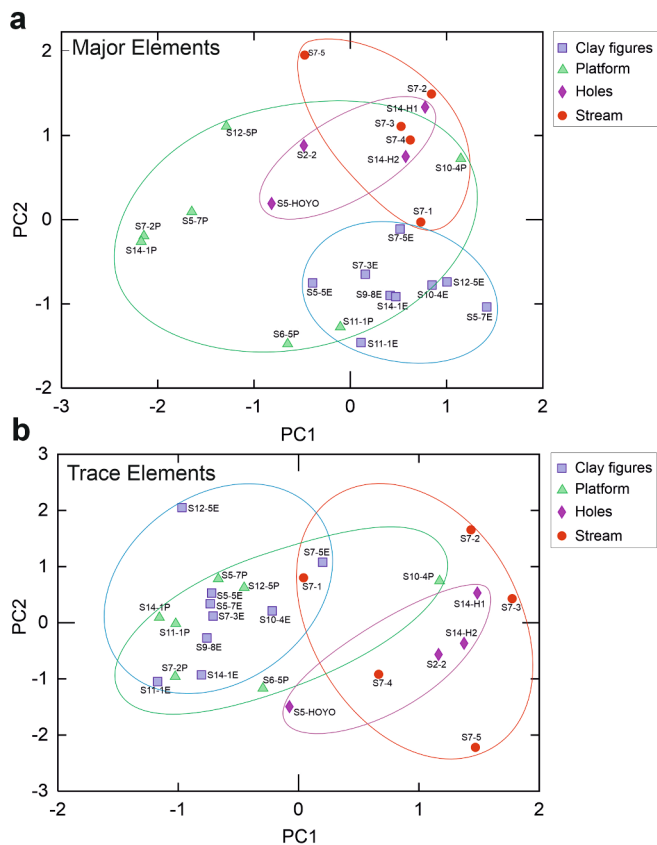


Fig. 19. PCA component analysis showing (a) major elements (SiO_2 , TiO_2 , Al_2O_3 , Fe_2O_3 , MnO , MgO , CaO , Na_2O , K_2O , P_2O_5) above and (b) traces (Ba, Ce, Cr, Ni, Pb, Rb, Sn, Sr, Y, Zn, Zr) below. Cumulative total variance explained by the first two principal components for major elements is 79.17%, and for trace elements is 78.31%. The ellipses were manually drawn to best fit and visually emphasize the groupings observed in the PCA score plots.

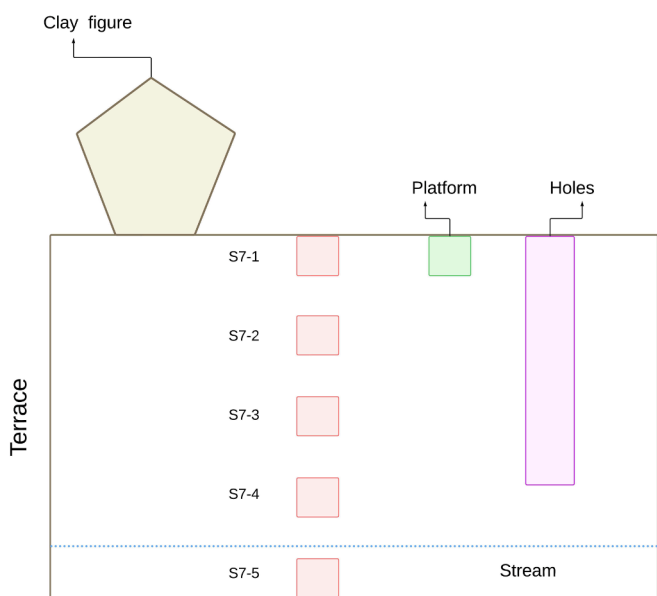


Fig. 20. Sketch showing the placement and spatial relation of samples in the stratigraphy of the terrace profile.

clay figures (E), holes and stream's profile also overlap perfectly, thus indicating that all have the same chemical composition, particularly with respect to the Main Elements. An interesting aspect to highlight is

that the composition of the trace elements of the sculpture samples is very similar to that of the platform samples and sample S7-1, thus possibly indicating that clay deposited on the upper levels was regularly used to produce clay figures. Nonetheless, the question remains about the function of the holes; thus, some other figures in the extensive repertoire that we did not sample may have been created with such material (Fig. 20).

