

Ochre use during the Upper Palaeolithic: a continuous record from Finca Doña Martina and Abrigo de la Boja rock-shelters, Mula, Murcia, Spain

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ABSTRACT

The use of colour is a common feature in Upper Palaeolithic sites. However, the analysis of ochre assemblages recovered in residential contexts is rarely the object of systematic analyses. Here we analyse two ochre assemblages, recovered in the Upper Palaeolithic levels of two sites, Abrigo de la Boja and Finca Doña Martina, located in Mula, Spain: 407 and 35 ochre pieces respectively, spanning the entire Upper Palaeolithic. We combined a technological analysis with an elemental and mineralogical characterisation of the archaeological ochre and natural ochre pieces collected in nearby outcrops. Our findings suggest that the inhabitants of the sites collected ochre of different compositions, from different proveniences. They consistently processed ochre using the same techniques throughout, probably to produce large quantities of powder and for a variety of purposes of a symbolic or functional nature.

1. Introduction

Systematic use of colouring materials, such as iron-rich rocks, also referred to as ochre (Cornell and Schwertmann, 2003; Popelka-Filcoff et al., 2008; Popelka-Filcoff and Zipkin, 2022; Rifkin, 2012; Triat, 2010; Watts, 2017), is a frequently found feature in Palaeolithic sites and is often regarded as evidence for complex behaviour (d'Errico and Stringer, 2011; Dapschauskas et al., 2022; McBrearty and Brooks, 2000; Rosso, 2022). In the past few years, several studies of Upper Palaeolithic contexts have focused on the geochemical characterisation of ochre related to cave art (Garate et al., 2004; Gay et al., 2016; Iriarte et al., 2009; Pomiès et al., 2000; Vignaud et al., 2006) and, more rarely, the analysis of colouring matter residues in portable art (Broglio, 1998; Buisson et al., 1989; Dalmeri et al., 2005; Roldán et al., 2013; Rosso et al., 2022; Wolf et al., 2018). However, comprehensive studies encompassing other functional or symbolic uses of ochre, aiming to

reconstruct the technical steps involved in the acquisition, production, and use of this material are rare (Arribas Pastor et al., 2021; Beck et al., 2011; Cavallo et al., 2017; Gialanella et al., 2011; Pradeau et al., 2014; San Juan, 1991; Velliky et al., 2018, 2019, 2021). Assemblages of ochre pieces recovered in residential Upper Palaeolithic sites, as opposed to cave art sites, have in fact rarely been the primary focus of systematic studies. In addition, very few studies analyse possible evolutionary trends in the use of this material. In this study, we present the first results of the technological and chemical analysis of ochre pieces found at Finca Doña Martina and La Boja rock-shelters, Mula, Spain. These two sites, featuring long stratigraphic sequences ranging from the Mousterian to the Epimagdalenian, represent a perfect setting to understand ochre use for activities that took place in a context apparently unrelated to cave art, and to explore diachronic changes and evolutionary trends in the use of this material.

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2. Archaeological setting

Finca Doña Martina (FDM, 38°04'43.21"N, 01°29'25.13"W) and Abrigo de la Boja (ADB, 38°04'43.37"N, 1°29'23.17"W) are two rock-shelters located 50m apart in the incised gorge forming the middle part of the Rambla Perea watercourse (Supplementary Information SI1), the main left tributary of the River Mula (Angelucci et al., 2018; Zilhão, 2022; Zilhão et al., 2017). Detailed information on the geological and geographical setting of the sites, the sites' stratigraphic sequences, dating, human occupation features, stone tool assemblages and excavation methods can be found elsewhere (Angelucci et al., 2018; Audiard et al., 2024; Zilhão et al., 2017).

Systematic excavations revealed, at both sites, stratigraphic successions where a basal Middle Palaeolithic is overlain by long Upper Palaeolithic assemblages. For a description of the sequence and dating of the stratigraphic units, see SI1 text 1. Previous studies of these sites show that, despite variations in climate and environments, the region was continuously occupied throughout the Upper Pleistocene, from at least MIS 5a onwards (Zilhão, 2022; Zilhão et al., 2016, 2017). The analysis of stone tool assemblages (Zilhão et al., 2017) suggests that both sites were mostly used in a residential manner with short, intermittent visits by small groups of people. However, a few variations were identified. At ADB, some occupations (e.g., OH20, OH4) were brief, while others (e.g., OH17, OH12a, OH10- OH11a, OH1) were more intensive or prolonged (Zilhão, 2022). At FDM, Epimagdalenian occupations revealed evidence for more frequent visits, and a more intensive use of the available space (Zilhão et al., 2017). No significant changes in site function were observed through time: use-wear evidence for domestic tasks, such as meat cutting, hide processing, and wood working, was frequently identified at both sites. However, in a few cases, such as ADB's OH4 (Early Magdalenian) and FDM's layers 8 and 7b (Aurignacian and Early Gravettian), human activities would seem to have been of a more logistical nature, with a focus on hunting-related tasks. Lateral variation between the two adjacent sites suggest that they must have functioned as a single, extensive locus of human activity (Zilhão et al., 2017). The two sites yielded possible evidence for ochre use, which is the object of this study. No art was detected in the walls of the rock-shelters. However, that nowadays no art is to be found on the walls of the rock-shelters does not mean that it did not once exist. Sedimentation at the sites proceeds mostly through the accumulation of silt, sand and clasts derived from wall degradation, wall spalling (Angelucci et al., 2018) and so, by Epi-palaeolithic times, it is to be expected that no art made in earlier periods would remain on walls that themselves remained no more.

FDM and ADB sites are characterised by a bedrock made of fine to very fine calcarenite containing fossils and fossil fragments, small rounded pebbles, and calcite crystals (Angelucci et al., 2018). Analysis of archaeological sediment from the two sites indicates that it mainly derives —except for layer 10, which yielded no colouring materials— from the physical disintegration of Upper Miocene walls by exfoliation, thermoclastism and wind erosion, and that it accumulated through gravitational processes combined with the action of low-energy run-off (see Angelucci et al., 2018 for a complete description of the main characteristics of stratigraphic successions and site formation dynamics). This indicates that the iron-rich rocks found at the sites, interpreted as colouring materials, were intentionally brought from elsewhere by the sites' inhabitants.

3. Material and methods

3.1. Material

3.1.1. Archaeological samples

Our study concerns the ochre assemblages recovered at FDM and ADB (Zilhão et al., 2017). Here we define the term "ochre" as a variety of rocks, from soil lumps to ore minerals, containing a high proportion of iron oxides, and characterised by yellow, orange, red or brown colours

or streaks (Cornell and Schwertmann, 2003; Hodgskiss, 2020; Rosso et al., 2023; Triat, 2010). These assemblages are currently kept at the Museo Ciudad de Mula, Spain. The Dirección General de Patrimonio Cultural (Consejería de Presidencia, Turismo, Cultura y Deportes, Región de Murcia) issued the permits to analyse the material and temporarily export ochre pieces (permit number and issue date: SME 132/2022, June 14, 2022).

All colouring material pieces from ADB and FDM were observed macroscopically. Among those, 22 representative pieces of the collection were selected (Table 1, SI3 Table 1): 16 from ADB (Fig. 1a) and 6 from FDM (Fig. 1b) for an exhaustive chemical-mineralogical study. This selection was made based on the macroscopic analysis of the pieces (raw material type, taking into account the range of colours and textures that characterise the collection), and on their cultural attribution. Given the scarcity of ochre in Epimagdalenian layers, we have not selected pieces from those layers for chemical analysis.

3.1.2. Geological samples

A geological survey in the vicinity of the two archaeological sites and the surrounding region was carried out (Supplementary Information SI2), targeting areas where the presence of iron oxides was deemed likely (Ruiz Montes, 2000). Geological samples were collected from natural sources with the aim of determining whether they could have provided the colouring material found at both sites. In total, samples from three locations were collected (Fig. 1c, Table 1, SI3 Table 1).

