

## **Relative and Absolute Chronologies, Iconographic Sequences. Pigments and Pictorial Micro-Stratigraphies: Aouinet Azguer 1 Rock Paintings (Lower Draa, Morocco)**

**Martí Mas** (UNED), **Abdelkhalek Lemjidi** (INSAP), **Mónica Solís** (UNED), **Rafael Maura** (UNED), **Enrique Parra** (IPCE), **Pedro Pablo Pérez** (IPCE), **Tariq Jawhari** (UB), **Ahmed Oumouss** (CNPR), **El Mahfoud Asmahri** (IRCAM), **Naima Oulmakki** (INSAP), **Guadalupe Torra** (Archaeologist), **Javier Pérez** (Wellrounded 360°), **María García** (UNED), **Alfredo Maximiano** (UNED)<sup>1</sup>

**Abstract:** Aouinet Azguer 1 is one of the most representative painting rock shelters in Morocco due to the quantity and characteristics of the images on display, which were carried out over an extended time lapse. In Aouinet Azguer 1 and 12, we find some zoomorphous figures outlined in red whose composition, size, style and technique bear no relation to the rest of the motifs. These figures correspond to an early chronocultural first phase, they were painted choosing privileged or central positions and in most cases we are not able to identify them since, even if by their volumetric shape the images seem to follow a naturalist tendency, their execution is highly conventional. In this study, the AMS <sup>14</sup>C dating was possible on an anthropomorph of the third phase also painted in red (3770 to 3650 Cal BC, minimum age), on the basis of a well-known technique (oxalates dating). These results were completed using a methodology that combines physicochemical and micro-stratigraphic analyses used to interpret this dating, which is difficult to get it. Considering the need to put in first place the preservation of the rock paintings under study we could not get another one. Furthermore, a meaningful number of questions arise related to dating and cultural relationship to the first pictorial phase which has been defined upon traditional archaeological criteria, which we intend to propose as our leading hypothesis, by means of the analysis of archaeological environment from the Atlantic area, proposing a probable date (Later Stone Age-Early Holocene).

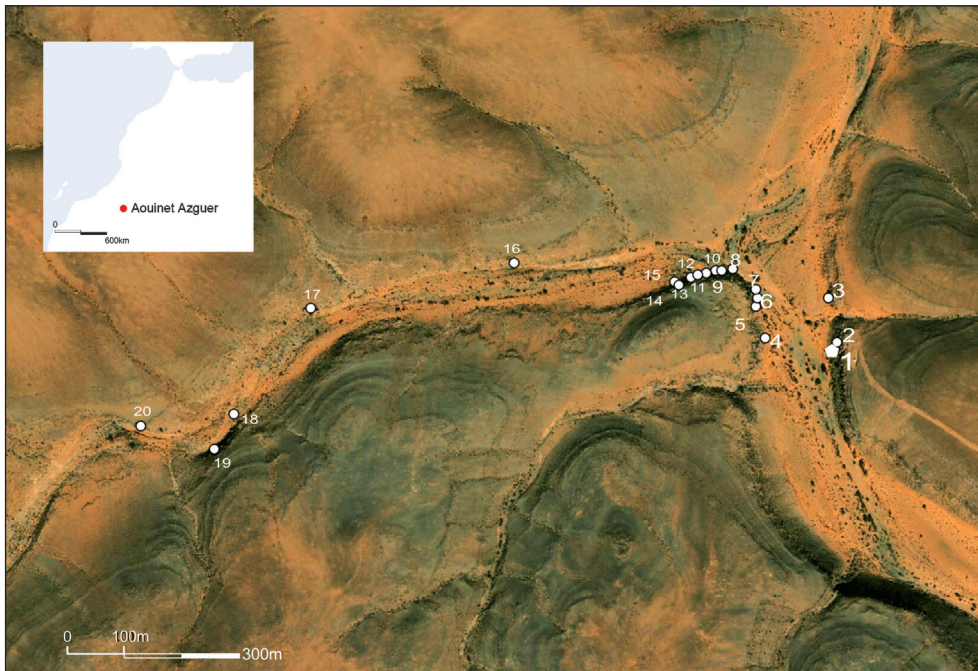
**Keywords:** Rock Art, Calcium Oxalate, Later Stone Age, Morocco, Atlantic Area.

---

1. Martí Mas, Departamento de Prehistoria y Arqueología, Universidad Nacional de Educación a Distancia – UNED, Madrid, co-director: *Tamanart Team*; Abdelkhalek Lemjidi, Institut National des Sciences de l'Archéologie et du Patrimoine – INSAP, Rabat, co-director: *Tamanart Team*; Mónica Solís, Departamento de Prehistoria y Arqueología, Universidad Nacional de Educación a Distancia – UNED, Madrid; Rafael Maura, Grupo de Investigación Paisajes, arquitecturas y cultura material en la Iberia antigua, Universidad Nacional de Educación a Distancia – UNED, Madrid; Enrique Parra, Instituto del Patrimonio Cultural de España – IPCE, Madrid; Pedro Pablo Pérez, Instituto del Patrimonio Cultural de España – IPCE, Madrid; Tariq Jawhari, Centres Científics y Tecnològics, Universitat de Barcelona – UB, Barcelona; Ahmed Oumouss, Centre National du Patrimoine Rupestre – CNPR, Agadir; El Mahfoud Asmahri, Institut Royal de la Culture Amazighe – IRCAM, Rabat; Naima Oulmakki, Institut National des Sciences de l'Archéologie et du Patrimoine – INSAP, Rabat; Guadalupe Torra, Archaeologist, Barcelona; Javier Pérez, Wellrounded 360° Fotografía, Nerja; María García, Departamento de Prehistoria y Arqueología, Universidad Nacional de Educación a Distancia – UNED, Madrid; Alfredo Maximiano, Departamento de Prehistoria y Arqueología, Universidad Nacional de Educación a Distancia – UNED, Madrid.

## 1. Introduction

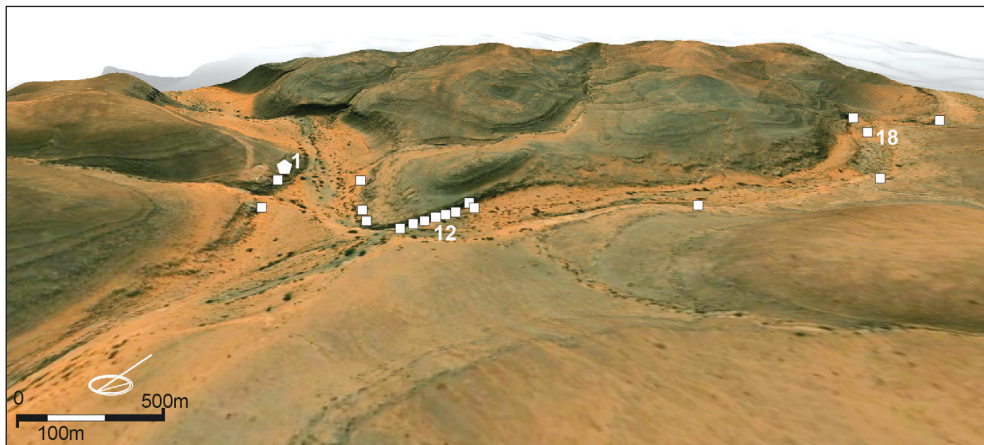
Since summer 2002 when a nomad woman named Fatimatou Malika Bent Benatta discovered some decorated rock shelters at Aouinet Azguer, Southeast of M'Sied (Tan Tan),<sup>2</sup> so far there have been located twenty rock shelters with prehistoric paintings (figs. 1 and 2). At the discovery time newspapers gave account of arguments amongst investigators whose opinions considered various chronologies based upon even opposing theories and methodologies.<sup>3</sup>



**Fig. 1:** Aouinet Azguer (Map of Morocco: data source [www.geamap.com](http://www.geamap.com) with gray Esri coverage layer. Orthophotos: Images taken from the free software (GNU license) SAS.Planet <http://www.sasgis.org/> with coverage: ESRI.ARCGIS.Clarity. Topographic data: GTOPO30, download of de NASA SRTM collection from de USGS Eart explorer portal. Data processed from platforms: QGIS 3.16.3 and Global Mapper 18).

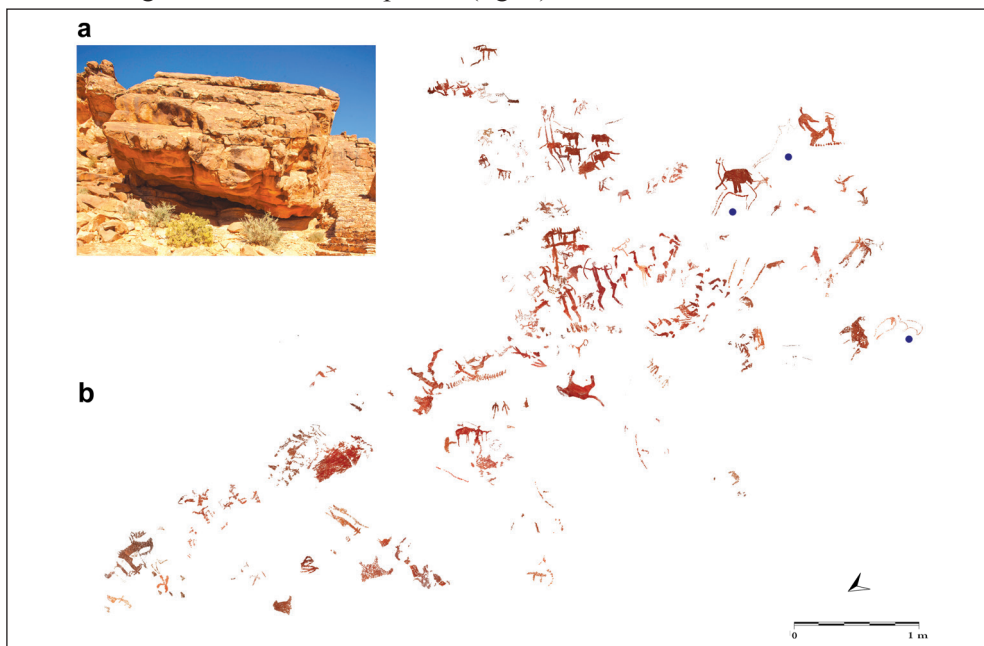
2. “Une découverte exceptionnelle,” *Aujourd’hui le Maroc* (October 4, 2002); “Les peintures de la discorde,” *Aujourd’hui le Maroc* (October 11, 2002).

3. Martí Mas Cornellà et al., “Proyecto Tamanart 2013-2014,” in *Excavaciones en el exterior 2013*, ed. C. Martín Morales, Informes y Trabajos 12 (Madrid: Ministerio de Educación, Cultura y Deporte, Subdirección General del Instituto del Patrimonio Cultural de España, 2015), 15-34; Martí Mas Cornellà et al., “Proyecto Tamanart 2014-2015,” in *Excavaciones en el exterior 2014*, ed. C. Martín Morales, Informes y Trabajos 14 (Madrid: Ministerio de Educación, Cultura y Deporte, Subdirección General del Instituto del Patrimonio Cultural de España, 2016), 12-27; Martí Mas Cornellà et al., “Proyecto Tamanart 2015-2016,” in *Excavaciones en el exterior 2015*, Informes y Trabajos 17 (Madrid: Ministerio de Cultura y Deporte, Subdirección General del Instituto del Patrimonio Cultural de España, 2019), 88-102.



**Fig. 2:** Aouinet Azguer (MDT 3D models. Orthophotos: Images taken from the free software (GNU license) SAS.Planet <http://www.sasgis.org/> with coverage: ESRI.ARCGIS.Clarity. Topographic data: GTOPO30, download of de NASA SRTM collection from de USGS Eart explorer portal. Data processed from platforms: QGIS 3.16.3 and Global Mapper 18).

It was made up of zoomorphs, anthropomorphs, chariots, abstract shapes and tiffinagh inscriptions. Figurative motifs were only outlined, filled or striped on the inside and different chromatic ranges are observed (red, brown, white...). The depicted fauna is diverse: elephants, giraffes, antelopes, gazelles, barbary sheeps, aurochs or bulls, carnivores, birds... Some of the abstract motifs are geometric patterns, also noting groups of fingering dots. We divide the graphic sequence of Aouinet Azguer 1 into different phases (fig. 3):



**Fig. 3:** a, Rock shelter (Aouinet Azguer 1). Geologically the Aouinet Azguer area falls within the Carboniferous materials of the North of the Tindouf Basin. b, Digital reproduction of pictorial composition. The most representative figures of phase 1 are marked.

Phase 1: It is characterised by zoomorphous figures of naturalistic tendency and the absence of anthropomorphous representations. In it, we consider a group of three outlined zoomorphs and other not defined shapes. Those red paintings are placed quite close to each other near the entrance of the rock shelter. From there on we may find the rest of the motifs on the ceiling and walls at successive phases.

Phase 2: This group is also comprised of naturalistic zoomorphous figures with inner filling, occasionally in very dark red tones, distinguished by an extreme degree of stylization and, some of them, by disproportionately elongated limbs. From time to time they may be found in couples or bigger groups. Due to different environmental conditions some of them show better appearance than others, depending on their location in the shelter.

Phase 3: The main feature is the addition of the male human figure into the iconographic repertoire. It is composed of very stylized forms, outlined and subsequently filled with stripes. These are zoomorphous figures and anthropomorphous males, usually in dark red tones. The anthropomorphs have been, occasionally, found in big formats showing a dexterous depiction of muscle, nudity and dynamic attitude, like those looking as athlete runners with small and elongated heads. They are found alone or in couples although do not appear to have any spatial link to the surrounding contemporary zoomorphs.