By characterizing the pastes of selected clay figures, we know their chemical and mineral properties and different degrees of modeling. However, the pressure exerted in the latter process did not entirely modify the natural stratigraphic structure. In this sense, it is possible to notice the disposition of such stratification in different manners and directions depending on the greater or lesser pressure used to give the clay figures the desired shape. Moreover, while all sampled terraces confirm that these are natural sediments, sample S6.4E (which supports a female figure) shows a low degree of bending; thus, the artisans slightly modified this particular deposit. It is possible, therefore, to anticipate that some areas closer to the wall of the caves were marginally prepared while shaping the clay figures. On the contrary, other supports required a higher degree of modeling depending on the context, such as S9-8E, in which clay was mixed with stone slabs to stabilize a female figure's head resting on the cave wall (Fig. 21). Thus, clay was combined with other materials, such as pebbles, plates or pieces of stalactites, to give shape and support the earthen figures when desired, hence probably expediting the production process by needing less clay and effort in its extraction.

Moreover, the slight amount of pressure employed to shape the clay observed in the similar stratigraphic structure in both (E) and (P) also tells us more about the creation process, in that wedging and kneading the clay was not essential to create a homogeneous mass often used in pottery making. In this sense, wedging was not vital to ensure moisture distribution or remove air pockets for fear of exploding during the firing process (see Rice 1987: 119) because modeling unfired clay deploys other aspects of production, human experience, and specialized knowledge (see Rice, 1999). Also, this clay's intrinsic chemical and mineralogical properties are abundantly found within the cave (Table 3) and produced a sufficiently malleable material while relatively wet. The malleability resulted in those properties but also in the context of the cave, in which constant environmental conditions (i.e., high humidity, template temperature, and lack of sun and strong air currents) allowed the clay to remain flexible—and thus less prone to breaking while manipulating it. In this sense, the low altitude, the few entrances to the cave (airflow), and the presence of the stream and fauna (i.e., bats) have also contributed to the maintenance of constant humidity and temperatures, favoring the preservation of the materials. Yet, a more detailed study is needed to know how these factors fluctuate over time.

Certain minerals, such as quartz and kfeldspar (Table 3), acted as embedded temper (Allegretta et al., 2015: 221). Sand, as a non-plastic material and degreaser (*desgrasante* among Spanish speakers, Rice, 1987, p. 412) to reduce stickiness, and water to add plasticity were at hand when needed in the underground stream close to the terraces. These multiple practical considerations were recognized by the people participating in the production process of the clay reliefs and sculptures. However, the presence of charcoal in the pastes, often in traces, is entirely random and not intentionally added as an aggregate or filler. Our multi-technical approach did not show external or odd materials added into the mix, which we would have considered, perhaps, part of ritual activities or distinctive of the culture. The charcoal fragments visible in almost all (P) and (E) samples are not fossil charcoal but pieces of burned wood or plant materials due to their high degree of preservation. Charcoal presence bears witness instead to human activity within the cave. Access to the different sections in the depths of the cave requires walking along the stream and constantly getting on top and down the terraces. Periodic visits to the site would have caused the softening and mixture of the components on the cave floor. Hence, the concentrations of charcoal mixed with clay on the cave floor attest to



Fig. 21. A female's head resting on the cave wall, displaying the slabs used during its creation through natural erosion. Note the traces of red and black pigment. (Photo by Leslie F. Zubieta Calvert, 2018; courtesy of: Secretaría de Cultura.-INAH.-MEX. Reproduction of image authorized by Instituto Nacional de Antropología e Historia de México. Any request to reuse this image needs to be addressed to the aforementioned authority.) (c) 2018 Zubieta INAH research permit 401.1S.3–2018/654.

multiple activities such as the production process of the clay figures and paintings, ritual performances, and visits to the cave in a humid environment. Therefore, the dated charcoal collected in various sections (Table 6) provides an initial chronological framework between 607 and 881 CE to imagine those activities occurring during that time.

The fragment of *Pinus* sp., identified in one of the charcoal samples associated with the “monkey-like” figure (S10-2_C; Fig. 22) and those of the *Gymnospermae* group level (S14-1-C and S15-2-C), allows us to assume that high natural resin content materials were used for various purposes, including their smell and smoke for ritual activities conveyed in historical accounts and Mesoamerica's iconography (e.g., Houston and Taube, 2000: 265) or perhaps to light up the cave for ritual purposes and to illumine the space while paintings on the walls or shaping the clay figures (Zubieta 2021: 1176). Moreover, in line with the objectives of this paper, it is important to note that until now, the evidence shows that the modeled clay figures were not intentionally burned. Still, the question persists about whether the heat of burning torches or woody material with other purposes would suffice to partially dry and, consequently, harden the clay figures' exterior in such a humid environment. These fascinating aspects stimulate further research.