1. Rambla Perea valley. Rambla Perea is a deep gorge located in the municipality of Mula along which runs a tributary of the Mula river. About 15 km separate the spring, at Fuente Caputa, from the mouth at Baños de Mula; Casa Perea lies in the middle part of the gorge. Several rock units with ages ranging from the Upper Cretaceous to the Quaternary and belonging to two different geological contexts are cut by the gorge: the Maláguide complex and the Mula and Fortuna Neogene basin. The collection of samples was restricted to the middle part of the watercourse, where the two archaeological sites are located. In this part, the cliffs are modelled into an Upper Miocene calcarenite rock formation with intercalations of sandstones and conglomerates, while the valley bottom cuts deformed Oligocene sandstone and marls. Selective erosion and karst dynamics led to the formation of the two rock-shelters (Angelucci et al., 2018). The survey at this location was carried out to verify whether potentially usable iron-rich materials existed in the Rambla Perea itself, either as inclusions or veins in the biocalcarene formation, or as pebbles transported by the watercourse or brought to the valley floor by gravitational dynamics. Samples from Rambla Perea valley are RP-J and RP-K.
2. Cerro de la Almagra. This small hill is located in Baños de Mula, 7 km south-west of the sites. The coloured travertines that outcrop there, which historically were the object of quarrying activities, are part of the so-called Neogene-Quaternary Archena – Mula basin (Loiseau et al., 1990). It is thought that these formations could be the result of bacterial activity linked to the area's present hydrothermalism (García del Cura et al., 2014, 2017). Broadly speaking, they are characterized by microcrystalline calcite associated with the presence of iron and manganese oxo-hydroxides, barite (BaSO₄), and celestite (SrSO₄). Samples from Cerro de la Almagra are ACT-F, ACM-G, ACM-H and ACM-I.
3. Mina María. It is an abandoned and flooded mining pit located in the Coto Minero de Cehégín, situated in the homonymous town 17 km north-west of the Rambla Perea sites. The iron-rich mineralisations appear within outcropping Triassic limestones, adopting a sub-concordant and concordant tabular morphology. There are several morphogenetic varieties, all of them related to the basic intratriassic volcanism of the area (ophitic masses). Magnetite (Fe₃O₄) is the major mineral phase and is partially transformed into hematite; ilmenite (FeTiO₃), chalcocopyrite (CuFeS₂) and pyrite (FeS₂)

Table 1

Description of analysed pieces. Modif: modifications; arch: archaeological pieces; geol: geological samples; FG: fine-grained; SPEC: specularite; HFG: hard fine-grained; CG: coarse-grained, FL: flaking marks, SM: smoothed areas.

Artefact category	Site	Inventory #	Analysis code	Cultural attribution	Macroscopic data			Figs
					Colour	Ochre type	Modif	
Arch	ADB	ADB 2010-1227	ADB-A	Upper Magdalenian	Orange	FG	no	Fig. 1a-A
Arch	ADB	ADB 2009-585	ADB-B	Upper Magdalenian	Red	FG	no	Fig. 1a-B
Arch	ADB	ADB 2010-1099	ADB-C	Upper Magdalenian	Red	FG	no	Fig. 1a-C
Arch	ADB	ADB 2014-1227	ADB-D	Middle Solutrean	Yellow	FG	no	Fig. 1a-D
Arch	ADB	ADB 2009-566	ADB-E	Upper Magdalenian	Yellow	FG	no	Fig. 1a-E
Arch	ADB	ADB 2009-631	ADB-F	Upper Magdalenian	Red	CG	FL	Fig. 1a-F
Arch	ADB	ADB 2014-1527	ADB-G	Lower Solutrean	Red	FG	no	Fig. 1a-G
Arch	ADB	ADB 2014-1172	ADB-H	Upper Solutrean	Grey	SPEC	no	Fig. 1a-H
Arch	ADB	ADB 2010-1163	ADB-I	Lower Solutrean	Red	FG	no	Fig. 1a-I
Arch	ADB	ADB 2014-3040	ADB-J	Late Aurignacian	Red	FG	FL	Fig. 1a-J
Arch	ADB	ADB 2014-3089	ADB-K	Late Aurignacian	Red	FG	no	Fig. 1a-K
Arch	ADB	ADB 2014-2786	ADB-L	Late Gravettian	Red	FG	SM	Fig. 1a-L
Arch	ADB	ADB 2014-3222	ADB-M	Evolved Aurignacian	Grey	SPEC	no	Fig. 1a-M
Arch	ADB	ADB 2014-2818	ADB-N	Early Gravettian	Red	FG	FL	Fig. 1a-N
Arch	ADB	ADB-2012-207	ADB-O	Early Gravettian	Yellow	FG	SM	Fig. 1a-O
Arch	ADB	ADB-2013-44	ADB-P	Evolved Aurignacian	Grey	HFG	FL	Fig. 1a-P
Arch	FDM	FDM-M5-31-A6	FDM-A	Upper Solutrean	Red	FG	no	Fig. 1b-A
Arch	FDM	FDM-M6-8-A6	FDM-B	Upper Solutrean	Red	FG	no	Fig. 1b-B
Arch	FDM	FDM-I6-39-A9	FDM-C	Middle Gravettian	Purple	FG	no	Fig. 1b-C
Arch	FDM	FDM-I5-29-A9	FDM-D	Middle Gravettian	Yellow	FG	no	Fig. 1b-D
Arch	FDM	FDM-J6-51-A8	FDM-E	Middle Gravettian	Grey	SPEC	no	Fig. 1b-E
Arch	FDM	FDM-J6-55-A10	FDM-F	Middle Gravettian	Orange	FG	no	Fig. 1b-F
Geol	Mina María	MM-A	MM-A	NA	Red	FG	–	Fig. 1c-A
Geol	Mina María	MM-B	MM-B	NA	Yellow	FG	–	Fig. 1c-B
Geol	Mina María	MM-C	MM-C	NA	Brown	FG	–	Fig. 1c-C
Geol	Mina María	MM-D	MM-D	NA	Red	FG	–	Fig. 1c-D
Geol	Mina María	MM-E	MM-E	NA	Purple	HFG	–	Fig. 1c-E
Geol	Almagra cantera	ACT-F	ACT-F	NA	Red	FG	–	Fig. 1c-F
Geol	Almagra camino	ACM-G	ACM-G	NA	Yellow	FG	–	Fig. 1c-G
Geol	Almagra camino	ACM-H	ACM-H	NA	Purple	FG	–	Fig. 1c-H
Geol	Almagra camino	ACM-I	ACM-I	NA	Red	FG	–	Fig. 1c-I
Geol	Rambla Perea	RP-J	RP-J	NA	Grey	FG	–	Fig. 1c-J
Geol	Rambla Perea	RP-K	RP-K	NA	Beige	CG	–	Fig. 1c-K

pseudomorphed by hematite (Fe_2O_3) and goethite (FeOOH) have also been documented (Metallogenetic map of Murcia). Samples from Mina María are MM-A, MM-B, MM-C, MM-D and MM-E.

3.2. Methods

3.2.1. Macroscopic and technological analysis

We recorded contextual, technological and morphometric information on all ochre pieces recovered at FDM and ADB: layer, square, quadrant, spit, layer, length, width, thickness of complete objects, weight, raw material type, colour and morphology of the piece. Raw material types, determined macroscopically, include fine-grained (FG), hard fine-grained (HFG), coarse-grained (CG), and specularite (SPEC) types (for a detailed description of each type, see SI3 text 1). Given the friable texture of the “specularite” ochre type found at ADB, these pieces were not measured, but only weighed, and single bags with multiple fragments of the same raw material type, likely from the same fractured piece, were counted as one single piece. Anthropogenic modifications (traces of flaking, striations, facets, smoothed areas, see definitions in SI3 text 2) were identified macro- and microscopically based on experiments and the literature (Hodgskiss, 2010; Rifkin, 2012; Rosso et al., 2017, 2023). All ochre types were photographed with Leica Z6 APO and Leica M165 C macroscopes.

3.2.2. Chemical analysis

Different methods can be used to characterise the composition of ochre (Dayet, 2021; Popelka-Filcoff et al., 2008; Rosso, 2022). As our aim is to conduct a first characterisation of the different raw material types present at the sites and comparing them to geological pieces, we

adopted a methodological approach that allows for mineralogical and elemental characterisation, as well as a morphological analysis of the analysed grains. To characterise the mineral composition of the ochre pieces, we used X-ray diffraction (XRD) over other methods such as micro-Raman spectroscopy (μ -RS), as XRD provides a more comprehensive identification of mineral phases rather than a single grain characterisation (Rosso et al., 2023). For elemental analysis, we used Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectroscopy (SEM-EDS). This method was selected because it allows analysing the elemental composition of specific particles, provides detailed imaging of microscopic structures (crystal aggregates), and enables the observation of component distribution though elemental mapping and assessment of the relative abundance of elements (Popelka-Filcoff and Zipkin, 2022; Rosso et al., 2023). These two methods were used to characterise the elemental and mineralogical composition of 22 archaeological ochre pieces and 11 geological samples (Table 1, SI3 Table 1).

Elemental analyses were performed at the *Centre Commun de Microscopie Appliquée (CCMA, Université Côte d'Azur)* through SEM-EDS using a Tescan Vega3 XMU scanning electron microscope (TESCAN FRANCE, Fuveau, France) equipped with an Oxford X-MaxN 50 EDX detector (Oxford Instruments, Abingdon, U.K.). Samples were observed with no prior metal or carbon coating. Most samples were observed under secondary vacuum (~ 5 – 10×10^{-2} Pa). Low vacuum mode (N_2 pressure = 20 Pa) was used when necessary. A 10 kV accelerating voltage was used for imaging using secondary and backscattered electrons, and 20 kV for EDS analyses. EDS spectra were processed with Aztec software (version 3.1, Oxford Instruments, UK).