Phase 4: It is characterized by the inclusion of female human figures into the expressive speech. These are anthropomorphous and zoomorphous motifs painted with red inner filling. Sometimes show a bigger size than those from the previous group, lesser stylized and often drawn with a darker homogeneous applied pigment. The anthropomorphs are depicted individually or in couples and they may appear hand in hand. The male figures are mainly archers, establishing an interesting difference compared to the previous phase, in which men do not carry arches in any case. Also, they do not seem to be so dexterously depicted, neither they show athletic dynamism and their heads are much more rounded. Their genitals are explicit. Females are drawn with a small head and stretched horizontal arms above side by side breasts. They seem to be wearing tube skirts. Zoomorphs are painted in diverse formats showing lesser details than the previous group ones. The series of fingering dots, aligned or clustered, would also be included in this phase (fig. 4).



**Fig. 4:** Superimpositions defining the first four phases stratigraphy at Aouinet Azguer 1. a: gazelle (phase 1) underlying an elephant (phase 2). b: Antelope (phase 2) underlying a male anthropomorph (phase 3). c: gazelle (phase 2) underlying remains of undefined figure (phase 3). d: running anthropomorph (phase 3) underlying an archer (phase 4). e: ostrich (phase 2) underlying an ostrich head (phase 4). f: zoomorph (phase 3) underlying a couple (female and archer) (phase 4).

The final phases involve some extremely synthesized human figures and other schematic signs; a set of geometric patterns and representations of chariots; and another group characterized by uncouth zoomorphous figures painted in reddish-brown tones with inner filling.<sup>4</sup>

Since the publication of the first absolute datings (AMS <sup>14</sup>C) of cave paintings made with charcoal,<sup>5</sup> many others have been dated worldwide. However, absolute datings of paintings whose composition does not include this material are not frequent. A small number of these, obtained from direct samplings of rock paintings from the Central Sahara, have been published.<sup>6</sup> As it was assumed then, the first dating (Lancusi)

4. Mas Cornellà et al., "Proyecto Tamanart 2013-2014," 15-34; Mas Cornellà et al., "Proyecto Tamanart 2014-2015," 12-27; Mas Cornellà et al., "Proyecto Tamanart 2015-2016," 88-102.

5. Hélène Valladas et al., "Direct radiocarbon dates for prehistoric paintings at the Altamira, El Castillo and Niaux caves," *Nature* 357 (1992): 68-70.

6. Jean-Loïc Le Quellec, "Périodisation et chronologie des images rupestres du Sahara central," *Préhistoires Méditerranéennes* 4 (2013): 1-47.

was provided from the binder.<sup>7</sup> Other datings of red and white representations made from organic material related to the paint were also obtained in the same area.<sup>8</sup> These studies have been questioned as they provide little supporting documentation as well as some contradictions.<sup>9</sup> Six other datings from organic material are also difficult to interpret since the material that has been dated could not be related to the execution of the paintings but to the last phase of biological activity.<sup>10</sup> Finally, closer to our under study zone, three absolute datings of paintings from Ifran-n-Taska were obtained.<sup>11</sup> These would coincide with other proposed stylistic sequences,<sup>12</sup> with the exception of an anthropomorph that would reveal an older age. The last attempt to absolute dating has been at a Legteitira Shelter (Tiris). It is an undefined zoomorph. Spectroscopy Raman analysis and SEM-EDX have proven contents of calcium oxalate. Although authors have asserted the need for a micro stratigraphic analysis, so far it has not been possible because they feared there would not have enough carbon left for dating. Nevertheless, it is suggested that the calcium oxalate has been found mostly upon pigment. They underline the coherence of the result in relation to the archaeological context and suggest that it would be a probable chronological estimate. This dating pinpoints between 3005/2990 – 2930/2980 Cal BC.<sup>13</sup>

Taking relative and absolute chronologies into account, we propose, as this article goal and according to the already pointed out in previous articles, to reaffirm the antiquity of the first paintings and engravings from the Lower Draa.<sup>14</sup>

7. Rosanna Ponti, “Datation de l’art rupestre préhistorique: Problèmes et premières expériences sur les peintures du Sahara libyenne,” in *XIII International Congress of Prehistoric and Protohistoric Sciences*. Forlì (Italia) 8-14 September 1996. Colloquia 15. The Prehistory of Africa, ed. G. Aumassip, J. Desmond Clark and F. Mori (Forlì: Abaco, 1996), 71-73; Massimo Sinibaldi, S. Marchese and C. Desiderio, “Radiocarbon Dates for Prehistoric Rock Paintings at Tadrart Acacus, Libya,” in *XIII International Congress of Prehistoric and Protohistoric Sciences*. Forlì (Italia) 8-14 September 1996. Colloquia 15. The Prehistory of Africa, ed. G. Aumassip, J. Desmond Clark and F. Mori (Forlì: Abaco, 1996), 75-83; Fabrizio Mori, *The great civilisations of the ancient Sahara. Neolithisation and the earliest evidence of anthropomorphic religions* (Roma: L’Erma di Bretschneider, 1998).

8. Rosanna Ponti and Massimo Sinibaldi, “Direct dating of painted rock art in the Libyan Sahara,” *Sahara* 16 (2005): 162-65.

9. Le Quellec, “Périodisation,” 1-47.

10. Andrea Zerboni, “Rock Art in the Central Sahara (SW Libya): A Geoarchaeological and Palaeoenvironmental Perspective,” in *International Colloquium. The Signs of Which Times? Chronological and Palaeoenvironmental Issues in the Rock Art of Northern Africa*. Brussels, 3-5 June, 2010, ed. D. Huyge, F. Van Noten and D. Swinne (Brussels: Royal Academy for Overseas Sciences, 2012), 175-95.

11. Ahmed Skounti et al., “Rock Art and Archaeology in Ifran-n-Taska (Eastern Jebel Bani, Morocco): First Results of the Moroccan-Italian Research Project,” in *International Colloquium. The Signs of Which Times? Chronological and Palaeoenvironmental Issues in the Rock Art of Northern Africa*. Brussels, 3-5 June, 2010, ed. D. Huyge, F. Van Noten and D. Swinne (Brussels: Royal Academy for Overseas Sciences, 2012), 109-36.

12. Joaquim Soler Subils, *Les pintures rupestres prehistòriques del Zemmur (Sahara Occidental)* (Girona: Documenta Universitaria, 2007).

13. Andoni Sáenz de Buruaga and Agustín Ezcurra, “Una estimación de cronología absoluta para una pintura rupestre del abrigo de Legteitira-6 (Agüenit, Sahara Occidental),” *Almogaren* 51 (2020): 57-73.

14. Abdelkhalek Lemjidi, “Arts rupestres,” in *Le Patrimoine Culturel du Maroc*, ed. A. Retnani (Casablanca: Éditions La Croisée des Chemins – Ministère de la Culture et de la Communication, 2019), 60-81.

## 2. Methodology

### 2.1. Documentation of rock art

The replica of motifs, compositions and painted panels is performed from high-definition calibrated digital photographs following computerised processes by means of image-editing software. The procedure consists in discriminating pigment and rock surface on the image to finally eliminate the latter so that only the painting remains. By carrying out the process on photographic support the images are not only more reliable and approached without preconceptions but we also avoid direct contact with depictions.<sup>15</sup>

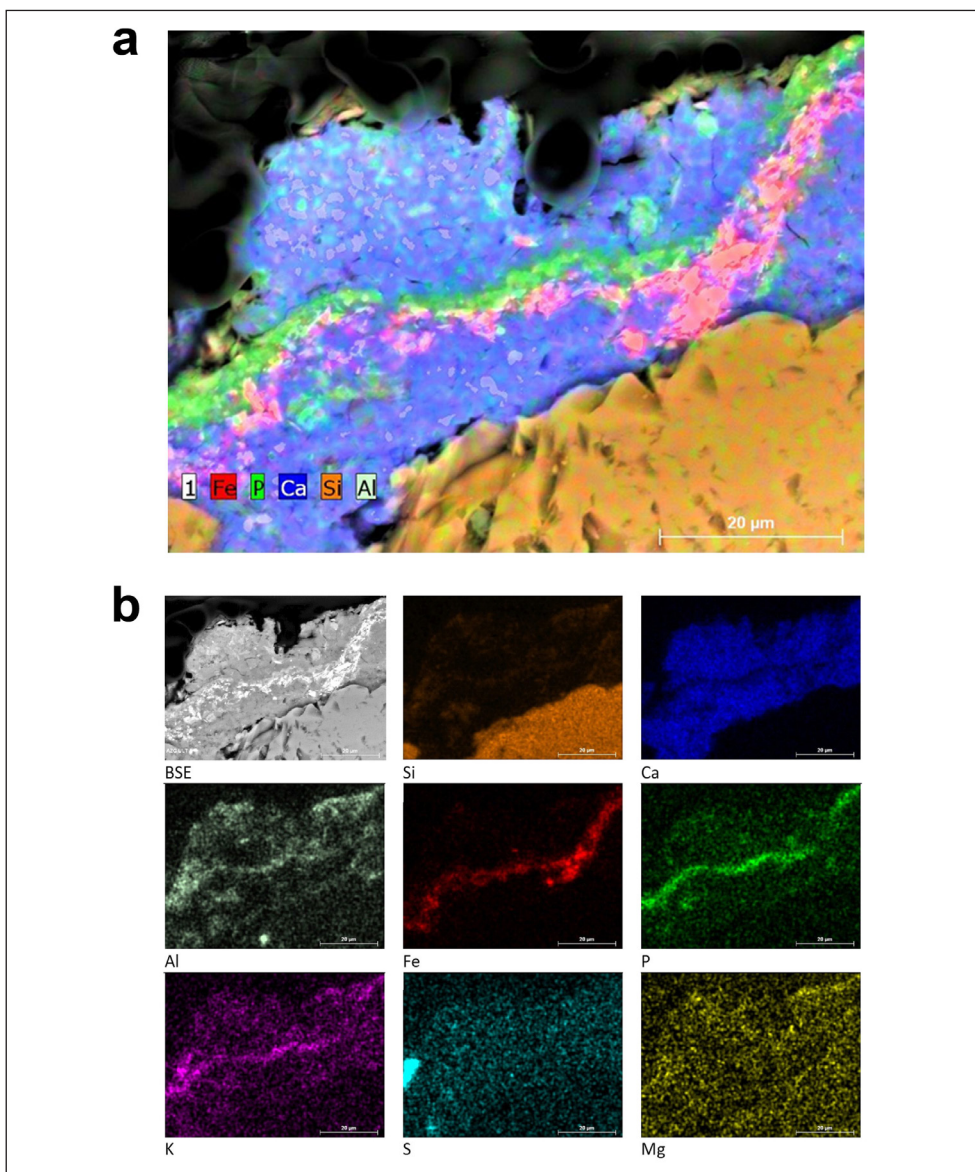
### 2.2. Sampling and sample preparation

To minimise the potential deterioration of the figure and to facilitate the extraction of the sample we target areas with small cracks, scales or flakes. The sampling techniques follow previously established protocols: micro-samples (smaller than 0.5 mm) are obtained using sterile scalpels (discarded after each and every use), latex gloves and masks to prevent any possible contamination.<sup>16</sup> Once they have been collected, they are stored in cryotubes or *Eppendorf* tubes. The entire process is documented through photographs. It was possible to obtain the sample from Aouinet Azguer 1 (slightly bigger in order to enable dating) because we noticed that part of the figure showed cracks which facilitated the collection of a flake that was peeling off and that would have been lost if we had not acted this way. Once in the laboratory, the samples were microphotographed, measured, embedded in methacrylate resin and cut transversely for their SEM-EDX and Raman analysis taking the micro-stratigraphy into consideration. In this case, this was the procedure for the SEM-EDX analyses, but it was noted that the resin had penetrated the sample eliminating or hiding the calcium oxalate (fig. 5a) and consequently distorting the results and hence altering the microtopography of the sample. Therefore, a different course of action was followed in regard to the Raman analyses as described below. Subsequently, we have worked it out based upon microfragments detached from de original sample.

---

15. Martí Mas Cornellà et al., "Reproducción digital, microfotografía estereoscópica y fotografía esférica aplicadas a la interpretación del arte rupestre prehistórico," *Cuadernos de Arte Rupestre* 6 (2013): 77-83.

16. Martí Mas et al., "Minateda rock shelters (Albacete) and post-palaeolithic art of the Mediterranean Basin in Spain: pigments, surfaces and patinas," *Journal of Archaeological Science* 40 (2013): 4635-47.



**Fig. 5:** SEM-EDX analysis. a, SEM image with a superimposition of the distribution of iron, phosphorus, calcium, silicon and aluminium. b, Individual distribution maps of different elements (silicon, calcium, aluminium, iron, phosphorus, potassium, sulphur and magnesium) on an image of retrodispersed electrons. There is a significant concentration of silicon (Si) in the substratum and lesser of it in the layer of paint and in the upper layer of oxalate. Calcium (Ca) appears associated with the oxalate layers. Aluminium (Al) appears related to the layer of paint and to the environmental deposits of sample surface silicated nature. Iron (Fe) is related to the layer of paint. A concentration of potassium (K) and especially phosphorus (P) appears on a thin level located above the iron-enriched layer. The magnesium (Mg) content is very low, showing a higher concentration on the external surface in relation to environmental particles.



### 2.3. Stereoscopic microscopy

A stereoscopic microscope was used to obtain an initial approximation to the visual aspect of the micro-sample (rock surface and superficial pigments). The stereoscopic microscope used was a Nikon SMZ 100 also employed to obtain the images of the samples studied. A reflected light microscope has been used for the textural and compositional description of the sample. An Olympus BX 51 with a fitted ultraviolet lighting system (Wood's lamp) was used.

