Journal of Archaeological Science 60 (2015) 84–98

Contents lists available at ScienceDirect

Journal of Archaeological Science

journal homepage: http://www.elsevier.com/locate/jas

Analysis of the red ochre of the El Mirón burial (Ramales de la Victoria, Cantabria, Spain)

Romualdo Seva Román [a, *], Cristina Biete Bañón [b], Mª Dolores Landete Ruiz [b]

[a] Archaeometry Unit, Research Technical Services, Dept. of Ecology, University of Alicante, Ctra. San Vicente s/n, E-03690, Alicante, Spain
[b] Archaeometry Unit, Research Technical Services, University of Alicante, Ctra. San Vicente s/n, E-03690, Alicante, Spain

ARTICLE INFO

Article history:
Available online 9 April 2015

Keywords:
Magdalenian burial
El Mirón Cave
Ochre
Hematite
Thin section
XRD
SEM-EDX
XRF
Raman spectroscopy
ICP-MS
Idiomorphic hematite

ABSTRACT

This article analyzes the ochre associated with the human burial of Magdalenian age in El Mirón Cave that, with its unique features (deep red color, brightness and particle size distribution), is clearly differentiated from ochres in other strata of the site.

The most common techniques in archaeological pigment characterization studies were used: binocular microscope inspection, thin sections, granulometry, X-ray diffraction (XRD), scanning electron microscopy (SEM-EDX), X-ray fluorescence (XRF), Raman spectroscopy and inductively coupled plasma mass spectrometry (ICP-MS).

The results obtained permit the characterization of special ochre in burial layer (hematite with idiomorphic crystallinity). Its origin is completely different from the samples from outcrops inside El Mirón Cave or obtained by prospecting in Carranza Valley. This type of hematite has been identified on the coast, in Santoña, about 26 km from the burial. Given its uniqueness, can be associated with some kind of ritual of the time whose roots lay in the Middle Palaeolithic and continued throughout the rest of Prehistory.

1. Introduction

The study of prehistoric ochre has mainly been focused on the analysis of raw materials and their uses (Eiselt et al., 2011; Marshall et al., 2005; Mooney et al., 2003; Popelka-Filcoff et al., 2007, 2008) (including for cave paintings [Hernanz et al., 2010; Iriarte et al., 2009], both by modern humans and by earlier species, including European Neandertals (Zilhão, 2001).

Numerous methods have been successfully tested to determine the nature and the provenance of the raw materials, such as scanning electron microscopy coupled with energy dispersive X-ray spectrometry (SEM-EDX), proton induced X-ray emission (PIXE), X-ray diffraction (XRD), FTIR and Raman spectrometry, X-Ray fluorescence (XRF), inductively coupled plasma mass spectrometry (ICP-MS), or instrumental neutron activation analysis (INAA) (see Beck et al., 2011; Bikiaris et al., 1999; David et al., 1993; d'Errico et al., , 2010; Eiselt et al., 2011; Jercher et al., 1998; Macdonald et al., 2013; Popelka-Filcoff et al., 2007, 2008; Scadding et al., 2015; Smith and Fankhauser, 2009).

The use of ochre in rituals is well documented in South Africa 75 kya (Dayet et al., 2013; Henshilwood et al., 2001, 2009; Rifkin, 2012), Mediterranean Levant and Europe since 60–40 kya, during the late Middle and early Upper Paleolithic (d'Errico et al., 1998, 2010; Hovers et al., 2003; Roebroeks et al., 2012; Zilhão et al., 2010), with a significant increase during the Gravettian (ca. 30–20 uncal. kya), particularly in burials, from Portugal and Wales to Russia.

Among the many strata excavated in the extensively studied El Mirón Cave (e.g., González-Morales and Straus, 2000, 2009; Straus et al., 2002; Straus and González-Morales, 2003, 2012a, 2012b), is Level 504, which contained the human burial preliminarily described by Straus et al. (2011) and studied in detail in this special issue of *JAS*. This level is composed essentially of hematite and iron oxides mixed with silt, limestone gravel, organic matter such as charcoal, lithic artifacts and bones. Its bright, dark red color distinguishes it from all other strata in the cave.

* Corresponding author. Tel.: +34 610488927.
E-mail addresses: romualdo.seva@ua.es (R.S. Román), cristina.biete@ua.es (C.B. Bañón), md.landete@ua.es (M.D. Landete Ruiz).

http://dx.doi.org/10.1016/j.jas.2015.03.033
0305-4403/

The sediments used to cover the burial were mixed with hematite specially extracted from particular outcrops, suggesting deep ritual connotations. It is noteworthy that (although degraded) the color of the pigment staining the eastern face of the large engraved block adjacent to the burial matches that of the ochre in the sediments of the grave. Although this pigment was not analyzed in this study, this will be done in the next phase of research in order to understand its possible relationship to the rest of the ochre pigments from El Mirón cave.