Mineralogical analyses were carried out at the X-ray diffraction

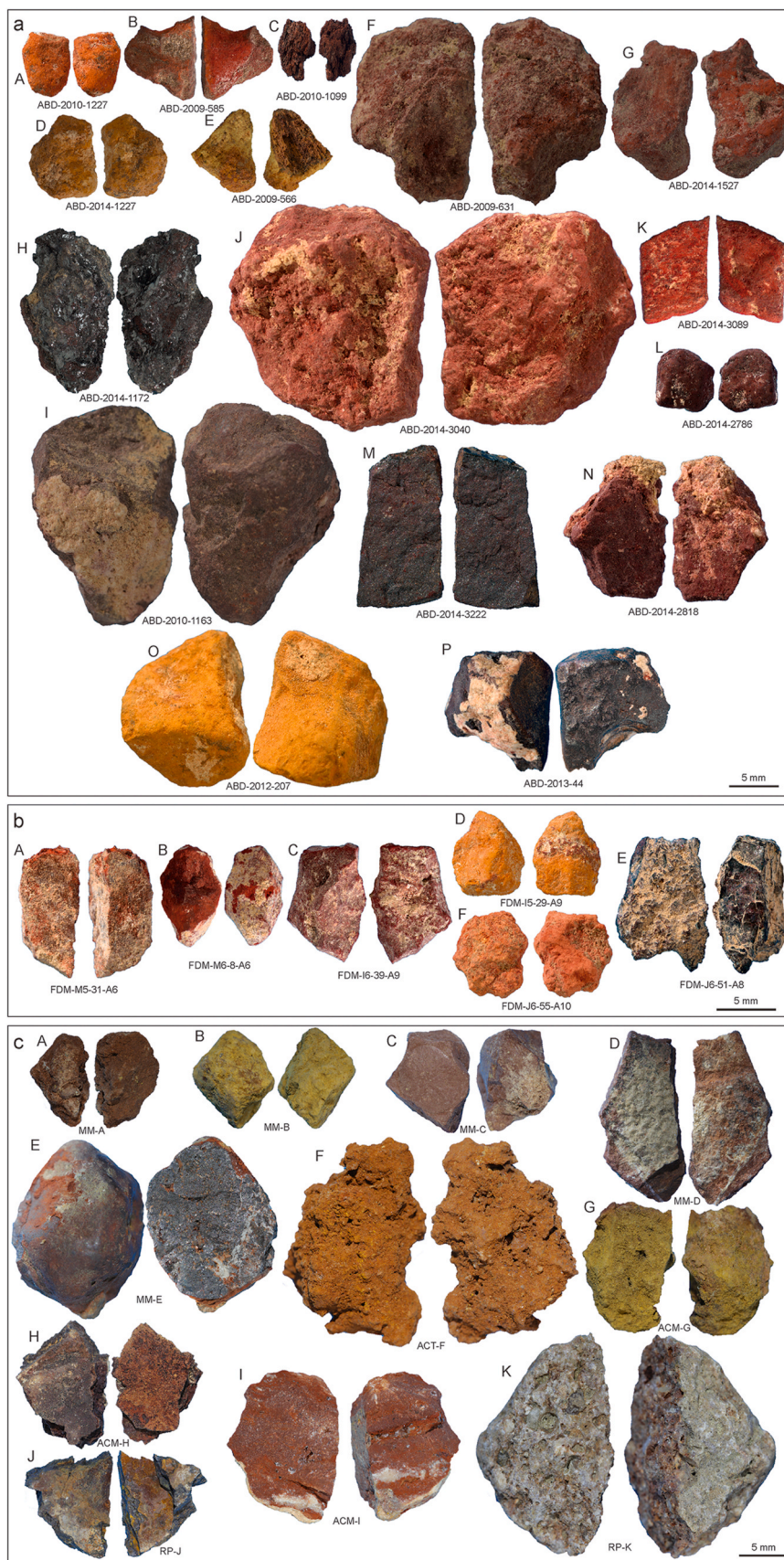


Fig. 1. Ochre pieces from Abrigo de la Boja (a), Finca Doña Martina (b) and collected geological samples (c). The letters correspond to the analysis codes given to the individual pieces listed in [Table 1](#).

facilities of the *Centres Científics i Tecnològics de la Universitat de Barcelona* after pulverization and homogenisation of the samples with an agate mortar and pestle and subsequent mounting in stainless steel sample holders (PW1811/16). Measurements were performed with a PANalytical X'Pert PRO MPD apha1 powder diffractometer in Bragg-Brentano $\theta/2\theta$ geometry and 240 mm radius. The instrument used Cu K α 1 radiation ($\lambda = 1.5406 \text{ \AA}$), with a working power of 45 kV–40 mA and the sample spinning at 2 rps. Qualitative phase analysis was carried out using the PDF (Powder Diffraction File) database, while their semi-quantification was done using the Rietveld method. To this end, all the mineral phases present in the sample were identified, the crystallographic information of each phase was collected, a model for the experimental arrangement was elaborated and, finally, the theoretical diffraction pattern was compared with the experimental one. Seriation of a presence-absence matrix of mineral phases (columns) identified by XRD was performed using PAST v. 4.17 software. The unconstrained mode was used.

4. Results

4.1. Macroscopic and technological analysis of archaeological ochre

4.1.1. Abrigo de la Boja

In total, 407 pieces of colouring materials (226g in mass) were found at ADB (Supplementary Information SI4). We excluded 66 pieces from our study: 11 were white in colour and their anthropogenic nature could not be confirmed, 7 were found in the topmost reworked Holocene layer, 12 were recovered while cleaning the sections or excavated surfaces and lack contextual data, and 36 come from a test-trench spit that cut through the boundary between the 11a and 11b Occupation Horizons (Lower Solutrean and Late Gravettian, respectively).

The ochre pieces from ADB display a variety of colours and shades (Figs. 1a–2 and 3a, SI4 Table 1). While purple, orange and brown pieces were identified, the most common colours are red, grey and yellow, accounting, respectively, for 57.2 %, 24.6 % and 12 % of the total number. A few pieces combining red and yellow (2.3 %) were also identified.

Among the identified raw material types (Figs. 1a–2 and 3b, SI4 Table 2), the FG type is the most common (69.2 %), followed by the “specularite” type (24 %). The CG type is rare (5 %) and only one piece of the HFG type was found (0.3 %).

At ADB, ochre pieces were found throughout the Upper Palaeolithic sequence (Fig. 3a and b, Supplementary Information SI4). Most pieces ($n = 176$) were found in the Solutrean (51.5 %), but the Aurignacian (13.2 %), the Gravettian (18.5 %), and the Magdalenian (14.7 %) also yielded a significant number of remains (around 40–60 pieces each).

The proportion of colours reveals interesting patterns (SI4 Table 1, Fig. 3a). Red is the most frequent colour in all levels. However, grey and yellow pieces are more frequent in the Gravettian and the Solutrean occupations, while a greater diversity of colours is found in the Magdalenian occupations.

As for raw material types, there are no significant changes across the sequence. The FG type is the most frequent in all layers (Fig. 3b, SI4 Table 2), and specularite is relatively more frequent in the Aurignacian, Gravettian and Solutrean occupations than in the Magdalenian and Epimagdalenian ones. The CG type is present in low proportions in all levels.

The fragmentation of ochre pieces is high. This is particularly the case with the “specularite” type, which is of very friable nature. Ochre pieces that could be measured (all except for the “specularite” type and the most fragmented pieces, $n = 188$) are small in size, with 183 pieces between 2 and 23 mm in length and only 5 pieces above 23 mm in length (SI4 Fig. 1, SI4 Table 3). Anthropogenic modifications are therefore difficult to detect. Only 14 pieces show possible modification marks; they come from the Aurignacian ($n = 2$), the Gravettian ($n = 4$), the Solutrean ($n = 5$) and the Magdalenian ($n = 3$). Among them, 12 are of

the FG type, and only 2 are of the CG and HFG types. Possible modifications include evidence for flaking (flake scars, flakes and fractures, Fig. 1a–f,J,N,P) ($n = 8$), possible smoothed areas ($n = 2$, Fig. 1a–L,O), and possible striations ($n = 4$). The anthropogenic nature of the latter two is, however, unclear.

4.1.2. Finca Doña Martina

At FDM, excavations yielded less evidence for ochre use than at ADB: 35 ochre pieces (69,62g in mass) (Supplementary Information SI5).

As in the case of ADB, they feature a variety of colours (Figs. 1b–4 and 3c, SI5 Table 1). Red pieces are again the most frequent (60 % of the assemblage). Yellow (11.4 %), red and yellow (8.6 %), grey (8.6 %), and orange (7 %) pieces are represented to a lesser degree. One purple and one brown piece were also detected.

Only the FG (Figs. 1b–4 and 3d, SI5 Table 2) and the “specularite” ochre (8.6 %) types were found at FDM (91.4 %). The other two ochre types identified at ADB (CG, HFG) could not be identified.

Unlike at ADB, most pieces were found in the Gravettian ($n = 21$), followed by the Solutrean ($n = 12$) and the Epimagdalenian ($n = 2$). Due to a hiatus, the Magdalenian is not represented in the FDM sequence, but the Mousterian and the Aurignacian are (Zilhão, 2022; Zilhão et al., 2017); they yielded no ochre pieces (Fig. 3c and d, Supplementary Information SI5). The variation across the Gravettian-to-Epimagdalenian sequence in the relative frequency of colours and raw material types is not significant: red ochre and the FG ochre type clearly dominate throughout (SI5 Tables 1 and 2, Fig. 3c and d). At FDM, ochre pieces are also much fragmented and small in size. The length of most pieces lies in the 1–14 mm range ($=27$); only 3 are larger, and there is no significant variation in size through time (SI5 Fig. 1, SI5 Table 3). Anthropogenic modifications are rare; no more than four, all from the Gravettian, were possibly modified (flaked).