### 2.4. Radiocarbon datings

The sample was dated by Beta Analytic Radiocarbon Dating Laboratory. The red paint could not be separated from the fragment of rock surface. It was subjected to low-temperature combustion to obtain the necessary amount of carbon to conduct the dating. We dated Aouinet Azguer 1 on the basis of a proven process<sup>17</sup> which has already been applied (and has been under continuous improvement) in the Mediterranean Arc of the Iberian Peninsula,<sup>18</sup> as well as in many other places: calcium oxalate dating. Some pigment characterisations have proven, in certain rock paintings, the absence of organic matter which could be considered as binder and, if such binder were to appear, it would be contaminated by old-fashioned methods of research and tracing and would, therefore, give a non-reliable dating. Only a virgin site, free of human intervention, could be taken into consideration.<sup>19</sup> This can be argued but Aouinet Azguer 1 is a relatively recent discovery that has not been systematically studied up until now; it had only been previously mentioned in some articles.<sup>20</sup> It should be noted that rock paintings (very scarce in Morocco, unlike engravings which are very abundant) had not been studied as carefully as they deserve, except by some works.<sup>21</sup>

---

17. Marvin W. Rowe, "Dating by AMS radiocarbon analysis," in *Handbook of rock art research*, ed. D.S. Whitley (Walnut Creek: Altamira Press, 2001): 139-66.

18. Juan Francisco Ruiz et al., "Premières datations radiocarbone d'encroûtements d'oxalate de l'art rupestre préhistorique espagnol – First radiocarbon dating of oxalate crusts over spanish prehistoric rock art," *International newsletter on rock art– Lettre internationale d'informations sur l'art rupestre* 46 (2006): 1-5.

19. Jacques Brunet, Sylvie Demailly and Pierre Vidal, "Résultats de l'étude de prélèvements de peintures des abris du Tassili N'Ajjer," *Ars Praehistorica* 7-8 (1988-1989): 293-303; Malika Hachid et al., "Quelques résultats du projet de datation directe et indirecte de l'art rupestre saharien," in *The Signs of Which Times? Chronological and Palaeoenvironmental Issues in the Rock Art of Northern Africa*. International Colloquium. Brussels, 3-5 June, 2010, ed. D. Huyge, F. Van Noten and D. Swinne (Brussels: Royal Academy for Overseas Sciences, 2012), 71-96.

20. Suzan Searight and Guy Martinet, "Peintures rupestres d'un nouveau genre dans le Sud marocain," *Sahara* 13 (2001-2002): 115-18; Suzan Searight-Martinet, "Holocene Rock Art in Morocco: Hard Facts, Hesitant Hypotheses and Hopeful Headway," in *International Colloquium. The Signs of Which Times? Chronological and Palaeoenvironmental Issues in the Rock Art of Northern Africa*. Brussels, 3-5 June, 2010, ed. D. Huyge, F. Van Noten and D. Swinne (Brussels: Royal Academy for Overseas Sciences, 2012), 97-108.

21. Renate Heckendorf and Abdellah Salih, "Les peintures rupestres au Maroc: État des connaissances," *Beiträge zur Allgemeinen und Vergleichenden Archäologie* 19 (1999): 233-257.

## 2.5. Raman spectroscopy

The micro-Raman spectra were obtained with a Jobin Yvon LabRam 800HR dispersive system coupled to an optical microscope Olympus BXFM. The CCD detector was cooled at  $-70^{\circ}\text{C}$ . The measurements were made with a solid state laser emitting at 532 nm. The laser power at the sample was low, i.e. 0.5 mW in order to avoid a possible degradation of the sample. No sample preparation was needed for the micro-Raman analysis. The cross-section of the tiny rock fragment was simply studied by mounting the sample on a special aluminium holder placed under the microscope objective. An x50 microscope objective was used and the Raman spectra were typically recorded with an acquisition time of 10 sec with 10 acquisition spectra. The Raman intensity of the different bands analysed in this study corresponds to the peak height of these bands after submitting all spectra to a background correction. It was decided to analyse the small fragment of painting without any sample preparation such as microtoming the cross-section, in order to avoid any kind of alteration of the sample. As a result, the fragment appears quite inhomogeneous when viewed with the optical microscope. This is the main reason why it was decided to repeat the Raman analysis along three different lines.

## 2.6. SEM-EDX

Scanning Electron Microscopy and microanalysis by Energy Dispersive X-Ray Spectroscopy have been used to complete the textural and compositional analysis of the micro-samples. The equipment used to carry out this analysis has been a Hitachi S-3400 located in the Centro de Microscopía Electrónica *Luis Bru* of the Universidad Complutense de Madrid.

## 2.7. Gas chromatography

A tiny fragment of pigment (0.0389 mg) was introduced in a 1.5 mL *Eppendorf* vial. 50  $\mu\text{L}$  of 6 M HCl was added. After gas evolution, dry nitrogen was passed through. The vial was immediately capped, and heated at  $110^{\circ}\text{C}$  in a microwave oven (900W, 45 min) using 250 mL of water bath to absorb the excess of microwave radiation. The vial was evaporated under vacuum. 50  $\mu\text{L}$  of acetonitrile (Baker, LC-MS grade) and 25  $\mu\text{L}$  of *tert*-butyldimethylsilyl trifluoroacetamide (TBDMS-TFA, Sigma) were added. The sample was sonicated for 6 min, centrifuged, and the liquid solution was injected (5  $\mu\text{L}$ ) in a gas chromatograph (Agilent 5820A) with a split / splitless injector working in splitless mode at  $290^{\circ}\text{C}$ . The capillary column (Agilent HT5 30 m, 0.25 mm internal diameter) was subjected to a temperature gradient of  $65^{\circ}\text{C}$  (2 min),  $20^{\circ}\text{C}/\text{min}$  to  $300^{\circ}\text{C}$  (15 min). The detector is a mass spectrometry detector Agilent MSD 5977 working at 70 eV.<sup>22</sup>

---

22. Maria Perla Colombini, Francesca Modugno and Erika Ribechini, "GC/MS in the Characterization of Lipids," in *Organic Mass Spectrometry in Art and Archaeology*, ed. M.P. Colombini and F. Modugno (Wiley, 2009), 191-214; Francesca Modugno and Erika Ribechini, "GC/MS in the Characterization of Resinous Materials," in *Organic Mass Spectrometry in Art and Archaeology*, ed. M.P. Colombini and F. Modugno (Wiley, 2009), 215-236; Maria Perla Colombini and Gwänelle Gautier, "GC/MS in the Characterization of Protein Paint Binders," in *Organic Mass Spectrometry in Art and Archaeology*, ed. M.P. Colombini and F. Modugno (Wiley, 2009), 237-60.

## 2.8. Petrographic microscope and X-ray diffraction

Geological characterisation of the rock surface was obtained from a sample taken outside the shelter. This was a white quartzite on fresh fracture which showed signs of rubefaction on its surface. The sample was obtained from a distant point away from the paintings without affecting by any means neither the decorated panels nor their visualisation. A fragment was analysed by X-ray diffraction and a thin rock section was studied through a petrographic microscope. Transmitted light optical microscopy has been used for the petrographic study with polarised light of the above-mentioned rock section. The section was prepared by the Servicio de Preparación de Muestras of the Departamento de Petrología y Geoquímica of the Universidad Complutense de Madrid. The petrographic microscopes used are Carl Zeiss Standard W.L. and Axiophot P.D. photomicroscope. The latter also used for taking the photographs. This analytical technique allows us to find out what the mineralogy, texture and classification of the rock are. The crystalline phases present in the rock have been identified through X-Ray Powder Diffraction (XRD) after the grinding process of the sample. The equipment used was a diffractometer Siemens D 5000 which features a programme of automatic identification of phases that counts with the support of a JPDS-PDF2 compounds database with 137.000 reference patterns of crystalline phases (mineral subbase with 15.800 patterns). The radiation is  $K\alpha$ Cu, graphite monochromator, with a potential difference in the tube of 40 Kv and an intensity of 20 mA in the filament. The analyses have been carried out in the  $2\theta$ : 2-60° range.

## 3. Aouinet Azguer 1

### 3.1. Datations, pigments and pictorial micro-stratigraphies

Here, we introduce the AMS  $^{14}\text{C}$  dating and interpretation of Aouinet Azguer 1's rock painting sample (664 mg) obtained from a large red anthropomorph (fig. 6). The result is 3770 to 3650 Cal BC (Beta-415957):

Measured Radiocarbon Age: 4690 +/- 30 BP.  $\delta^{13}\text{C}$ : -10.4 o/oo. Conventional Radiocarbon Age: 4930 +/- 30 BP. 2 Sigma calibration (INTCAL13): Cal BC 3770 to 3650 (Cal BP 5720 to 5600) (fig. 7).

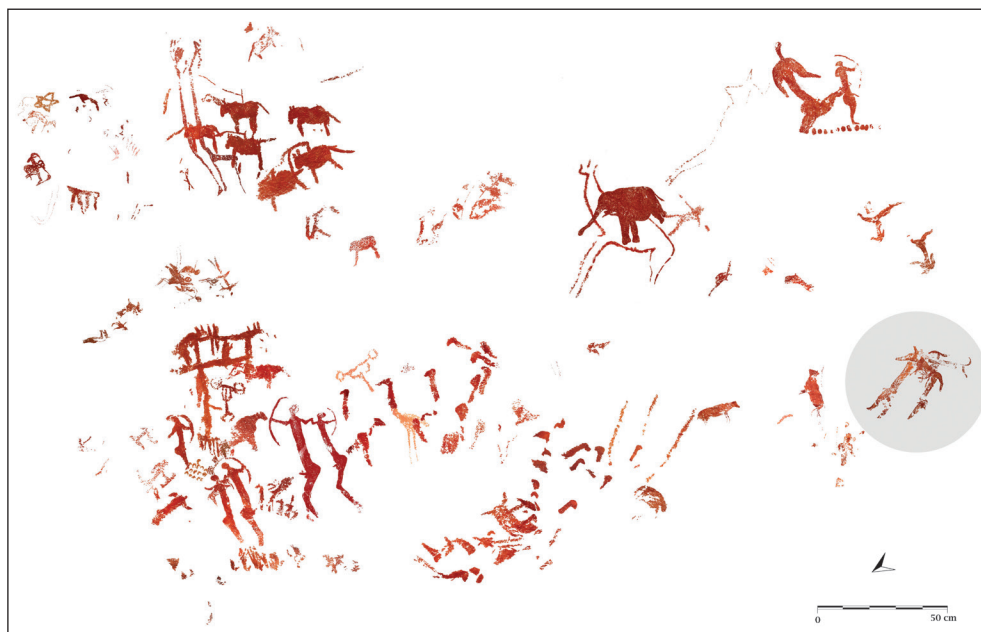
In view of its success, we tried to date a second fragment at Aouinet Azguer 12 (rock shelter 5 before the last surveys)<sup>23</sup> from a central red outlined zoomorph but it was only possible to obtain a sample of 34 mg without altering the figure (fig. 8). The results have been negative as there was not enough carbon.

A very small fragment was analysed by Raman spectroscopy. Three different transversal lines were analysed from the edge of the fragment towards the interior of the rock (cross-section of micro-sample) (fig. 9). The origin, i.e. 0  $\mu\text{m}$ , was defined at the edge of the sample that was in contact with air. Along the analysed lines,

23. Mas Cornellà et al., "Proyecto Tamanart 2013-2014," 15-34; Mas Cornellà et al., "Proyecto Tamanart 2014-2015," 12-27; Mas Cornellà et al., "Proyecto Tamanart 2015-2016," 88-102.

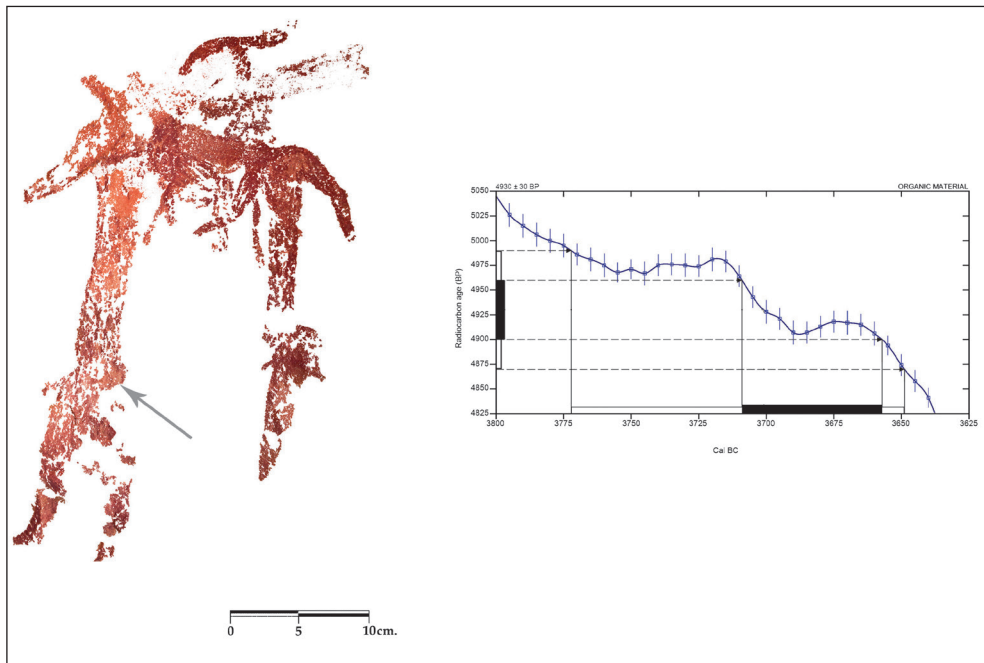
three compounds were detected, calcium oxalate, hematite and quartz, sometimes mixed to varying degrees. The spectra of calcium oxalate obtained were found to correspond to well-crystallised whewellite  $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ .<sup>24</sup> For example, figure 9d presents the Raman spectrum obtained at position 8  $\mu\text{m}$  of line 1. This spectrum shows the Raman bands of both whewellite and hematite, indicating therefore a mixture of whewellite and hematite.