Finally, most of the radiocarbon dates acquired in this study, pointing to the Late Classic period (600–900 CE) or “Epiclassic” as coined by Wigberto Jiménez Moreno (1966: 49), is a period marked by the fall of Teotihuacan in the Central Highlands of Mexico (Moragas Segura, 2013; López Pérez and Nicolás Careta, 2023: 1026–1029) and by the eventual urban explosion of Monte Albán in the central valleys of Oaxaca and its abandonment around 850 CE (Winter, 2001: 293–298). The Late Classic/Epiclassic period falls into much debated chronological divisions in the Valley of Oaxaca (beyond the scope of the present study) known as Monte Albán IIIB–IV, Monte Albán IIIB and IV, and the Xoo Phase (Bernal, 1965: 804; Caso et al., 1967; Fahmel Beyer, 2015: 266, 272; Marcus and Flannery, 1990: 191–192; Martínez López et al., 2000:

1; Smith and Lind, 2005). Yet, it is too early to know the social, cultural and inter-regional implications of deploying chronological referents from the Central Highlands or the Central Valleys of Oaxaca in the Sierra Mixe of Oaxaca (see Fournier et al., 2006: 53, 72–75 for further discussion on the uncritical adoption of the Epiclassic term). Of interest here is the indication that the cave was visited earlier, during the Late Preclassic period, confirmed by S10-2_C (calibrated 151 BCE to 8 CE), which was found below the foot of a “monkey-like” sculpture. Such chronological range prompts us to imagine the production process of the clay figures through time in various events, perhaps to communicate multiple narratives with different levels of meanings (Zubieta, 2021: 1184).

5. Conclusions

Most clay figures inside the Cueva del Rey Kong-Oy (or Kondoy) were produced as *reliefs* whose volume protrudes from the cave's floor; thus, using the term “sculpture” as a general designation does not accurately describe the technique deployed by the artisans. Given the abundance and intrinsic properties of the natural deposits of clayey sediments inside the cave, this material was used as the primary raw resource. The malleability and elasticity of the provisions acquired inside the cave facilitated shaping, by hand, the clay figures in Cueva del Rey Kong-Oy. The artisans guaranteed that their earthen creations would last longer and stand erect by utilizing a mix of resources such as clay, pebbles, rocks and speleothems (Zubieta, 2021: 1177). Thus, the “volume” seems to be the vehicle of creative expression. The floor acted as a horizontal plane from where clay reliefs were projected from the floor's surface at different heights, almost acquiring a sculptoric category but not yet reaching a tridimensional form. However, the current appreciation of the volume of the clay reliefs, the treatment of the exterior surfaces (i.e., incising, impressing), or the application of slips or



Fig. 22. “Monkey-like” clay figure at the back of the wall. Note the placement of the extracted charcoal embedded at the base of its right foot. (Photo by Leslie F. Zubieta Calvert, 2018; courtesy of: Secretaría de Cultura.-INAH.-MEX. Reproduction of image authorized by Instituto Nacional de Antropología e Historia de México. Any request to reuse this image needs to be addressed to the aforementioned authority.) (c) 2018 Zubieta INAH research permit 401.IS.3–2018/654.

paint is arbitrary since the degree of erosion suffered over time (i.e., stream’s rising water, humidity) is yet unknown.

Analyzing the pastes allows us to explore some aspects of the elaboration of the clay figures through analytical methods. Similar characteristics between platform and clay figure samples confirm that endokarstic clay in the cave was used in the production process and that no tempering materials were added. Moreover, the collected clay was not subjected to high mixing and refining procedures, such as purification through decanting processes of aqueous solutions or mixing. In addition, the analyses of the black layers inside the samples and on the external part of specific samples show that artisans knew how to extract the manganese from clayey deposits *in situ* and use it to decorate specific clay figures, thus offering evidence of the provenience of the black pigment.

Moreover, the composition of the clay was similar in all the samples from different sections of the cave, thus suggesting that the methods and materials used in their production could have lasted centuries. Although there is likely evidence of a much earlier cave occupation in the archaeological deposits, as mentioned, most of the radiocarbon dates acquired in this study, pointing to the Late Classic/Epiclassic period (600–900 CE), would indicate the end episodes in which the cave was visited. However, although more radiocarbon dates are needed to better

understand the cave’s occupation, this systematic study has provided us with the first chronological framework linked to the production and interaction with unfired clay reliefs and sculptures in Mesoamerica’s underworld.

CRediT authorship contribution statement

Leslie F. Zubieta Calvert: Writing – review & editing, Writing – original draft, Visualization, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization. **Raffaella De Luca:** Writing – review & editing, Validation, Investigation, Formal analysis. **Adriano Guido:** Writing – review & editing, Validation, Investigation, Formal analysis. **Alessandra Pecci:** Writing – review & editing, Project administration, Methodology. **Domenico Miriello:** Writing – review & editing, Supervision, Methodology, Investigation, Formal analysis.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jasrep.2025.105277>.

Data availability

Data will be made available on request.

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