4.2. Chemical analysis

4.2.1. Archaeological ochre

We conducted structural and elemental analyses (Tables 2 and 3 respectively, SEM images in Figs. 5 and 6, Supplementary Information SI6) on ochre samples from each cultural horizon found at ADB and FDM. For a detailed description of the chemical composition of the different raw material types at each site, see SI6 texts 1 and 2. The elemental and structural analyses reveal that the ochre pieces from ADB (Fig. 5) and FDM (Fig. 6) are characterised by significant variability in terms of morphology and composition. This is particularly the case with the red fine-grained ochre pieces, macroscopically characterised as the FG type, dominant at both sites. They exhibit agglomerates of Fe particles, present in variable proportions (SI6 Fig. 1), and typically characterised as hematite or goethite, as well as magnetite in the case of ADB's Magdalenian pieces. They usually appear as small sub-micrometric grains, and more rarely, as larger platy, acicular, or octahedral grains. Embedded in the iron-rich matrix, variable amounts of different compounds (Supplementary Information SI6), from tectosilicates and phyllosilicates, to sulphates or carbonates in different forms are also found. This high variability in composition is also observed in the CG ochre type, present at ADB in low proportions. On the other hand, the metallic-grey ochres, categorised as specularite (frequent at both sites) and the HFG type (only one piece at ADB), are characterised by very homogenous compositions and morphologies. They contain pure oxides-hydroxides, associated with small amounts of carbonates, in a foliated morphology in the case of specularite, and in the form of compact agglomerates of submicrometric grains in the case of HFG.

4.2.2. Geological ochre

The structural and elemental analyses of the geological samples (Figs. 1c and 7, Table 1, SI3 Table 1) also show a variety of mineral and elemental associations (Tables 4 and 3 respectively, SEM images in Fig. 8, detailed SEM-EDS results in SI6 Table 1). For a detailed

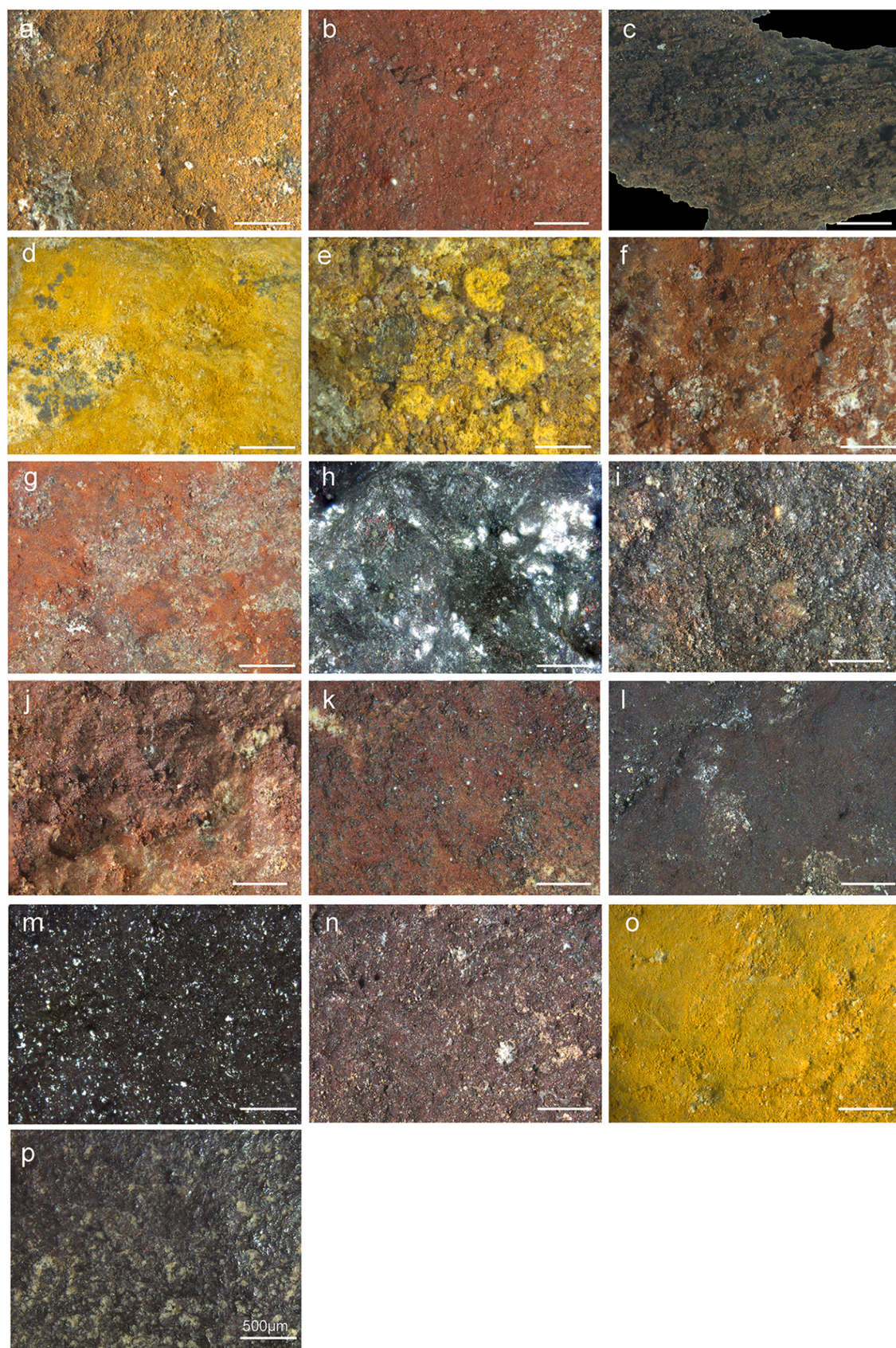


Fig. 2. Detailed view of ochre pieces from Abrigo de la Boja. The letters correspond to the analysis codes given to the individual pieces listed in [Table 1](#).

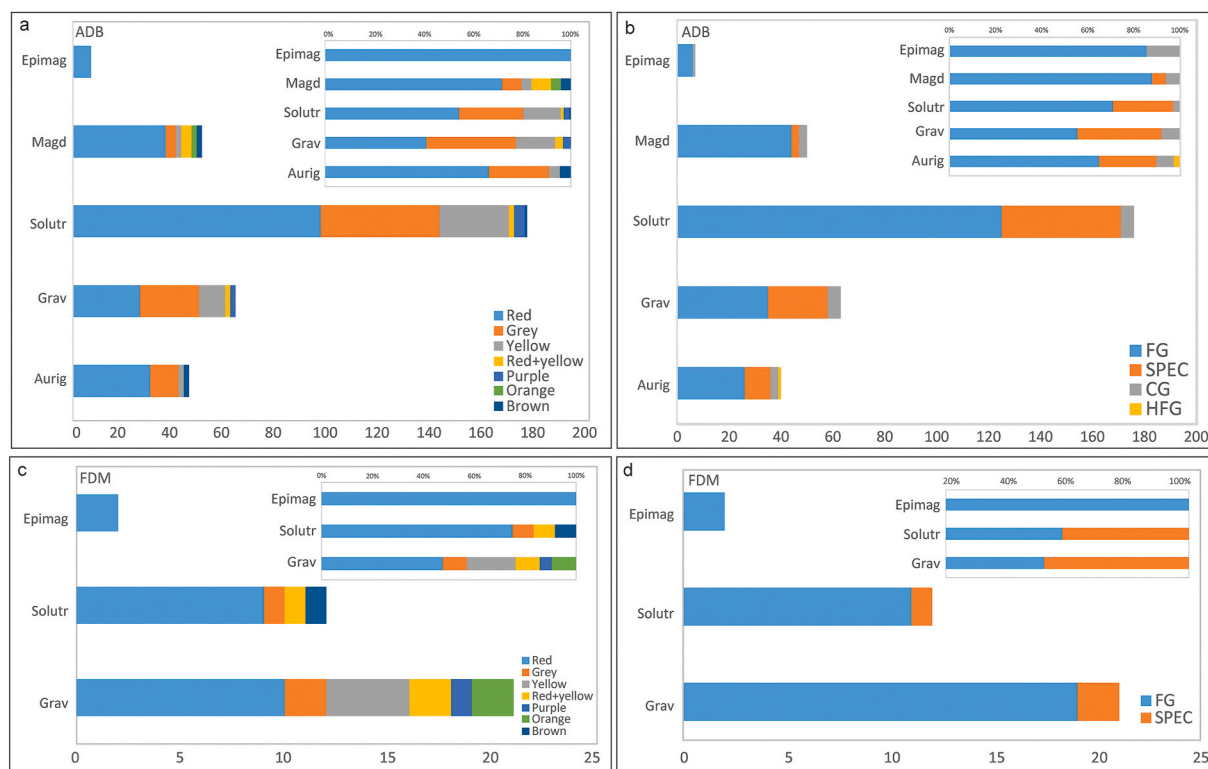


Fig. 3. Vertical distribution of ochre pieces at Abrigo de la Boja (a,b) and Finca Doña Martina (c,d) per colour (a,c) and raw material type (b,d). Y-axis values represent the number of ochre pieces by cultural phase. Epimag: Epimagdalenian; Magd: Magdalenian; Solutr: Solutrean; Grav: Gravettian; Aurig: Aurignacian, FG: fine-grained ochre type, SPEC: specularite ochre type, HFG: hard fine-grained ochre type, CG: coarse-grained ochre type. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