Figures 9a, b and c show the evolution of the Raman intensity of the bands at 1462, 222 and 463  $\text{cm}^{-1}$  (oxalate, hematite and quartz), along the transversal distance for each of the three lines which present a very similar pattern. First, a large amount of calcium oxalate is detected close to the surface in contact with air; then it is found to decrease abruptly and seems to disappear between 10-20 microns approximately, depending on the line. Calcium oxalate reappears again at a distance of about 25-50  $\mu\text{m}$ , reaching its maximum intensity at a distance varying between 30 to 70  $\mu\text{m}$ , and then it decreases progressively disappearing above a distance of 35-85  $\mu\text{m}$ . Further in these three plots, haematite is found to appear between the two layers of calcium oxalate described above and the thickness of the haematite layers varies between 16 and 60  $\mu\text{m}$  approximately. Finally, quartz, which corresponds to the rock substrate, is detected at a distance between 35 and 70  $\mu\text{m}$ .

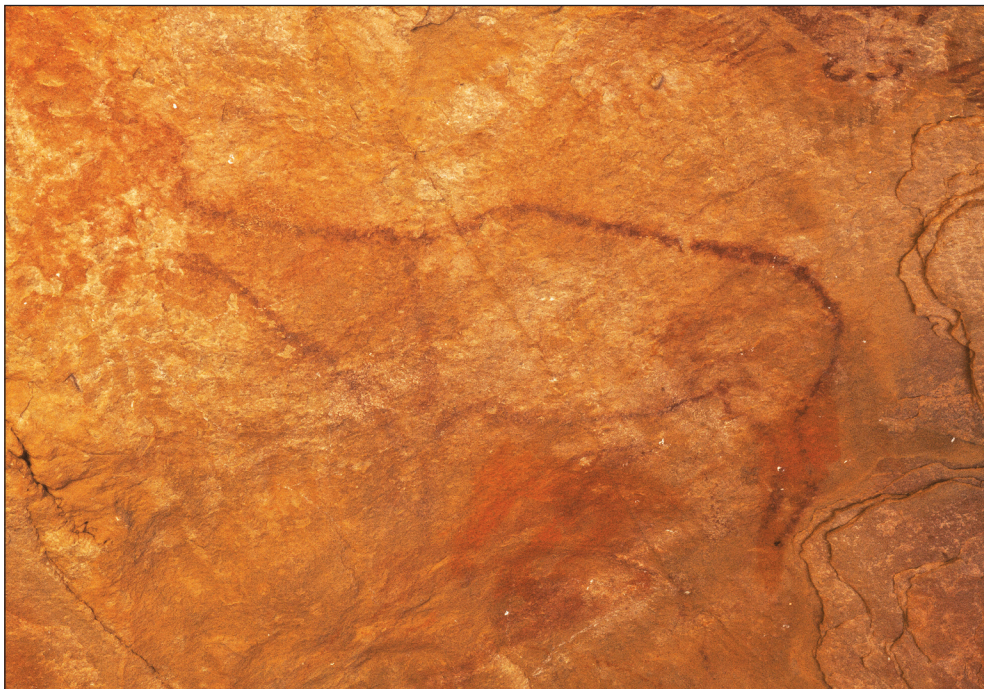


**Fig. 6:** Composition chosen for sampling.

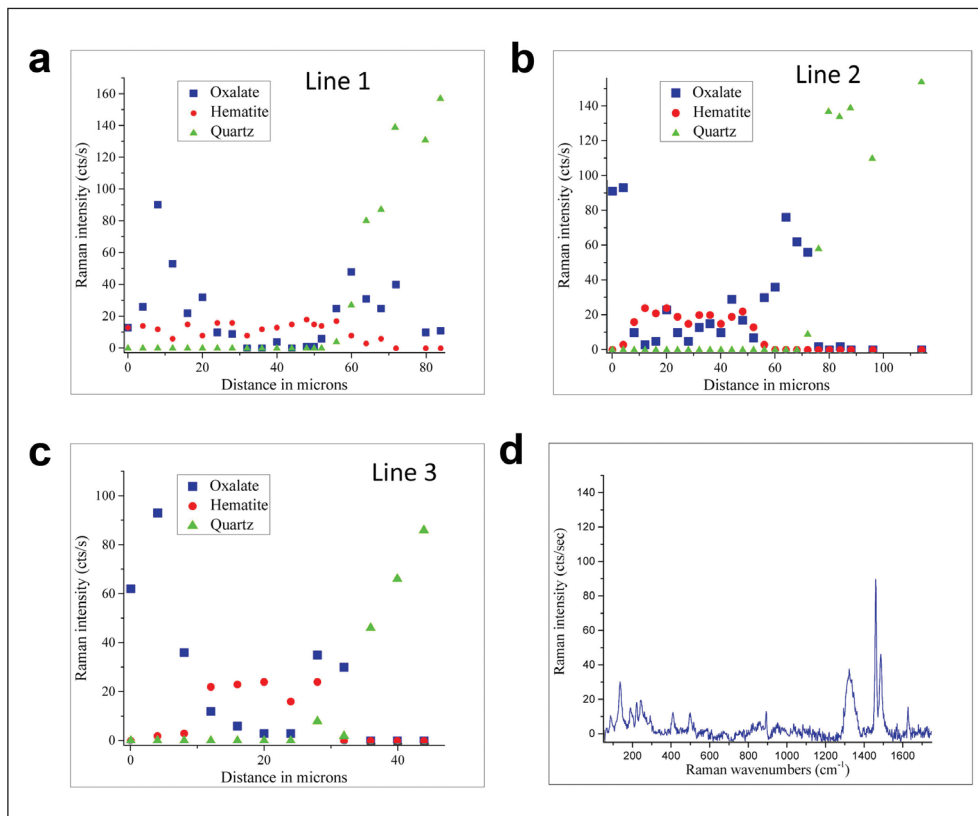
24. Ray L. Frost and Matt L. Weier, "Thermal treatment of whewellite – a thermal analysis and Raman spectroscopic study," *Thermochimica Acta* 409 (2004): 79-85; Claudia Conti et al., "Micro-Raman depth profiling on polished cross-sections: the mapping of oxalates used in protective treatment of carbonatic substrate," *Journal of Raman Spectroscopy* 39 (2008): 1307-8; Claudia Conti et al., "Synthesis of calcium oxalate trihydrate: New data by vibrational spectroscopy and synchrotron X-ray diffraction," *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy* 150 (2015): 721-30.



**Fig. 7:** Sampled red anthropomorph (Aouinet Azguer 1 third phase) and calibration curve of radiocarbon age into calendar years. The arrow indicates the point where the sample was obtained.



**Fig. 8:** Central representation of the rocky wall at Aouinet Azguer 12 (antelope).

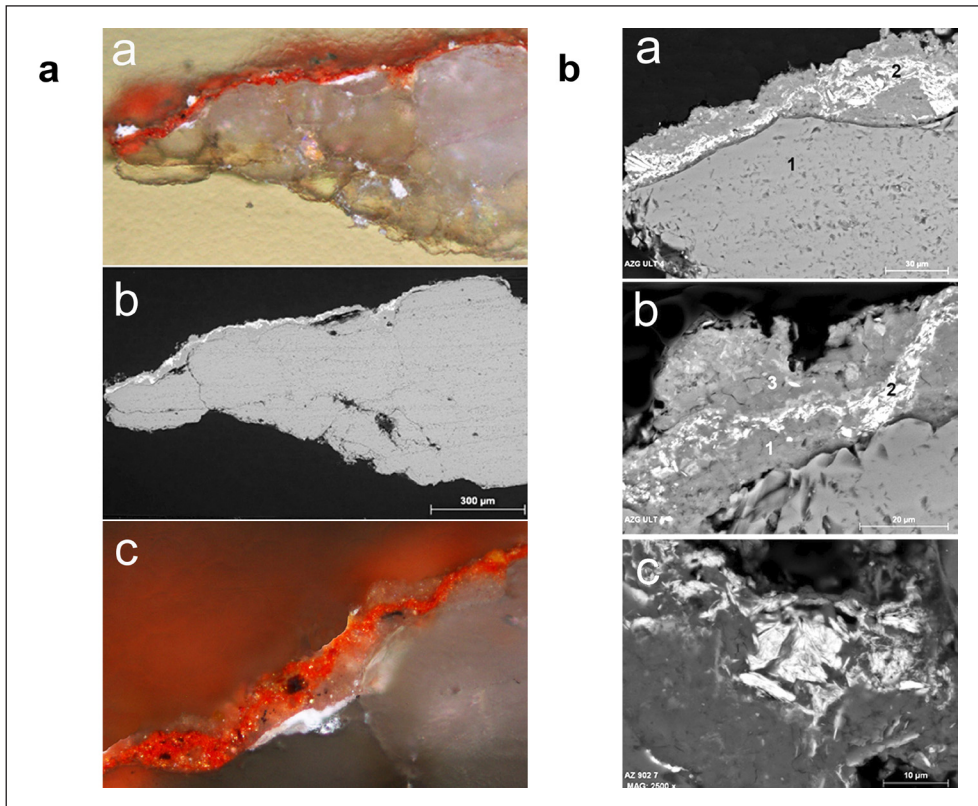


**Fig. 9:** Lines and position (8  $\mu\text{m}$  of line 1) analysed by Raman. a, Line 1. b, Line 2. c, Line 3. d, Raman spectrum obtained along line 1 at the position 8  $\mu\text{m}$ . The baseline was corrected. The fact that we have found a strip without calcium oxalate in the interior of the red pictorial layer confirms that this would not be present in the raw material (pigment) used to paint.

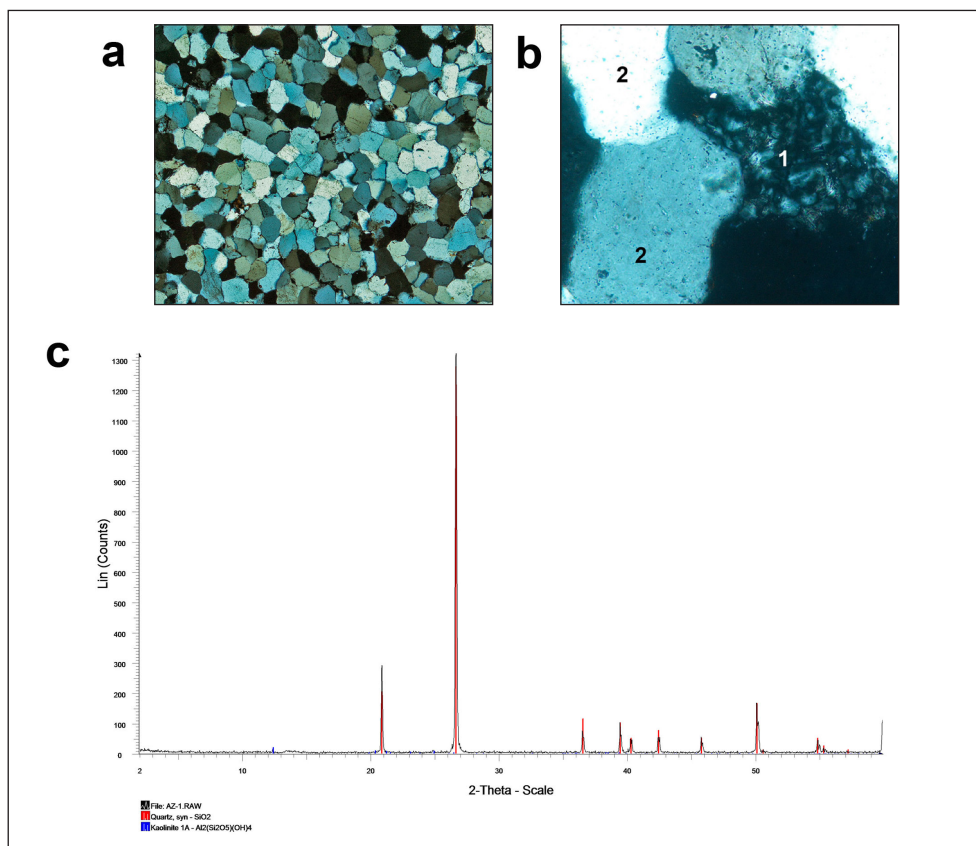
The study through reflected-light optical microscopy and SEM-EDX of a stratigraphic section perpendicular to the coat of paint indicates the existence of a rock surface composed of siliceous grains. A homogeneous layer of varying thickness formed of calcic and siliceous material appears above this surface. The next layer, fairly thin, is rich in silicon and, particularly, in iron. Finally, the outer layer (in contact with air) is characterised by high calcium contents, although in some areas there are also significant contents of silicon and aluminium and, in others, concentrations of sulphur and calcium (fig. 10).

The above data are fairly consistent with those given by Raman spectroscopy: the rock surface corresponds to a quartzite formed by medium sand-sized quartz grains with a rounded morphology (fig. 11). A layer of varying thicknesses spanning from 5 to 60  $\mu\text{m}$  formed mainly by calcium oxalate and silicates appears above this irregular siliceous material (maximum thicknesses are circumstantial, appearing on fissures and concavities or, for example, amongst grains of quartz that can favour the formation of punctual accumulations of oxalate, although in other cases this concentration is practically non-existent. The layer of paint has an average

thickness of 10  $\mu\text{m}$  and is formed by iron oxides (hematite) and lesser amounts of iron-enriched silicates. Finally, we identified a layer 10 to 20  $\mu\text{m}$  thick, high in oxalates and with lesser amounts of silicates (that fails to appear in some areas) above the layer of paint. These thicknesses need to be considered with caution: they would be larger if we take into account the problematic explicitly stated regarding the microtopography of the sample (see Methodology. 2.2. *Sampling and sample preparation*). Environmental deposits of silicates and gypsum appear on a discontinuous basis above this layer (fig. 12). Elements distribution map provides a graphic illustration of this (fig. 5).