This article concludes that special ochre was used for covering the body, defined by its unique features and suggests its possible origin, differentiating it, from other ochres found in Magdalenian levels of El Mirón, where they seem to have had other (non-funerary) uses.

2. Material and methods

The materials anayzed are 24 ochres collected in the stratigraphy of various Magdalenian occupations layers in other parts of the cave vestibule and 4 samples from stratigraphic units 503, 504, 505 and 506 in the burial area of square X7 (Table 1; Figs. 1–6).

Other ochres from known ferrous outcrops that we sampled in the Upper and Middle Carranza River valley (provinces of Vizcaya and Cantabria, approximately 10–20 km from El Mirón) were also analyzed.

Samples 1–12 are small pieces of ochre selected from sediments during excavation, in different squares: J2, J4 (level 17); T9 (level 116), U9 (level 119 and 119.2), U10 (level 119 and 119.2), V10 (level 120) and X7 (level 501 and 504) -Figs. 1 and 2. All ochres chronologically fit between the Initial and Lower Magdalenian period. The total sample weight is about 1 g. Some samples are from the same level and square: samples 6A, 6B and 6C (U10 square – level 119), and 7A and 7B (U9 square – level 119.2).

Table 1
Description of analyzed samples.

	Sample	Level	Square
Ochres	1	17	J4
	2	17	J2
	3	116	T9
	4	119	U9
	5	119	U9
	6A	119	U10
	6B		
	6C		
	7A	119.2	U9
	7B		
	8	119.2	U10
	9	120	V10
	10	501	X7
	11 (504A)	504	X7
	12 (504B)	504	X7
	1M	Lower and Middle	
	2M	Magdalenian	
	3M		
	4M		
	5M		
	6M		
	7M		
	8M		
	9M		
	1P	Prospection of the	
	2P	headwaters Carranza River	
	3P		
	4P		
	5P		
	6P		
	7P		
Sediments	503 sed	503	X7
	504 sed	504	X7
	505 sed	505	X7
	506 sed	506	X7

Samples 1M–9M belong to the V9 square (Initial and Lower Magdalenian). They were selected by their nature and different colors as shown in Fig. 6 for example, 2M and 3M samples were selected for their metallic luster. The total sample weight is about 1 g.

Samples 11 and 12 correspond to the burial deposit, Level 504 (clearly differentiated from levels 503 and 505, as seen in Fig. 5). All of Level 504 displayed many, very small bright fragments, which later were identified as hematite (Image 12, Fig. 6). It also contained visually reddish and brown – violet ochre fragments and powder (Image 10, 11 and 12, Fig. 6).

Sediment samples (about 100 g) were also selected to analyze the burial stratigraphic context.

The ochre samples were first dried at 100 °C in an oven overnight, to remove moisture. These samples were used to thin sections, binocular microscope inspection and microscopy studies.

The dried samples were ground into powder using an agate mortar and pestle, and powder samples were analyzed by inductively coupled mass spectrometry, Raman spectroscopy, X-ray diffraction and X-ray fluorescence spectrometry.

We employed the following analytical methods and instrumentation:

A Nikon SMZ 645 binocular microscope (with 8–50 magnification), was used to observe the morphological structure of the ochres.

Mineralogical analysis of ochre in thin sections was performed using a Nikon OPTIPHOT 2 – POL polarized light microscope. Images were captured by a Color View 12 camera with a Soft Imaging System.

Scanning electron microscopy (SEM) was applied in order to obtain the elemental composition of the samples in terms of percentages of the main chemical elements by means of an energy-dispersive X-ray microanalysis probe (EDX). We used an Hitachi S-3000N device, equipped with a secondary electron detector (photomultiplier type e 3.5 nm resolution), a semiconductor backscattered electron detector (5 nm resolution), as well as an X-ray detector that was able to detect elements from carbon to uranium. We also studied the structural morphology of the ochre. The scanning microscope is able to work in variable pressure mode for observation of nonconductive specimens without coating with any conductive material. Different samples were chosen from level 504: reddish ochre, fragments rich in hematite (small and bright), and sediment. Also a sample from the Carranza River prospection was analyzed.

The composition of the samples was confirmed with X-ray fluorescence spectrometry (XRF), using a PW2400 Philips MagiX PRO instrument with SuperQ-IQ + software.

Mineral composition of the samples was measured on a Bruker D8 Advance X-ray Diffractometer, with CuKα radiation (40 kV, 40 mA). Each sample was scanned over the 2-theta range 4–85° with a step size of 0.05° and a count time of 3 s per step. The obtained X-ray diffractograms were interpreted using the DIFFRAC plus Basic, Evaluation Release 2009 software.

Raman spectra were recorded using a LabRAM Jobin-Yvon HORIBA Raman dispersive spectrometer, with a laser emitting at 633 nm. The power radiation measured under the Nikon ×50 microscope objective was about 0.4 mW. The spectra were recorded as a sum of two 100 s scans. The calibration of the spectrometer was done with a Si standard (main band: 520,5 cm^{-1}).