Table 2

Identification and semi-quantification of crystalline phases composing the archaeological samples. Results in weight percentages. *Very minor phase (not semi-quantified). Crystalline phases (according to [Whitney and Evans, 2010](#)): Ab: albite; Brt: barite; Cal: calcite; Mcal: magnesian calcite; Cry: cryptomelane; Dol: dolomite; Gth: goethite; Gp: gypsum; Hem: hematite; Ill: illite; Jrs: jarosite; Mag: magnetite; Mc: microcline; Ms: muscovite; Njrs: natrojarosite; Qz: quartz. Sample ADB-C is mostly composed by amorphous or low crystalline phases.

Code	Culture-stratigraphic unit	Silicates					Sulphates				Carbonates			Oxides-hydroxides			
		Tectosilicates			Phyllosilicates												
		Ab	Mc	Qz	Ill	Ms	Brt	Gp	Jrs	Njrs	Cal	Mcal	Dol	Cry	Gth	Hem	Mag
ADB-A	Upper Magdalenian				*						82.4					1.8	15.8
ADB-B	Upper Magdalenian			15.1		15.4										45.5	25
ADB-C	Upper Magdalenian	*	*	*							*						
ADB-E	Upper Magdalenian			1.7					18.8	27.6					44.9		
ADB-F	Upper Magdalenian										69.2					19.9	10.9
ADB-H	Upper Solutrean										5.8	1.9					92.3
ADB-D	Middle Solutrean							0.5			0.2				99.3		
ADB-G	Lower Solutrean			29.2							25	12.4				33.4	
ADB-I	Lower Solutrean										3.4		76		1.4	19.2	
ADB-N	Early Gravettian							1.6			0.4		57.5			40.5	
ADB-O	Early Gravettian			3.2	*						20.7		1.1		75		
ADB-L	Late Gravettian													7.6	3.2	89.2	
ADB-J	Late Aurignacian			22.8		1.1	2.8	1.7			52.7					19.2	
ADB-K	Late Aurignacian			0.4												99.6	
ADB-M	Evolved Aurignacian		*	2.8												97.2	
ADB-P	Evolved Aurignacian			1.7				1.2							12	85.1	
FDM-A	Upper Solutrean		0.9	32.9	*						65.6		0.6				
FDM-B	Upper Solutrean														1.9	98.1	
FDM-C	Middle Gravettian										0.1		75.9			24.1	
FDM-D	Middle Gravettian														100		
FDM-E	Middle Gravettian			3.5	*						1.2				5.1	90.2	
FDM-F	Middle Gravettian			2.9					89.5		2.3					5.3	

Table 3

Summarised results of SEM-EDS analyses conducted on ochre pieces. *Interpretation in brackets. Plat: platy; aggl: agglomerate; ang: angular; irr: irregular; acic: acicular; subcirc: subcircular; oct: octahedral; tab: tabular.

Sample	SEM observations and EDS analysis			
	Clay-sized particles (<4 µm)		Silt and sand-sized particles (>4 µm)	
	Shape	Composition*	Shape	Composition*
ADB-A	Aggl/irr	Fe (oxide/hydroxide)	Irr	Fe (oxide/hydroxide)
	Aggl	Ca (carbonate)	Irr	Si (silicate)
	–	–	Irr	Ca (carbonate)
	–	–	Plat	Si, Al, K, Fe (mica)
	–	–	Irr	Ca, S (Ca sulphate)
ADB-B	Aggl	Fe (oxide/hydroxide)	Irr	Fe, Ti (oxide/hydroxide)
	Aggl	Si, Fe, Al (clay minerals)	Irr/plat	Si (silicate)
	Aggl	Ca, S (Ca sulphate)	Plat	Fe, Si, Al (mica)
	–	–	Ang/irr	Ca (carbonate)
	–	–	Irr	Cl, Na (sodium chloride)
ADB-C	Irr	Fe (oxide/hydroxide)	Plat	Fe (oxide/hydroxide)
	–	–	Subc/irr	Si (silicate)
	–	–	Plat	Ca, Si, Mg (mica)
	–	–	Subc/irr	Ca, Mg (carbonate)
ADB-D	Irr	Fe (oxide/hydroxide)	Irr	Si, Ca (feldspar)
	Aggl	Si, Fe, Al, Mg (clay minerals)	Ang	Ca (carbonate)
	–	–	Ang	Fe, Ti (oxide/hydroxide)
ADB-E	Irr	Fe (oxide/hydroxide)	Plat	Fe (oxide/hydroxide)
	–	–	Oct	Fe, S (Fe sulphate)
	–	–	Irr	Si, Al (feldspar)
	–	–	Ang	Si (silicate)
ADB-F	Aggl/irr	Fe (oxide/hydroxide)	Irr	Fe (oxide/hydroxide)
	–	–	Irr/Subc	Si (silicate)
	–	–	Ang	Si, Al, K (feldspar)
	–	–	Ang/irr	Ca (carbonate)
ADB-G	Aggl	Fe (oxide/hydroxide)	Plat	Si, Fe, Al, K (mica)
	Aggl	Fe, Ti (oxide/hydroxide)	Irr	Ca, Mg (carbonate)
	Aggl	Si (silicate)	Irr	Ca (carbonate)
	Aggl	Si, Fe, Al (clay minerals)	–	–
ADB-H	Irr	Fe (oxide/hydroxide)	Plat	Fe (oxide/hydroxide)
	–	–	Ang	Ca (carbonate)
	–	–	Irr/plat	Ca, Mg (carbonate)
ADB-I	Aggl/plat	Fe (oxide/hydroxide)	Plat	Fe (oxide/hydroxide)
	Plat	Ti, Fe (oxide/hydroxide)	Ang	Si (silicate)
	Aggl	Si (silicate)	Ang	Ca, S (Ca sulphate)
	Aggl	Ca, Mg (carbonate)	Irr	Si, Al, Na (feldspar)
	Aggl	Ca (carbonate)	–	–
ADB-J	Aggl/irr	Fe (oxide/hydroxide)	Ang	Si (silicate)
	Aggl/ang	Si (silicate)	Plat	Ba, S (sulphate)
	Aggl	Ca (carbonate)	Acic	Sr, S, Ba (sulphate)
	Aggl	Fe, Si, Al, Ca (clay minerals)	–	–
	Aggl	Ba, S (sulphate)	–	–
ADB-K	Aggl/irr	Fe (oxide/hydroxide)	Plat	Fe (oxide/hydroxide)
	Aggl	Si (silicate)	Irr	Ti, Fe (oxide/hydroxide)
	–	–	Irr	Si (silicate)
	–	–	Plat	Fe, Si, Al (mica)
	–	–	Irr	Ca, Si (feldspar)
	–	–	Irr	Ba, S (sulphate)
	–	–	Irr	Ca (carbonate)
ADB-L	Aggl	Fe (oxide/hydroxide)	Irr	Mn, Fe (oxide/hydroxide)
	Aggl	Mn, Fe (oxide/hydroxide)	Irr	Ca (carbonate)
ADB-M	–	–	Irr	Si (silicate)
	–	–	Plat	Fe (oxide/hydroxide)
ADB-N	Aggl/irr	Fe (oxide/hydroxide)	Plat	Si (silicate)
	Irr	Ca (carbonate)	Acic	Sr, S (sulphate)
	–	–	Irr	Si, Al, Ca (feldspar)
	–	–	Irr/plat	Ca, Mg (carbonate)
	–	–	Plat	Ca, S (sulphate)
ADB-O	Aggl/irr	Fe (oxide/hydroxide)	Irr	Fe (oxide/hydroxide)
	Aggl	Fe, Si, Al (clay minerals)	Irr	Ca (carbonate)
	Irr	Si (silicate)	–	–
ADB-P	Aggl/ang	Fe (oxide/hydroxide)	Ang	Fe (oxide/hydroxide)
	Irr	Ca (carbonate)	Ang	Si (silicate)

(continued on next page)

Table 3 (continued)