**Fig. 10:** Cross-sections and SEM images of painting micro-sample. a: a and b, general image of optical microscopy and electron microscopy corresponding to the SEM-EDX analysed sample. c, optical microscopy detailed image where the red pictorial layer stands out against the quartzitic substratum. b: a, quartzitic substratum (1) and red paint layer (2). b, detail where the presence of the hematite-enriched red paint layer (2) between the layers of oxalates and silicates (1 and 3) is observed. c, detail where the hematite grains which characterise the red paint layer can be seen.



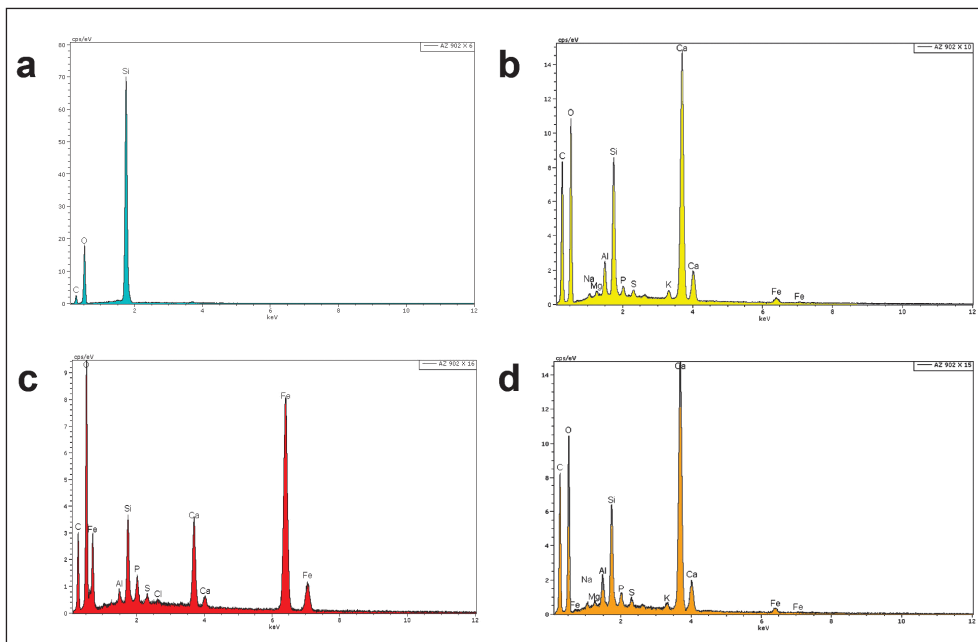
**Fig. 11:** Fine-grained sandstone (125-250  $\mu\text{m}$ ) to medium-grained (250-500  $\mu\text{m}$ ). a, General microscopic appearance of the quartzitic substratum mainly formed by grains of quartz. b, Detailed image where cementation of kaolinitic clays (1) amongst quartz grains (2) can be seen. c, Diffractogram corresponding to the quartzitic rock substratum.

The interpretation of some of these elements has been approached in other works in which the raw material used for painting (iron oxides and terrigenous sediments) could possibly also come from the lithogenesis of the surrounding environment.<sup>25</sup>

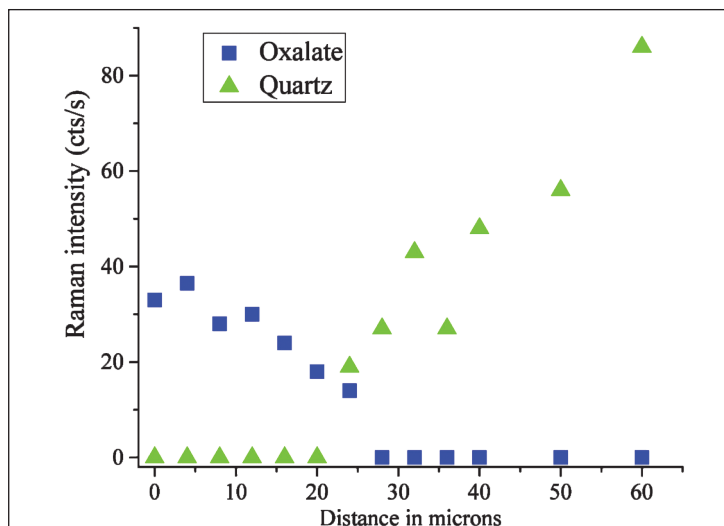
Furthermore, an additional Raman line was carried out along a non-painted microfragment which was obtained very close to the painted sample (fig. 13). A clear Raman signal of calcium oxalate is observed near the external surface and its intensity appears to decrease progressively inside the rock down to 24  $\mu\text{m}$ , value where the quartz signal is first detected. Above this value, the Raman signal of calcium oxalate has totally disappeared and only quartz is detected. These results indicate that in the non-painted rock, calcium oxalate is found near the surface in contact with air and its thickness is about 20 microns. No hematite was observed along the cross-section.

25. Mas et al., "Minateda," 4645-46.





**Fig. 12:** EDX microanalysis. a, rock substratum. b, oxalates and silicates lower layer. c, red paint layer. d, oxalates and silicates upper layer.



**Fig. 13:** Raman line of a cross-section on a non-painted fragment.

In order to identify and evaluate the approximate amount of organic material, total acid hydrolysis of the paint sample was achieved. The main fraction is represented by little acidic metabolite molecules (fig. 14). Two compounds are prominent (fig. 15): glycolic acid and oxalic acid. Some other metabolite molecules are detected, e.g. maleic and succinic acids, which also belong to general metabolic chains of aerobic

microorganisms.<sup>26</sup> The second category is compounds related to lipids. These should be triglycerides such as glycerol and saturated fatty acids (mainly palmitic and stearic acids) that should have some relation to the possible original organic binder of the pigments as well as to biodeterioration and contamination due to human or animal contact with the painted surface, also considering other interesting hypothesis.<sup>27</sup>

The total amount of organic compounds detected, excluding oxalic acid, can be approximately calculated from the total area of the chromatogram and means less than 0'0001 mg. Some anions (inorganic salts: phosphate and borate) are detected together with this organic compounds. Phosphate could be related to the use of organic proteinaceous materials as milk, collagen or blood. Also, it could be deposited by microorganisms as part of their biochemical activity. Borate is normally associated with trace minerals that can be found in the surroundings of the archaeological site.

We have dated the calcium oxalate which appears imbricated with hematite on the outer surface and inter-linked with the rock substratum and hematite. In the second case, it could be either a degradation of the organic substances (binders),<sup>28</sup> an important hypothesis in this case, or be present in the rock surface prior to painting (precipitated from oxalic acid secreted during the biological activity of microorganisms). If the latter were the case, bearing also in mind that neither its quantity nor its intensity seems to be very significant, it could contribute to age the dating. However, the abundance of oxalate inter-linked with hematite on the outer layer, taking into account that it obviously presents a rejuvenation process, would lead us to give the dating a more recent date than that of the execution of the painting, although the pigment coat could also be sealing the calcium oxalate of the interior (in contact with the rock surface), thus preventing its recovery (when the sample was subjected to a low combustion temperature to obtain carbon), in which case, the oxalate of the inner part would not be represented in the result and the painting would be even older.

---

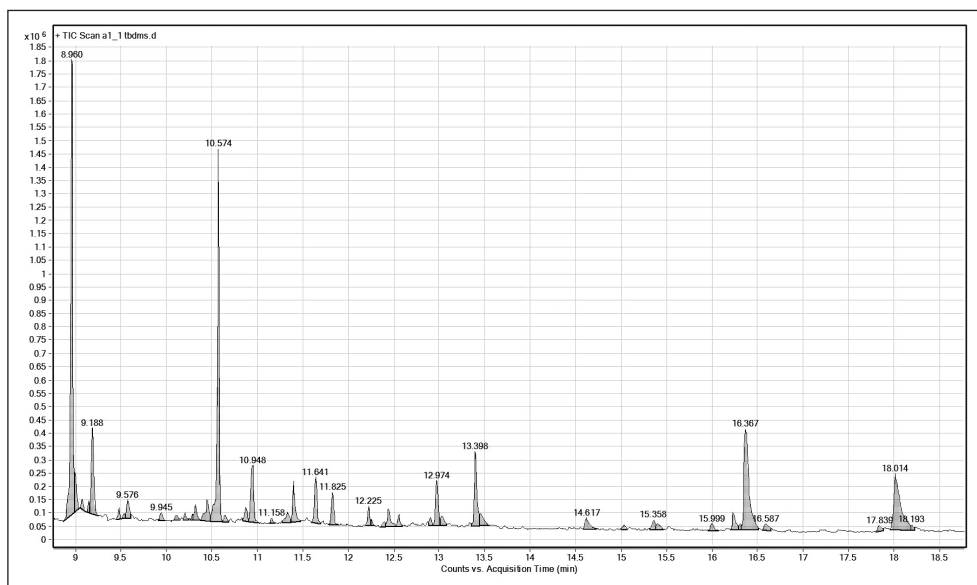
26. Kiyoshi Nakayama and Masako Ushijima, "D-malic acid production from maleic acid using microorganism: Screening of microorganism," *Biotechnology Letters* 15 (1993): 271-76; Hyohak Song and Sang Yup Lee, "Production of succinic acid by bacterial fermentation," *Enzyme and Microbial Technology* 39 (2006): 352-61.

27. Danae Fiore et al., "Chemical analyses of the earliest pigment residues from the uttermost part of the planet (Beagle Channel region, Tierra de Fuego, Southern South America)," *Journal of Archaeological Science* 35 (2008): 3047-56.

28. Pagona Maravelaki-Kalaitzaki, "Black crusts and patinas on Pentelic marble from the Parthenon and Erechtheum (Acropolis, Athens): characterization and origin," *Analytica Chimica Acta* 532 (2005): 187-98.

Compound Label	RT	Name
8.713; Acetic acid, [(tert-butyl dimethylsilyl)oxy]-, tert-butyl dimethylsilyl ester; C <sub>14</sub> H <sub>32</sub> O <sub>3</sub> Si <sub>2</sub>	8,713	Acetic acid, [(tert-butyl dimethylsilyl)oxy]-, tert-butyl dimethylsilyl ester
9.188; Bis(dimethyl-t-butylsilyl) oxalate; C <sub>14</sub> H <sub>30</sub> O <sub>4</sub> Si <sub>2</sub>	9,188	Bis(dimethyl-t-butylsilyl) oxalate
10.574; Tris(tert-butyl dimethylsilyl) borate; C <sub>18</sub> H <sub>45</sub> BO <sub>3</sub> Si <sub>3</sub>	10,574	Tris(tert-butyl dimethylsilyl) borate
11.162; Bis(dimethyl-t-butylsilyl) maleate; C <sub>16</sub> H <sub>32</sub> O <sub>4</sub> Si <sub>2</sub>	11,162	Bis(dimethyl-t-butylsilyl) maleate
11.401; Bis(dimethyl-t-butylsilyl) succinate; C <sub>16</sub> H <sub>34</sub> O <sub>4</sub> Si <sub>2</sub>	11,401	Bis(dimethyl-t-butylsilyl) succinate
12.976; Phosphoric acid, tris(tert-butyl dimethylsilyl) ester; C <sub>18</sub> H <sub>45</sub> O <sub>4</sub> PSi <sub>3</sub>	12,976	Phosphoric acid, tris(tert-butyl dimethylsilyl) ester
14.437; Heptanedioic acid, bis(tert-butyl dimethylsilyl) ester; C <sub>19</sub> H <sub>40</sub> O <sub>4</sub> Si <sub>2</sub>	14,437	Heptanedioic acid, bis(tert-butyl dimethylsilyl) ester
14.616; Tetradecanoic acid, tert-butyl dimethylsilyl ester; C <sub>20</sub> H <sub>42</sub> O <sub>2</sub> Si	14,616	Tetradecanoic acid, tert-butyl dimethylsilyl ester
15.356; Octanedioic acid, bis(tert-butyl dimethylsilyl) ester; C <sub>20</sub> H <sub>42</sub> O <sub>4</sub> Si <sub>2</sub>	15,356	Octanedioic acid, bis(tert-butyl dimethylsilyl) ester
16.235; Nonanedioic acid, bis(tert-butyl dimethylsilyl) ester; C <sub>21</sub> H <sub>44</sub> O <sub>4</sub> Si <sub>2</sub>	16,235	Nonanedioic acid, bis(tert-butyl dimethylsilyl) ester
16.366; Hexadecanoic acid, tert-butyl dimethylsilyl ester; C <sub>22</sub> H <sub>46</sub> O <sub>2</sub> Si	16,366	Hexadecanoic acid, tert-butyl dimethylsilyl ester
17.841; trans-9-Octadecenoic acid, tert-butyl dimethylsilyl ester; C <sub>24</sub> H <sub>48</sub> O <sub>2</sub> Si	17,841	trans-9-Octadecenoic acid, tert-butyl dimethylsilyl ester
18.015; Octadecanoic acid, tert-butyl dimethylsilyl ester; C <sub>24</sub> H <sub>50</sub> O <sub>2</sub> Si	18,015	Octadecanoic acid, tert-butyl dimethylsilyl ester

**Fig. 14:** Compounds detected after hydrolysis and silylation with TBDMSTFA of the paint sample. Acidic metabolite molecules. All of them belong to basic metabolism, so it is difficult to relate them to specific bacteria, algae, lichens or fungi activity.