Sample	SEM observations and EDS analysis			
	Clay-sized particles (<4 µm)		Silt and sand-sized particles (>4 µm)	
	Shape	Composition*	Shape	Composition*
FDM-A	Aggl/irr	Fe (oxide/hydroxide)	Irr	Mn (oxide)
	Aggl/irr	Si (silicate)	Irr	Ca (carbonate)
	Aggl	Ca (carbonate)	–	–
FDM-B	Aggl	Fe (oxide/hydroxide)	Irr	Ca (carbonate)
	Aggl	Ca (carbonate)	–	–
FDM-C	Aggl/irr	Fe (oxide/hydroxide)	Irr/plat	Si (silicate)
	Irr	Ca, Mg (carbonate)	–	–
FDM-D	Aggl/subc	Fe (oxide/hydroxide)	Plat	Si (silicate)
	Aggl/subc	Si (silicate)	Plat	Ca (carbonate)
FDM-E	–	–	Plat	Fe (oxide/hydroxide)
FDM-F	Aggl	Fe (oxide/hydroxide)	Ang	Fe (oxide/hydroxide)
	Aggl	Ba, S (Ba sulphate)	Irr/Ang	Si (silicate)
	Aggl	Si (silicate)	–	–
MM-A	Irr	Fe, Ti (oxide/hydroxide)	Irr	Ti (oxide)
	–	–	Ang/irr	Si (silicate)
	–	–	Irr	Ca, Si, Al (feldspar)
	–	–	Ang	Ca, Mg (carbonate)
	–	–	Irr	Ca (carbonate)
MM-B	Aggl/irr	Fe (oxide/hydroxide)	Tab	Fe (oxide/hydroxide)
	Irr	Ti (oxide)	Ang	Si (silicate)
	Aggl/irr	Si (silicate)	Tab	Si, K, Al (feldspar)
	Irr/tab	Ba, S (sulphate)	Ang	Ca, Mg (carbonate)
	–	–	Irr	Ca (carbonate)
MM-C	Aggl	Fe (oxide/hydroxide)	Ang	Fe (oxide/hydroxide)
	Aggl	Si (silicate)	Irr	Ca (carbonate)
MM-D	–	–	Ang	Si (silicate)
	–	–	Ang	Ca (carbonate)
	–	–	Ang/irr	Ca, Si, Al (feldspar)
	–	–	Ang	Si, Ca, Al, Na (feldspar)
MM-E	–	–	Ang	Fe (oxide/hydroxide)
	–	–	Ang/plat	Si, Al (mica)
ACT-F	Aggl/irr	Fe (oxide/hydroxide)	Irr	Fe (oxide/hydroxide)
	Aggl	Ca (carbonate)	Ang	Ca (carbonate)
ACM-G	Aggl	Fe (oxide/hydroxide)	Plat	Fe (oxide/hydroxide)
	Aggl	Si (silicate)	Irr	Si (silicate)
	Aggl/plat	Ca, Si, Fe (clay minerals)	–	–
	Irr	Ca (carbonate)	–	–
ACM-H	Irr	Fe (oxide/hydroxide)	–	–
ACM-I	Aggl	Fe (oxide/hydroxide)	Ang	Ca, Si, Al (feldspar)
	Plat	Ca, Fe, Si, Al (clay minerals)	Ang	Ca (carbonate)
RP-J	Aggl/subc	Fe (oxide/hydroxide)	Acic/irr	Fe (oxide/hydroxide)
	–	–	Ang	Ca (carbonate)
RP-K	Aggl	Fe (oxide/hydroxide)	Irr/Subc	Si (silicate)
	Aggl	Si (silicate)	Ang/irr	Ca (carbonate)
	Aggl	Si, K, Al (clay minerals)	–	–
	Irr	Si, Ca, Al (clay minerals)	–	–
	Ang	Ca (carbonate)	–	–

description of the chemical composition of geological samples, see SI6 text 3. Most ochre pieces found during our survey (all except samples MM-E and RP-K) correspond macroscopically to the FG ochre type, but show significant differences in composition and grain morphology. At Rambla Perea, goethite and magnetite-rich ochre characterised by an acicular morphology was found. At Cerro de la Almagra, ochre displays a range of mineral associations, including carbonates with iron oxide-hydroxides, more or less pure iron hydroxides, oxide-hydroxide combinations, and complex associations incorporating sulphates and silicates. At Mina María, samples are more varied in composition, containing tectosilicates, phyllosilicates, carbonates, and iron oxides in different proportions. One sample compatible with the CG ochre type

(rich in calcite and quartz) and one sample that matches the HFG type (foliated Fe-rich structure) were found at Rambla Perea and Mina María, respectively.

4.2.3. Compositional variability of archaeological and geological ochre

The variability in the mineralogical composition of the ADB and FDM ochre shows interesting patterns when conducting seriation analyses of the XRD results (Fig. 9). Hematite and calcite are the most frequent minerals at both sites, throughout both sequences. However, these two minerals seem to be more frequently associated with goethite, gypsum and dolomite in the Gravettian and the Solutrean. The pieces found in the Aurignacian, and in particular in the Magdalenian, show in general

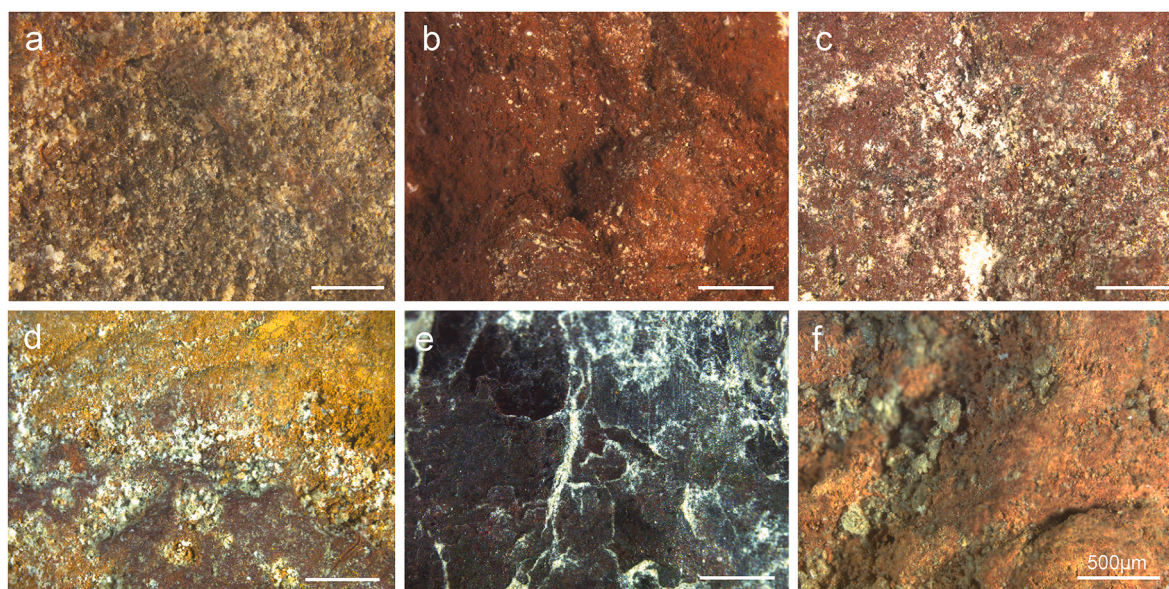


Fig. 4. Detailed view of ochre pieces from Finca Doña Martina. The letters correspond to the analysis codes given to the individual pieces listed in Table 1.

different mineral associations, of a more diverse nature.

The natural ochre pieces collected at Rambla Perea, Cerro de la Almagra, and Mina María also display a diverse range of morphologies and minerals. Seriation analyses of XRD results including the geological pieces (Fig. 10) show that Mina María samples in general differ from most of the archaeological pieces, while the mineralogical associations identified at Cerro de la Almagra match more closely the archaeological pieces.

By combining results of SEM-EDS and XRD analyses, we observe that, in some cases, archaeological pieces classified in the FG type share similarities with some of the geological samples. Among the yellow pieces, for instance, ADB-F,N,J (Gravettian and Solutrean, respectively) could potentially match some of the Cerro de la Almagra samples (ACM-G), in that they are composed of agglomerates of submicrometric particles characterised as goethite, with quartz and calcite inclusions. They show, however, differences in grain morphology. Among the red pieces, the notable presence of calcite on some of the archaeological pieces (ADB-F,N,J and FDM-A,C for instance) resembles that which is observed in the Cerro de la Almagra and Mina María samples, although the Mina María samples are more massive in texture than the archaeological ones. Also, the occurrence of magnetite in pieces containing agglomerates of submicrometric Fe particles, as in ADB-A,B (Magdalenian), was also observed in one Mina María sample and in one piece collected at Rambla Perea.

On the other hand, some of the FG archaeological pieces match none of the geological samples. This is the case, for instance, of two yellow pieces, namely, ADB-E (Magdalenian), which displays characteristic octahedral jarosite minerals, or FDM-D, which contains carbonate and silicate-rich platy particles. Among the red pieces, geological samples from Mina María appear to differ from the archaeological ones, as they are rich in phyllosilicates such as muscovite or celestite, even though two samples (ADB-B and ADB-J, Magdalenian and Aurignacian, respectively) also include muscovite.

Despite the significant occurrence of specularite at both sites, we were unable to identify this raw material type in any of the three surveyed locations. The CG and HFG types, which are rare at the sites, were also rarely found during the survey.