**Fig. 15:** Sample chromatogram. Glycolic acid and oxalic acid mean more than 50% of the total integration.

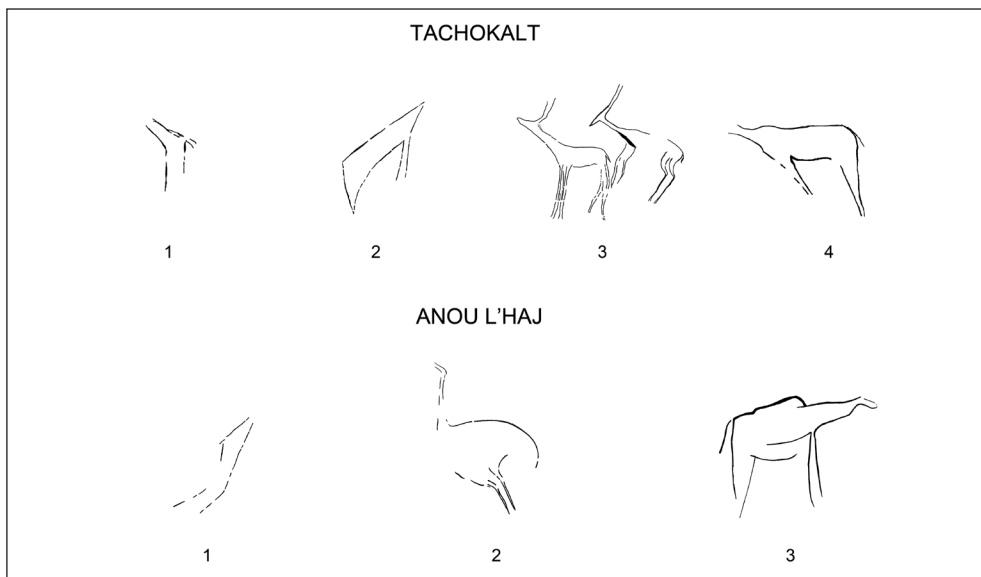
### 3.2. Archaeological considerations

The first phase figures in Aouinet Azguer are located centered at the panel or perform a main role (entrance of cavities) to the pictorial composition in shelters 1 and 12, determining the subsequent repertoire. They show elongated bodies and a volumetric structure with details that emphasize anatomical references, with a marked naturalistic tendency, although it is generally difficult to identify the species depicted, which invite us to compare them with the first and second phases of Tachokalt and Anou L'haj, in the Tamanart Valley. They also share similar execution techniques (outlined) and a distinctive stylish configuration if compared to later phases, regardless of the first ones are paintings and the other engravings, added to lack of anthropomorphic motifs. Whenever it is possible to determine a particular theme at Aouinet Azguer, it will be similar to those at Tachokalt and Anou L'haj, gazelles and antelopes, although here we find just those two species. We can survey similar specific iconographic structures between both rock art sites: Aouinet Azguer 12 antelope depiction (fig. 16 phase 1: 1) and indeterminate zoomorph from Tachokalt (fig. 17, 4); gazelle back quarters at Aouinet Azguer 1 (fig. 16, phase 1: 2) and the ones from Tachokalt (fig. 17, 3); or front part of one of the zoomorphs at Aouinet Azguer 12 (fig. 16, phase 1: 4) and the apparent antelope at Anou L'haj (fig. 17, 3).<sup>29</sup>

29. Martí Mas et al., "Prehistoric Fine-Line Rock Engravings in Tamanart (Morocco): Tachokalt and Anou L'haj," *Hesperis-Tamuda* LVI (2021): 235-71.

PHASE 1	PHASE 2	PHASE 3	PHASE 4	PHASE 5	PHASE 6	PHASE 7

**Fig. 16:** Taking into account pictorial stratigraphies at Aouinet Azguer 1, 12 and 18 (rock shelter 9 before the last surveys), defined upon techniques, styles, themes, typologies and, strongly upon overlaying and underlaying, a hypothesis defining a probable time sequence has been proposed. Aouinet Azguer 1, 12 and 18 are important examples because of quantity and characteristics of collected figures, which have been inter related in order to define technical and stylistic criteria. Just at Aouinet Azguer 1 and 12 we may place samples to first phase while Aouinet Azguer 18 is a paradigm to the final phases.



**Fig. 17:** First phases depictions at Tachokalt and Anou L'haj.

Attempts have been made to link the first engravings phases at Tamanart and south European art (Late Pleistocene – Early Holocene). In that sense, it should be highlighted the antelope at shelter number 12 of Aouinet Azguer (fig. 8). Both graphic records could be due to human groups with similar cultural structures, in touch to each other, or not.<sup>30</sup>

Since 20.000 BP the climatic conditions and palaeoenvironmental characteristics of the Lower Draa valley and North African Atlantic area were different from those of the rest of the Sahara<sup>31</sup> and would imply a possible human settlement.<sup>32</sup> The further goes research at, the more archaeological evidences of humane settlement are found.<sup>33</sup>

30. Mas et al., “Prehistoric,” 235-71.

31. Renate Heckendorf, *Bubalin und Bovidien in Südmarokko. Kontext, Klassifikation und Chronologie der Felsbilder im mittleren Draa-Tal* (Wiesbaden: Reichert Verlag, 2008); Andoni Sáenz de Buruaga, “Nuevos datos cronométricos del ambiente climático del sur del Sahara Occidental en el Holoceno antiguo y medio (ca. 12.000-5.500 cal. BP), e implicaciones en la ocupación del territorio,” *Sautuola XXI* (2016): 301-14.

32. Abdellah Salih, Jorge Onrubia-Pintado and M. Noçairi, “El Aioun-Tindouf,” in *Paléomilieux et Peuplement Préhistoriques Sahariens au Pléistocène Supérieur – Palaeoenvironments and Prehistoric Populations of the Sahara in the Upper Pleistocene*. PICG 252. Solignac, 13-15 juin 1991 (Paris: Éditions L’Harmattan, 1997), 21-36.

33. Jorge Onrubia-Pintado and Aziz Ballouche, “Les industries épipaléolithiques de Taoulekt. Les sites TR-3 et TR-15,” in *Le bassin de Tafaya (Maroc atlantique, 28° Nord). Paléoenvironnements, paléanthropologie, préhistoire*, ed. J. Riser (Paris: L’Harmattan, 1996), 153-90; Nick Barton et al., “The Late Upper Palaeolithic Occupation of the Moroccan Northwest Maghreb During the Last Glacial Maximum,” *African Archaeological Review* 22 (2005): 77-100; Nick Barton et al. (ed.), *Cemeteries and Sedentism in the Later Stone Age of NW Africa: Excavations at Grotte des Pigeons, Taforalt, Morocco*. Monographien des Römisch-Germanischen Zentralmuseums 147 (Mainz: Römisch-Germanisches Zentralmuseum, Leibniz-Forschungsinstitut für Archäologie, 2019); Abdeljalil Bouzouggar et al., “Le Paléolithique supérieur au Maroc: apport des sites du Nord-Ouest et de l’Oriental,” in *IV Simposio de Prehistoria Cueva de Nerja. La Cuenca Mediterránea durante el Paleolítico Superior. 38.000-10.000 años*, eds. J.L. Sanchidrián Torti, A.M. Márquez Alcántara and J.M. Fullola Pericot (Nerja: Fundación Cueva de Nerja, 2005), 138-50; Abdeljalil Bouzouggar et al., “Les principales découvertes archéologiques dans les séquences paléolithiques des grottes de Taforalt et du Rhafas (Maroc Oriental),” *Hespéris-Tamuda* LIV (3) (2019): 75-88; Roland Nespoulet et al., “Palaeolithic and Neolithic Occupations in the Témara Region (Rabat, Morocco): Recent Data on Hominin Contexts and Behavior,” *African Archaeological Review* 25 (2008): 21-39; Deborah I. Olszewski, Utsav A. Schurmans and Beverly A. Schmidt, “The Epipaleolithic (Iberomaurusian) from Grotte des Contrebandiers, Morocco,” *African Archaeological Review* 28 (2011): 97-123; Nick Barton and Abdeljalil Bouzouggar, “Hunter-gatherers of the Maghreb 25,000-6,000 years ago,” in *The Oxford Handbook of African Archaeology*, ed. P. Mitchell and P. Lane (Oxford: Oxford University Press, 2013), 431-43; Baraka Raïssouni et al. ed., *Carta arqueológica del Norte de Marruecos (2008-2012). Prospección y yacimientos, un primer avance*. I. Villes et sites archéologiques du Maroc V (Cádiz: Servicio de Publicaciones de la Universidad de Cádiz, 2015); Ferran Borrell et al., “Early Holocene Occupations at Ashash Rock Shelter (Zemmur, Western Sahara),” *Journal of African Archaeology* 16 (2018): 83-103; José Ramos-Muñoz et al., “The archaeological results of prehistoric societies obtained in the Archaeological Map of North Morocco Project,” *L’Anthropologie* 126 (2022): 103016; Mehdi Zouak et al., “Les occupations humaines du Nord du Maroc, du Paléolithique Moyen et du Paléolithique Supérieur. Nouvelles données sur la base des recherches archéologiques des projets de 2005 à 2020,” *L’Anthropologie* 126 (2022): 103019; Martí Mas Cornellà et al., “Proyecto Tamanart (Marruecos): Documentación de yacimientos con arte rupestre,” in *Excavaciones en el exterior 2012*, ed. C. Martín Morales, Informes y Trabajos 11 (Madrid: Ministerio de Educación, Cultura y Deporte, Subdirección General del Instituto del Patrimonio Cultural de España, 2014), 11-30.

At the same time, the documented artistic manifestations, both mobiliary art<sup>34</sup> and rock or cave paintings or engravings,<sup>35</sup> can be dated in between Later Stone Age and Early Holocene, as other publications have already pointed out for the beginning of rock art in Morocco.<sup>36</sup>

The AMS <sup>14</sup>C dating of the anthropomorph of the third phase from Aouinet Azguer 1 is consistent with the works published on neighbouring geographic areas. The third phase is made up by similar style to those from phases 2 and 4. Phases 6 and 7 would connect with the Amazigh culture,<sup>37</sup> also considering a previous schematic phase, number 5.

#### 4. Conclusions

A debate opens on the first phase of the pictorial sequence of Aouinet Azguer 1 and 12 and, although it is a challenge, we propose as a working hypothesis an older age for the paintings of this phase (Later Stone Age and/or Early Holocene), as occurs with the first two of Tachokalt and Anou L'haj (Tamanart Valley).

It is imperative to relate those sites graphic sequences (paintings and engravings) to the archaeological context of the North Africa Atlantic area, mainly on regards to the most antique depictions, so leaving apart hypothetical interpretations related to Central Sahara, where climatic and palaeoenvironmental conditions are not

---

34. Henriette Camps-Fabrer, *Matière et art mobilier dans la préhistoire nord-africaine et saharienne*. Mémoires du Centre de Recherches Anthropologiques, Préhistoriques et Ethnographiques V (Paris: Arts et Métiers Graphiques, 1966); Abdeljalil Bouzouggar et al., "Reevaluating the Age of the Iberomaurusian in Morocco," *African Archaeological Review* 25 (2008): 3-19.

35. Josef Eiwanger, "An der Nahtstelle zweier Kontinente," *Archäologie in Deutschland* 2 (2003): 14-18; Renate Heckendorf, "L'art rupestre post-paléolithique en plein air du Présahara marocain," in *Grabados rupestres de la fachada atlántica europea y africana – Rock Carvings of the European and African Atlantic Façade*, ed. R. de Balbín Behrmann et al., BAR International Series 2043 (Oxford: Archaeopress, 2009), 275-83; José Ramos-Muñoz et al., "Abrigo y cueva de Marsa. Un nuevo depósito estratigráfico con arte rupestre prehistórico en el norte de Marruecos," in *Actes du Colloque La Maurétanie & le monde méditerranéen*. Tétouan, 24-26 novembre 2016, ed. M. Ghottes et al. (Tétouan: Université Abdelmalek Es-Saâdi, Faculté des Lettres et Sciences Humaines, 2018), 172-93; Diego Salvador Fernández Sánchez et al. ed., *Tajo de las Abejeras y cueva de las Estrellas* (Castellar de la Frontera, Cádiz) (Málaga: ArdalesTur Ediciones, 2019); Hassan Aouraghe, Ramón Viñas and Abdelhadi Ewague, "Art rupestre préhistorique de la grotte du Chameau au Maroc oriental – The prehistoric rock art of Grotte du Chameau in Eastern Morocco," *International newsletter on rock art – Lettre internationale d'informations sur l'art rupestre* 90 (2021): 20-6; Hassan Aourhague et al., "Primeras evidencias de arte rupestre prehistórico en la Cueva del Camello (Zegzel, Berkane, Marruecos)," *Cuadernos de Arte Prehistórico* 11 (2021): 1-18; Gwenola Graff and Jules Masson Mourey, "'Pister' les images préhistoriques: un exemple saharien", *L'Anthropologie* 126 (2022): 103001; Zouak et al., "Les occupations," 103019.

36. Abdelkhalek Lemjidi, Hassan Aouraghe and Abderrahman Atmani, "L'art rupestre de Figuig et Ich (Maroc oriental): nouvelles données," *Journal of Materials and Environmental Sciences* 7 (2016): 3718-39; Abdelkhalek Lemjidi, "Arts rupestres marocains, un miroir esquissant des paléo-paysages maghrébins," in *La Conférence Internationale RALI 2015*. Marrakech 5-11 octobre 2015, ed. K. El Hariri and H. Chennaoui Aoudjehane (Rabat: Ministère de l'Energie, des Mines et du Développement Durable, 2018), 137-48; Lemjidi, "Arts rupestres," 60-81; Mas et al., "Prehistoric," 235-71.

37. María García Algarra, "Las inscripciones rupestres tiffinagh en Aouinet Azguer 9 (Tan Tan, Marruecos)," *Revista Otarq* 3 (2018): 7-22.

comparable. Chronologically, the third stage of Aouinet Azguer 1 would be placed already in Neolithic, according to the dating obtained, although probably still among hunter-gatherers. On the basis of the iconographic analysis, we could assert that producers would have settled down during the fifth phase, and cultural changes would have taken place at the end of the sequence with the Amazigh culture.

Previous trials of absolute dating from direct sampling, as well as those raised here, are defining and refining a rigorous methodology that can contribute to likely, much more concrete future results. Traditional studies based on themes, styles, techniques, typologies, superimpositions and infraimpositions, space or landscape..., remain to be indispensable in order to provide and contrast relevant data.