5. Discussion

The rock-shelters Abrigo de la Boja and Finca Doña Martina are characterised by a high degree of stratigraphic integrity and their sequences virtually span the entire Upper Palaeolithic, providing a unique record of the acquisition, transport and use of ochre within a residential setting, at a crucial period for our understanding of the development of complex cultures.

The analysis of the two ochre assemblages indicates that, throughout the Upper Palaeolithic, the inhabitants of both sites brought with them ochre collected elsewhere in the landscape. The absence of ochre in the Aurignacian of FDM may be attributed to lateral variation in the emplacement of specific activity areas relative to the fixed position of the excavation trench, or to the transient nature of the occupation. The latter may well apply to the absence of ochre in both sites' Middle Palaeolithic occupations, but note that one Mousterian sidescraper from Finca Doña Martina features ochre residues (Zilhão, 2022) and use-wear analysis of the stone tools from the nearby Middle Palaeolithic site of Cueva Antón showed that ochre was regularly used for the processing of hides (Deschamps et al., 2022).

At ADB, ochre use increases gradually from the Aurignacian to the Gravettian, peaks in the Solutrean, and gradually decreases towards the top of the sequence. In contrast, at FDM, ochre use starts and reaches its highest levels during the Gravettian and then progressively declines. Previous studies (Zilhão, 2022; Zilhão et al., 2017) have shown that some occupations at the site were brief, while others were more intense or prolonged. The Upper Solutrean at ADB featured more prolonged occupations, which is consistent with our observations. However, at FDM, Epimagdalenian layers seem to reveal a more frequent use of the site, yet there is a clear drop in the amount of ochre found in those levels. This discrepancy could suggest either a change in the locations where ochre-related activities took place, a change in the way ochre was processed that resulted in less tangible evidence, or a cultural shift leading to a gradual decline of activities related to the use of colouring materials. The latter seems the most likely explanation, as ADB also features a significant reduction in ochre use during the Epimagdalenian.

The macroscopic examination of archaeological pieces from ADB and FDM reveals a diverse array of colours, textures, and grain sizes. However, a clear preference for red ochre, composed of a high-quality fine-grained raw material (FG type), rich in hematite, and, to a lesser degree,

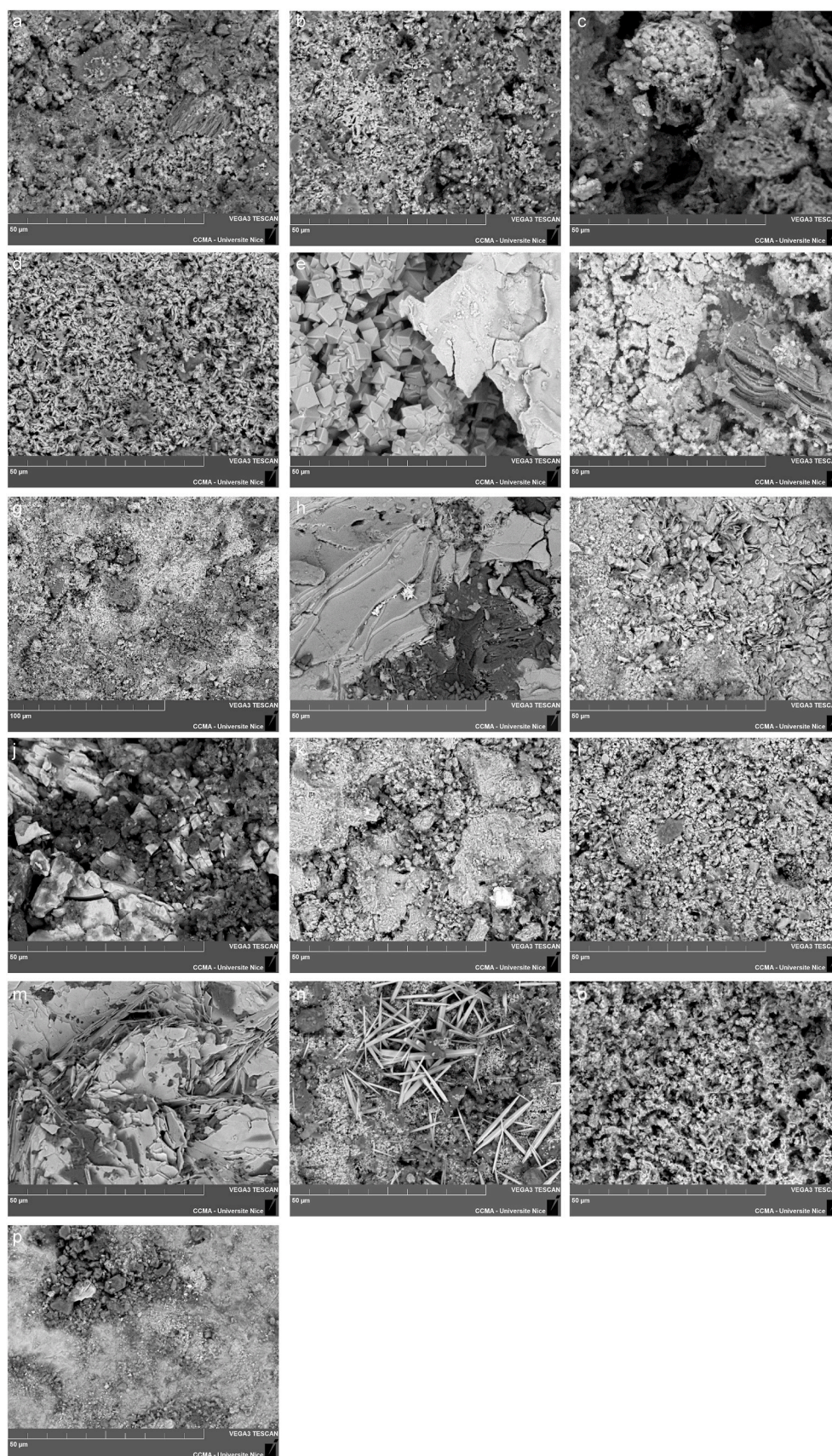


Fig. 5. SEM images of ochre pieces from ADB. All figures are in backscattered electron (BSE) mode. The letters correspond to the analysis codes given to the individual pieces listed in [Table 1](#).

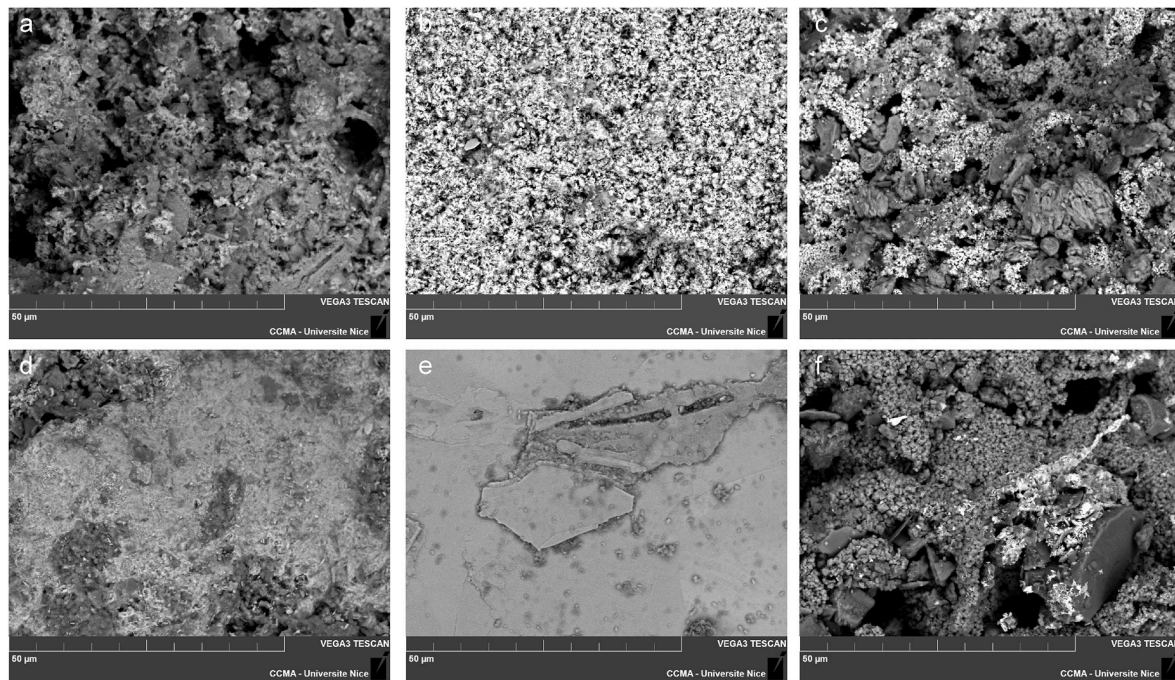


Fig. 6. SEM images of ochre pieces from FDM. All figures are in backscattered electron (BSE) mode. The letters correspond to the analysis codes given to the individual pieces listed in [Table 1](#).

specular hematite, was observed at both sites across all cultural horizons. The chemical characterisation of these raw materials further confirms their diversity, revealing various mineral and elemental associations, and suggests that ochre was likely sourced from different outcrops. Although red fine-grained ochre dominates in all layers, variations in mineralogical associations within this type of ochre were identified throughout the sequence. While ochre pieces from the Gravettian and the Solutrean are generally richer in goethite and dolomite, the pieces from the Magdalenian show a different, more varied array of minerals. This may indicate that, while people searched for ochre with similar features all through the regional Upper Palaeolithic, the sources of raw material, the access to the outcrops, or the exchange networks underwent changes through time, and may have been more diverse during the Magdalenian.