**Acknowledgements** The *Tamanart Project* has been carried out from 2011 to 2016 thanks to the funding of the Instituto del Patrimonio Cultural de España of the Dirección General de Bellas Artes y Patrimonio Cultural of the Secretaría de Estado de Cultura of the Ministerio de Educación, Cultura y Deporte of the Spanish Government. *Ayudas en régimen de concurrencia competitiva para proyectos arqueológicos en el exterior*. The *Tamanart Project* is part of the framework of the DPC/UNED agreement signed between the Direction du Patrimoine Culturel (Rabat) of the Ministère de la Culture of the Kingdom of Morocco and the Universidad Nacional de Educación a Distancia – UNED (Madrid). In 2018 and 2020-2022 fieldwork in Tamanart and Azguer is supported by the *Fundación Palarq*. We must be grateful to Dr Darden Hood (Beta Analytic Radiocarbon Dating Laboratory) for his comments as well as to Amanda Caro and Carlos Luis Pérez for the revision of the English text.

### Bibliography

- Aourhague, Hassan, Ramón Viñas and Abdelhadi Ewague. “Art rupestre préhistorique de la grotte du Chameau au Maroc oriental – The prehistoric rock art of Grotte du Chameau in Eastern Morocco.” *International Newsletter on Rock Art – Lettre internationale d’informations sur l’art rupestre* 90 (2021): 20-6.
- Aourhague, Hassan, Ramón Viñas, Abdelhadi Ewague and Aicha Oujâa. “Primeras evidencias de arte rupestre prehistórico en la Cueva del Camello (Zegzel, Berkane, Marruecos).” *Cuadernos de Arte Prehistórico* 11 (2021): 1-18.
- Barton, Nick, Abdeljalil Bouzouggar, Simon N. Collcutt and Louise T. Humphrey (eds). *Cemeteries and Sedentism in the Later Stone Age of NW Africa: Excavations at Grotte des Pigeons, Taforalt, Morocco*. Monographien des Römisch-Germanischen Zentralmuseums 147. Mainz: Römisch-Germanisches Zentralmuseum, Leibniz-Forschungsinstitut für Archäologie, 2019.
- Barton, Nick and Abdeljalil Bouzouggar. “Hunter-gatherers of the Maghreb 25,000-6,000 years ago.” In *The Oxford Handbook of African Archaeology*, eds. P. Mitchell and P. Lane, 431-43. Oxford: Oxford University Press, 2013.
- Barton, Nick, Abdeljalil Bouzouggar, Simon N. Collcutt, Rowena Gale, Thomas F.G. Higham, Louise T. Humphrey, Simon Parfitt, Edwards Rhodes, Chris B. Stringer and F. Malek. “The Late Upper Palaeolithic Occupation of the Moroccan Northwest Maghreb During the Last Glacial Maximum.” *African Archaeological Review* 22 (2005): 77-100.
- Borrell, Ferran, G. Alcalde, Ignacio Clemente, Maria Saña, Joaquim Soler and Narcís Soler. “Early Holocene Occupations at Ashash Rock Shelter (Zemmur, Western Sahara).” *Journal of African Archaeology* 16 (2018): 83-103.



- Bouzougar, Abdeljalil, Nick Barton, Louise Humphrey, E.H. Talbi, Lahbib Boudchiche, Johannes Krause and Jean-Jacques Hublin. "Les principales découvertes archéologiques dans les séquences paléolithiques des grottes de Taforalt et du Rhafas (Maroc Oriental)." *Hespéris-Tamuda* LIV (3) (2019): 75-88.
- Bouzougar, Abdeljalil, Nick Barton, S. Blockley, C. Bronk-Ramsey, Simon N. Collcutt, Rowena Gale, Thomas Higham, Louise Humphrey, Simon Parfitt, Elaine Turner and Steve Ward. "Reevaluating the Age of the Iberomaurusian in Morocco." *African Archaeological Review* 25 (2008): 3-19.
- Bouzougar, Abdeljalil, Nick Barton, Simon N. Collcutt, Simon Parfitt, Thomas Higham, Edwards Rhodes and Rowena Gale. "Le Paléolithique supérieur au Maroc: apport des sites du Nord-Ouest et de l'Orient." In *IV Simposio de Prehistoria Cueva de Nerja. La Cuenca Mediterránea durante el Paleolítico Superior: 38.000-10.000 años*, eds. J.L. Sanchidrián Torti, A.M. Márquez Alcántara and J.M. Fullola Pericot, 138-50. Nerja: Fundación Cueva de Nerja, 2005.
- Brunet, Jacques, Sylvie Demailly and Pierre Vidal. "Résultats de l'étude de prélèvements de peintures des abris du Tassili N'Ajjer." *Ars Praehistorica* 7-8 (1988-1989): 293-303.
- Camps-Fabrer, Henriette. *Matière et art mobilier dans la préhistoire nord-africaine et saharienne*. Mémoires du Centre de Recherches Anthropologiques, Préhistoriques et Ethnographiques V. Paris: Arts et Métiers Graphiques, 1966.
- Colombini, Maria Perla, Francesca Modugno and Erika Ribechini. "GC/MS in the Characterization of Lipids." In *Organic Mass Spectrometry in Art and Archaeology*, eds. M.P. Colombini and F. Modugno, 191-214. Wiley, 2009.
- Colombini, Maria Perla and Gwänelle Gautier. "GC/MS in the Characterization of Protein Paint Binders." In *Organic Mass Spectrometry in Art and Archaeology*, eds. M.P. Colombini and F. Modugno, 237-260. Wiley, 2009.
- Conti, Claudia, Marco Casati, Chiara Colombo, Elena Possenti, Marco Realini, G. Diego Gatta, Marco Merlini, Luigi Brambilla and Giuseppe Zerbi. "Synthesis of calcium oxalate trihydrate: New data by vibrational spectroscopy and synchrotron X-ray diffraction." *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy* 150 (2015): 721-30.
- Conti, Claudia, Giorgio Fustella, Chiara Colombo, Marco Realini and Giuseppe Zerbi. "Micro-Raman depth profiling on polished cross-sections: the mapping of oxalates used in protective treatment of carbonatic substrate." *Journal of Raman Spectroscopy* 39 (2008): 1307-8.
- Eiwanger, Josef. "An der Nahtstelle zweier Kontinente." *Archäologie in Deutschland* 2 (2003): 14-8.
- Fernández Sánchez, Diego Salvador, José Ramos Muñoz, Hipólito Collado Giraldo, Eduardo Vijande Vila and Antonio Jesús Luque Rojas (eds). *Tajo de las Abejeras y cueva de las Estrellas (Castellar de la Frontera, Cádiz)*. Málaga: ArdalesTur Ediciones, 2019.
- Fiore, Danae, Marta Maier, Sara D. Parera, Luis Abel Orquera and Ernesto Piana. "Chemical analyses of the earliest pigment residues from the uttermost part of the planet (Beagle Channel region, Tierra de Fuego, Southern South America)." *Journal of Archaeological Science* 35 (2008): 3047-56.
- Frost, Ray L. and Matt L. Weier. "Thermal treatment of whewellite – a thermal analysis and Raman spectroscopic study." *Thermochimica Acta* 409 (2004): 79-85.
- García Algarra, María. "Las inscripciones rupestres tiffinagh en Aouinet Azguer 9 (Tan Tan, Marruecos)." *Revista Otarq* 3 (2018): 7-22.
- Graff, Gwenola and Jules Masson Mourey. "'Pister' les images préhistoriques: un exemple saharien." *L'Anthropologie* 126 (2022) 103001.

- Hachid, Malika, Jean-Loïc Le Quellec, Ali Amara, Lucile Beck, Abdelkader Heddouche, Evelyne Kaltnecker, Sophia Lahlil, Souhila Merzoug, Christophe Moreau, Anita Quiles and Hélène Valladas. "Quelques résultats du projet de datation directe et indirecte de l'art rupestre saharien." In *The Signs of Which Times? Chronological and Palaeoenvironmental Issues in the Rock Art of Northern Africa. International Colloquium*. Brussels, 3-5 June, 2010, ed. D. Huyge, F. Van Noten and D. Swinne, 71-96. Brussels: Royal Academy for Overseas Sciences, 2012.
- Heckendorf, Renate. "L'art rupestre post-paléolithique en plein air du Présahara marocain." In *Grabados rupestres de la fachada atlántica europea y africana – Rock Carvings of the European and African Atlantic Façade*, eds. R. de Balbín Behrmann et al., BAR International Series 2043, 275-83. Oxford: Archaeopress, 2009.
- Heckendorf, Renate. *Bubalin und Bovidien in Südmarokko. Kontext, Klassifikation und Chronologie der Felsbilder im mittleren Draa-Tal*. Wiesbaden: Reichert Verlag, 2008.
- Heckendorf, Renate and Abdellah Salih. "Les peintures rupestres au Maroc: État des connaissances." *Beiträge zur Allgemeinen und Vergleichenden Archäologie* 19 (1999): 233-57.
- Le Quellec, Jean-Loïc. "Périodisation et chronologie des images rupestres du Sahara central." *Préhistoires Méditerranéennes* 4 (2013): 1-47.
- Lemjidi, Abdelkhalek. "Arts rupestres." In *Le Patrimoine Culturel du Maroc*, ed. A. Retnani, 60-81. Casablanca: Éditions La Croisée des Chemins – Ministère de la Culture et de la Communication, 2019.
- Lemjidi, Abdelkhalek. "Arts rupestres marocains, un miroir esquissant des paléo-paysages maghrébins." In *La Conférence Internationale RALI 2015. Marrakech 5-11 octobre 2015*, eds. K. El Hariri and H. Chennaoui Aoudjehane, 137-48. Rabat: Ministère de l'Énergie, des Mines et du Développement Durable, 2018.
- Lemjidi, Abdelkhalek, Hassan Aouraghe and Abderrahman Atmani. "L'art rupestre de Figuig et Ich (Maroc oriental): nouvelles données." *Journal of Materials and Environmental Sciences* 7 (2016): 3718-39.
- Maravelaki-Kalaitzaki, Pagona. "Black crusts and patinas on Pentelic marble from the Parthenon and Erechtheum (Acropolis, Athens): characterization and origin." *Analytica Chimica Acta* 532 (2005): 187-98.
- Mas, Martí, Alberto Jorge, Beatriz Gavilán, Mónica Solís, Enrique Parra and Pedro-Pablo Pérez. "Minateda rock shelters (Albacete) and post-palaeolithic art of the Mediterranean Basin in Spain: pigments, surfaces and patinas." *Journal of Archaeological Science* 40 (2013): 4635-47.
- Mas Cornellà, Martí, Abdelkhalek Lemjidi, Rafael Maura, Mónica Solís, Ahmed Oumouss, Guadalupe Torra, Eloísa Bernáldez, Esteban García-Viñas, El Mahfoud Asmhri, Javier Pérez, Pedro Pablo Pérez and Begoña González. "Prehistoric Fine-Line Rock Engravings in Tamanart (Morocco): Tachokalt and Anou L'haj." *Hespéris-Tamuda* LVI (2021): 235-71.
- Mas Cornellà, Martí, Abdelkhalek Lemjidi, Ahmed Oumouss, Guadalupe Torra Colell, Rafael Maura Mijares, Mónica Solís Delgado, Javier Pérez González, María García Algarra, El Mahfoud Asmhri, Naima Oulmakki, Mercedes Farjas Abadía and Beatriz Gavilán Ceballos. "Proyecto Tamanart 2015-2016." In *Excavaciones en el exterior 2015*, Informes y Trabajos 17, 88-102. Madrid: Ministerio de Cultura y Deporte, Subdirección General del Instituto del Patrimonio Cultural de España, 2019.
- Mas Cornellà, Martí, Abdelkhalek Lemjidi, Ahmed Oumouss, Guadalupe Torra Colell, Rafael Maura Mijares, Mónica Solís Delgado, Javier Pérez González, El Mahfoud Asmhri, Naima Oulmakki, Zineb Amrani, Mercedes Farjas Abadía, Beatriz Gavilán Ceballos and María García Algarra. "Proyecto Tamanart 2014-2015." In *Excavaciones*

- en el exterior 2014*, ed. C. Martín Morales, Informes y Trabajos 14, 12-27. Madrid: Ministerio de Educación, Cultura y Deporte, Subdirección General del Instituto del Patrimonio Cultural de España, 2016.
- Mas Cornellà, Martí, Abdelkhalek Lemjidi, Ahmed Oumouss, Guadalupe Torra Colell, Rafael Maura Mijares, Mónica Solís Delgado, Javier Pérez González, Alberto Jorge García, Naima Oulmakki, Zineb Amrani, El Mahfoud Asmhri, Mercedes Farjas Abadía, Sara Gálvez Horrillo, Jesús Romero Pérez and Beatriz Gavilán Ceballos. "Proyecto Tamanart 2013-2014." In *Excavaciones en el exterior 2013*, ed. C. Martín Morales, Informes y Trabajos 12, 15-34. Madrid: Ministerio de Educación, Cultura y Deporte, Subdirección General del Instituto del Patrimonio Cultural de España, 2015.
- Mas Cornellà, Martí, Abdelkhalek Lemjidi, Ahmed Oumouss, Guadalupe Torra Colell, Rafael Maura Mijares, Mónica Solís Delgado, Javier Pérez González, Pedro Pablo Pérez García, Mercedes Farjas Abadía, José María Bezares Torrón, David Jiménez Espartero and Beatriz Gavilán Ceballos. "Proyecto Tamanart (Marruecos): Documentación de yacimientos con arte rupestre." In *Excavaciones en el exterior 2012*, ed. C. Martín Morales, Informes y Trabajos 11, 11-30. Madrid: Ministerio de Educación, Cultura y Deporte, Subdirección General del Instituto del Patrimonio Cultural de España, 2014.
- Mas Cornellà, Martí, Rafael Maura Mijares, Mónica Solís Delgado and Javier Pérez González. "Reproducción digital, microfotografía estereoscópica y fotografía esférica aplicadas a la interpretación del arte rupestre prehistórico." *Cuadernos de Arte Rupestre* 6 (2013): 77-83.
- Modugno, Francesca and Erika Ribechini. "GC/MS in the Characterization of Resinous Materials." In *Organic Mass Spectrometry in Art and Archaeology*, eds. M.P. Colombini and F. Modugno, 215-36. Wiley, 2009.
- Mori, Fabrizio. *The great civilisations of the ancient Sahara. Neolithisation and the earliest evidence of anthropomorphic religions*. Roma: L'Erma di Bretschneider, 1998.
- Nakayama, Kiyoshi and Masako Ushijima. "D-malic acid production from maleic acid using microorganism: Screening of microorganism." *Biotechnology Letters* 15 (1993): 271-76.
- Nespoulet, Roland, Mohamed Abdeljalil El Hajraoui, Fethi Amani, Abdelwahed Ben Ncer, André Debénath, Abdelaziz El Idrissi, Jean-Paul Lacombe, Patrick Michel, Aïcha Oujaa and Emmanuelle Stoetzel. "Palaeolithic and Neolithic Occupations in the Témara Region (Rabat, Morocco): Recent Data on Hominin Contexts and Behavior." *African Archaeological Review* 25 (2008): 21-39.
- Olszewski, Deborah I., Utsav A. Schurmans and Beverly A. Schmidt. "The Epipaleolithic (Iberomauresian) from Grotte des Contrebandiers, Morocco." *African Archaeological Review* 28 (2011): 97-123.
- Onrubia-Pintado, Jorge and Aziz Ballouche. "Les industries épipaléolithiques de Taoulekt. Les sites TR-3 et TR-15." In *Le bassin de Tarfaya (Maroc atlantique, 28° Nord). Paléoenvironnements, paléanthropologie, préhistoire*, ed. J. Riser, 153-90. Paris: L'Harmattan, 1996.
- Ponti, Rosanna and Massimo Sinibaldi. "Direct dating of painted rock art in the Libyan Sahara." *Sahara* 16 (2005): 162-65.
- Ponti, Rosanna. "Datation de l'art rupestre préhistorique: Problèmes et premières expériences sur les peintures du Sahara libyenne." In *XIII International Congress of Prehistoric and Protohistoric Sciences. Forlì (Italia) 8-14 September 1996. Colloquia 15. The Prehistory of Africa*, eds. G. Aumassip, J. Desmond Clark and F. Mori, 71-3. Forlì: Abaco, 1996.
- Raissouni, Baraka, Darío Bernal, Abdelaziz El Khayari, José Ramos and Mehdi Zouak (eds). *Carta arqueológica del Norte de Marruecos (2008-2012). Prospección y yacimientos*,

- un primer avance*. I. Villes et sites archéologiques du Maroc V. Cádiz: Servicio de Publicaciones de la Universidad de Cádiz, 2015.
- Ramos-Muñoz, José, Mehdi Zouak, Eduardo Vijande-Vila, Juan Jesús Cantillo-Duarte, Salvador Domínguez-Bella, Antonio Barrena-Tocino, Sergio Almisas-Cruz, Ali Maate, José María Gutiérrez-López, Abdelaziz El Idrissi, Rodrigo Ortiz-Vázquez and Pablo Ramos-García. "The archaeological results of prehistoric societies obtained in the Archaeological Map of North Morocco Project." *L'Anthropologie* 126 (2022): 103016.
- Ramos Muñoz, José, Mehdi Zouak, Sergio Almisas Cruz, Antonio Barrena Tocino, Salvador Domínguez-Bella, Eduardo Vijande Vila, Juan Jesús Cantillo Duarte, Ali Maate, Pedro Cantalejo, José María Gutiérrez, José María Martínez, Julián Riquelme, María Blanca Ruiz Zapata, María José Gil García, Joaquín Rodríguez Vidal and Ignacio Clemente Conte. "Abrigo y cueva de Marsa. Un nuevo depósito estratigráfico con arte rupestre prehistórico en el norte de Marruecos." In *Actes du Colloque La Maurétanie & le monde méditerranéen*. Tétouan, 24-26 novembre 2016, ed. M. Ghottes et al., 172-193. Tétouan: Université Abdelmalek Es-Saâdi, Faculté des Lettres et Sciences Humaines, 2018.
- Rowe, Marvin W. "Dating by AMS radiocarbon analysis." In *Handbook of rock art research*, ed. D.S. Whitley, 139-66. Walnut Creek: Altamira Press, 2001.
- Ruiz, Juan Francisco, Martí Mas, Antonio Hernanz, Marvin W. Rowe, Karen L. Steelman and José María Gavira. "Premières datations radiocarbone d'encroûtements d'oxalate de l'art rupestre préhistorique espagnol – First radiocarbon dating of oxalate crusts over spanish prehistoric rock art." *International newsletter on rock art – Lettre internationale d'informations sur l'art rupestre* 46 (2006): 1-5.
- Sáenz de Buruaga, Andoni and A. Ezcurra. "Una estimación de cronología absoluta para una pintura rupestre del abrigo de Legteitira-6 (Agüenit, Sahara Occidental)." *Almogaren* 51 (2020): 57-73.
- Sáenz de Buruaga, Andoni. "Nuevos datos cronométricos del ambiente climático del sur del Sahara Occidental en el Holoceno antiguo y medio (ca. 12.000-5.500 cal. BP), e implicaciones en la ocupación del territorio." *Sautuola* XXI (2016): 301-14.
- Salih, Abdellah, Jorge Onrubia-Pintado and M. Noçairi. "El Aioun-Tindouf." In *Paléomilieus et Peuplement Préhistoriques Sahariens au Pléistocène Supérieur – Palaeoenvironments and Prehistoric Populations of the Sahara in the Upper Pleistocene*. PICG 252. Solignac, 13-15 juin 1991, 21-36. Paris: Éditions L'Harmattan, 1997.
- Searight, Suzan and G. Martinet. "Peintures rupestres d'un nouveau genre dans le Sud marocain." *Sahara* 13 (2001-2002): 115-18.
- Searight-Martinnet, Suzan. "Holocene Rock Art in Morocco: Hard Facts, Hesitant Hypotheses and Hopeful Headway." In *International Colloquium. The Signs of Which Times? Chronological and Palaeoenvironmental Issues in the Rock Art of Northern Africa*. Brussels, 3-5 June, 2010, eds. D. Huyge, F. Van Noten and D. Swinne, 97-108. Brussels: Royal Academy for Overseas Sciences, 2012.
- Sinibaldi, Massimo, S. Marchese and C. Desiderio. "Radiocarbon Dates for Prehistoric Rock Paintings at Tadrart Acacus, Libya." In *XIII International Congress of Prehistoric and Protohistoric Sciences. Forlì (Italia) 8-14 September 1996. Colloquia 15. The Prehistory of Africa*, eds. G. Aumassip, J. Desmond Clark and F. Mori, 75-83. Forlì: Abaco, 1996.
- Skounti, Ahmed, Daniela Zampetti, Naima Oulmakki, Rosanna Ponti, Alessandra Bravin, Kamal Tajeddine and Mustapha Nami. "Rock Art and Archaeology in Ifran-n-Taska (Eastern Jebel Bani, Morocco): First Results of the Moroccan-Italian Research Project." In *International Colloquium. The Signs of Which Times? Chronological and*

- Palaeoenvironmental Issues in the Rock Art of Northern Africa. Brussels, 3-5 June, 2010*, eds. D. Huyge, F. Van Noten and D. Swinne, 109-36. Brussels: Royal Academy for Overseas Sciences, 2012.
- Soler Subils, Joaquim. *Les pintures rupestres prehistòriques del Zemmur (Sahara Occidental)*. Girona: Documenta Universitaria, 2007.
- Song, Hyohak and Sang Yup Lee. "Production of succinic acid by bacterial fermentation." *Enzyme and Microbial Technology* 39 (2006): 352-361.
- Valladas, Hélène, Hélène Cachier, P. Maurice, Federico Bernaldo de Quirós, Jean Clottes, Victoria Cabrera Valdés, Paloma Uzquiano and Maurice Arnold. "Direct radiocarbon dates for prehistoric paintings at the Altamira, El Castillo and Niaux caves." *Nature* 357 (1992): 68-70.
- Zerboni, Andrea. "Rock Art in the Central Sahara (SW Libya): A Geoarchaeological and Palaeoenvironmental Perspective." In *International Colloquium. The Signs of Which Times? Chronological and Palaeoenvironmental Issues in the Rock Art of Northern Africa. Brussels, 3-5 June, 2010*, eds. D. Huyge, F. Van Noten and D. Swinne, 175-95. Brussels: Royal Academy for Overseas Sciences, 2012.
- Zouak, Mehdi, Eduardo Vijande-Vila, Juan Jesús Cantillo-Duarte, Salvador Domínguez-Bella, Ali Maate, Pedro Cantalejo Duarte, A. Moreno-Márquez, Abdelaziz El Idrissi, Ignacio Clemente-Conte, J.A. Riquelme Cantal, Brahim Ouchao, María Blanca Ruiz Zapata, María José Gil-García, Diego Fernández-Sánchez, Pablo Ramos-García, José Luis Ramírez-Amador and José Ramos-Muñoz. "Les occupations humaines du Nord du Maroc, du Paléolithique Moyen et du Paléolithique Supérieur. Nouvelles données sur la base des recherches archéologiques des projets de 2005 à 2020." *L'Anthropologie* 126 (2022) 103019.

العنوان: التأريخ النسبي والمطلق، والتسلسل الأيقونوگرافي، والأصباغ وميكروستراتيگرافيا الرسوم المتراكبة: لوحات صباغية من ملاجئ عينات أزكر الصخرية (درعة السفلى، المملكة المغربية)

ملخص: عينات أزكر-1، واحد من أغنى ملاجئ الرسوم الصباغية الصخرية بالمغرب، ويعود الفضل في ذلك لعدد الرسوم وخصائصها، ومنها إنجاز اللوحات على مدى فترات زمنية طويلة. في أزكر-1 وأزكر-12، أنجزت بعض الرسوم المكبرة بلون أحمر، ولا علاقة لتكوينها وحجمها وأسلوبها وتقنياتها ببقية الرسوم. تتوافق هذه الرسوم مع المرحلة الأولى من تاريخ الثقافات البكرة بالجنوب، وقد تم إنجازها باختيار مواقع متميزة ومحورية في مساحات اللوحات. ويصعب، في جل الحالات، استقراء هذه الأخيرة لأن إنجازها- وإن كانت قياساتها كبيرة- حاكى نزعة أسلوب قديم. وفي هذه الدراسة، كان تأريخ  $^{14}\text{C}$  AMS (قياس الطيف الكنتلي المسرع)، ممكناً على عينة من رسم لشكل آدمي باللون الأحمر، يعود للمرحلة الثالثة (3770 إلى 3650 قبل الميلاد) (تأريخ مُعبر، حد أدنى لِعمر العينة)، على أساس التقنية المعروفة بتأريخ الأكسالات. وقد استُكملت هذه النتائج باستخدام منهجية صعبة ومعقدة، تجمع بين التحليلات الفيزيائية والكيميائية وميكرو- استراتيگرافية دقيقة من أجل تفسير هذا التأريخ المطلق. وبالنظر إلى ضرورة الحفاظ على الرسوم الصخرية، وكونها أولوية الأولويات، لم نأخذ عينة صباغية إضافية واكتفت الدراسة بعينة واحدة. وتُطرح أسئلة كثيرة حول التاريخ والعلاقة مع المرحلة الثقافية التصويرية الأولى (القديمة) التي تم تحديدها وفقاً لمعايير أثرية تقليدية، والتي نقترحها فرضية مرجحة باستقراء البيئة الأثرية في الشريط الساحلي للمحيط الأطلسي، وبالتالي اقتراح تاريخ مُرجح وهو نهاية العصر الحجري العلوي وبداية الهولوسين.

الكلمات المفتاحية: الفن الصخري، أكسالات الكالسيوم، العصر الحجري العلوي، أزكر، المغرب، الشريط الساحلي الأطلسي.

**Titre: Chronologies relatives et absolues, séquences iconographiques, pigments et micro-stratigraphies picturales: peintures rupestres d'Aouinet Azguer 1 (Bas Draa, Maroc)**

**Résumé:** Aouinet Azguer 1, est l'un des abris sous roche peints les plus représentatifs au Maroc et ce en raison de la quantité et les caractéristiques des images exposées, qui ont été réalisées sur un temps long. Dans Aouinet Azguer 1 et 12, on trouve des figures zoomorphes rouges silhouettes dont la composition, la taille, le style et la technique n'ont aucun rapport avec le reste des représentations. Ces figures correspondent à une première phase chronoculturelle précoce, elles ont été peintes en choisissant des positions privilégiées, centrales et dans la plupart des cas nous ne sommes pas en mesure de les identifier puisque, même si par leur forme volumétrique ces images semblent suivre une tendance naturaliste, leur exécution est très conventionnelle. Dans cette étude, la datation AMS  $^{14}\text{C}$  a été possible sur un anthropomorphe, de la troisième phase, également peint en rouge (3770 à 3650 Cal BC, âge minimum), sur la base d'une technique bien connue (datation des oxalates). Ces résultats ont été complétés à l'aide d'une méthodologie combinant des analyses physico-chimiques et micro-stratigraphiques. Considérant la nécessité de mettre en avant la préservation des peintures rupestres étudiées, nous n'avons pas pu obtenir une autre datation. En outre, un nombre important de questions se posent liées à la datation et à la relation culturelle avec la première phase picturale qui a été définie sur des critères archéologiques traditionnels, que nous proposons comme hypothèse principale, au moyen de l'analyse de l'environnement archéologique de l'espace littoral atlantique, proposant une date probable du Later Stone Age (LSA) à début Holocène.

**Mots-clés:** Art rupestre, oxalate de calcium, LSA, Maroc, espace littoral atlantique.