Comparison between the chemical-mineralogical data of the archaeological and natural fragments indicates that a few archaeological FG ochre pieces may potentially match natural ochre found at Cerro de la Almagra and, less probably, Mina María. However, most pieces of the FG type do not correspond in composition and grain morphology to the natural samples. Additionally, specular hematite was not detected in any of the sampled natural outcrops. Hematite is one of the most common minerals in the Region of Murcia. Its crystalline habit can be very variable, ranging from micaceous varieties (such as those found in Jumilla), to granular, massive (such as those from Mina María) and even earthy types (such as the ones samples in Cerro de la Almagra). In this region, hematite can appear associated with any genetic type of sediments and rocks, whether in continental sands and clays, in pisolitic limestones, in igneous rocks, in metamorphic rocks, in the oxidation zone of hydrothermal deposits, by alteration of sulphides and iron carbonates, etc. Therefore, the identification of the source areas would require a systematic study of different outcrops in the region. Despite this, our analysis suggests that the inhabitants of ADB and FDM did not obtain their ochre from the immediate vicinity of the sites, in the Rambla Perea itself, but likely sourced raw materials from different outcrops situated at least a few kilometres away.

Once brought to the site, how was ochre processed and used? The assemblages from ADB and FDM feature few, if any, ochre pieces with

unambiguous modification marks and are characterised by the presence of small, highly fractured pieces throughout. This seems to indicate that ochre was probably crushed to exhaustion to produce powder, leaving only a few large pieces and a considerable amount of waste from the crushing process. The fact that no variations were observed in the different cultural horizons reflects a technical system that persisted and was consistently transmitted through time, despite probable changes in raw material acquisition strategies. Transmission electron microscopy (TEM) analyses may in the future be conducted to identify microstructural changes observed on thermally induced transformation of goethite into hematite as described by [Menu and Pomiès \(1999\)](#), to identify possible evidence of ochre heating.

As for the function of ochre, crushing usually allows producing a larger quantity of ochre powder than grinding, and in a shorter period of time. The exclusive use of this technique seems to indicate the need for large quantities of ochre, which could indicate a use for functional activities such as tanning hides ([Rifkin, 2012](#)). This is consistent with evidence for hide processing identified at the sites through the analysis of use-wear on lithics ([Zilhão et al., 2017](#)).

While the rock-shelter walls (made of fine to very fine calcarenite) are suitable for the production of rock art, no direct evidence of such activity was observed. As sediment at both sites is primarily derived from wall disintegration ([Angelucci et al., 2018](#)), it is likely that any evidence of art may have disappeared over time. Although no ochre residues were detected on clasts resulting from wall degradation, taphonomic processes may account for such an absence. Therefore, we cannot exclude that the ochre was also used for the production of parietal art at these sites. On the other hand, an ornamental shell assemblage featuring ubiquitous red ochre staining has been reported from ADB's Aurignacian ([Zilhão et al., 2017](#)), which supports the interpretation of a symbolic use.

Coupled with the variety of ochre found at the site and the persistence of this cultural feature through time, this observation would seem to indicate that ochre use in the Rambla Perea sites was part of a deeply rooted tradition and was likely used for a variety of purposes, including both functional and symbolic activities.

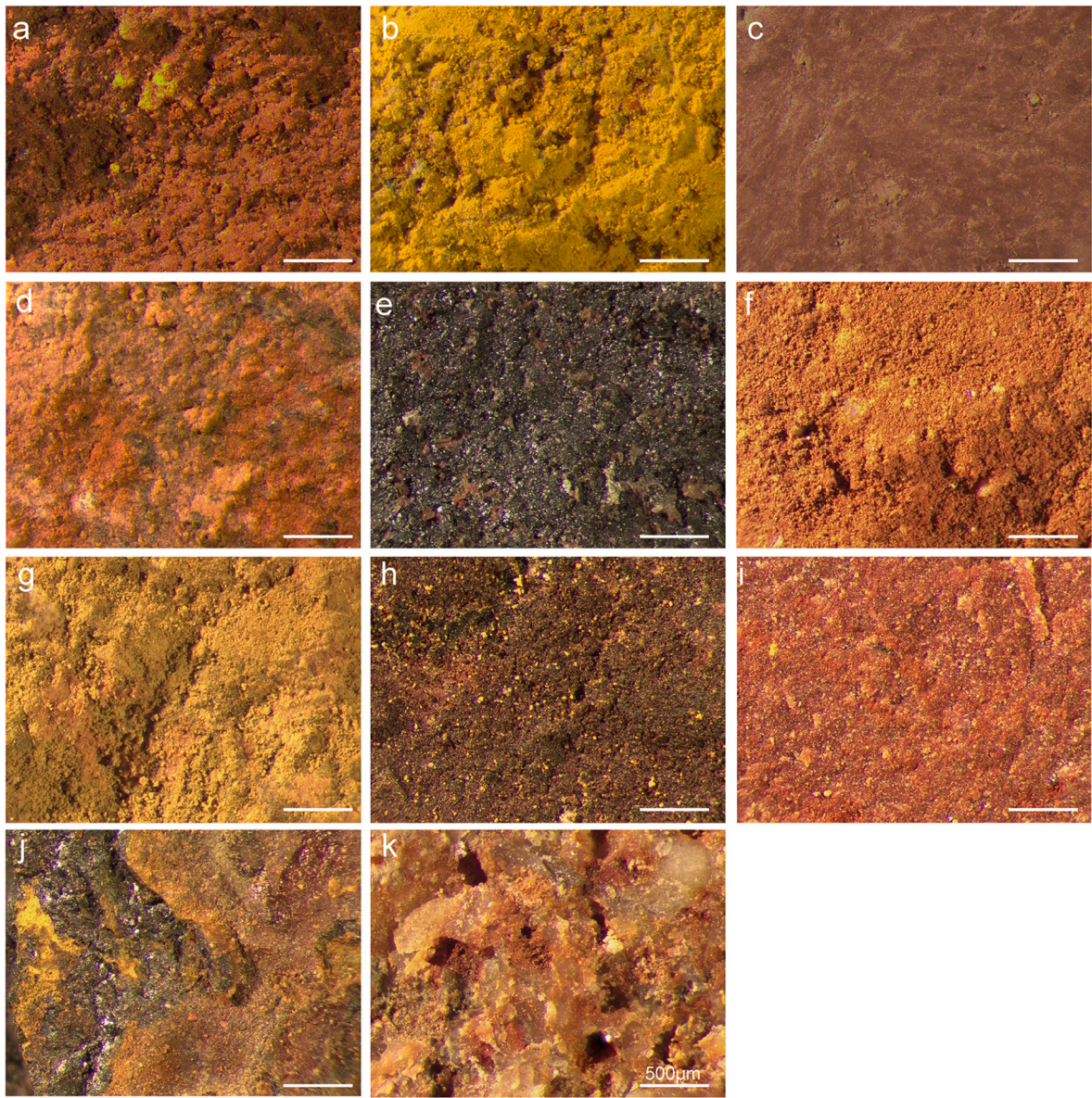


Fig. 7. Detailed view of collected geological samples The letters correspond to the analysis codes given to the individual pieces listed in Table 1.

Table 4
Identification and semi-quantification of crystalline phases composing the geological samples. Results in weight percentages. *Very minor phase (not semi-quantified). Crystalline phases (according to Whitney and Evans, 2010): Ab: albite; Cal: calcite; Clc: clinocllore; Clt: celestine; Dol: dolomite; Gth: goethite; Gp: gypsum; Hem: hematite; Mag: magnetite; Mc: microcline; Ms: muscovite; Qz: quartz; Vrm: vermiculite.

Code	Silicates						Sulphates		Carbonates		Oxides-Hydroxides		
	Tectosilicates			Phyllosilicates			Clt	Gp	Cal	Dol	Gth	Hem	Mag
	Ab	Mc	Qz	Clc	Ms	Vrm							
MM-A	1.5	1.4	6.2		51.6				35.6	0.6		3.1	
MM-B			*		*	*			*			*	
MM-C			*	*	*				*				
MM-D			5.1		8.8				81.6	1		3.5	
MM-E				5.1					23			29.6	42.2
ACT-F			1.4				2.8	55.9	35.6		33.9		
ACM-G			1.5						0.9		97.6		
ACM-H											32.8	67.2	
ACM-I			0.3						90.7		8	1	
RP-J											70.7		29.3
RP-K			5.7				0.7		92.7	0.9			

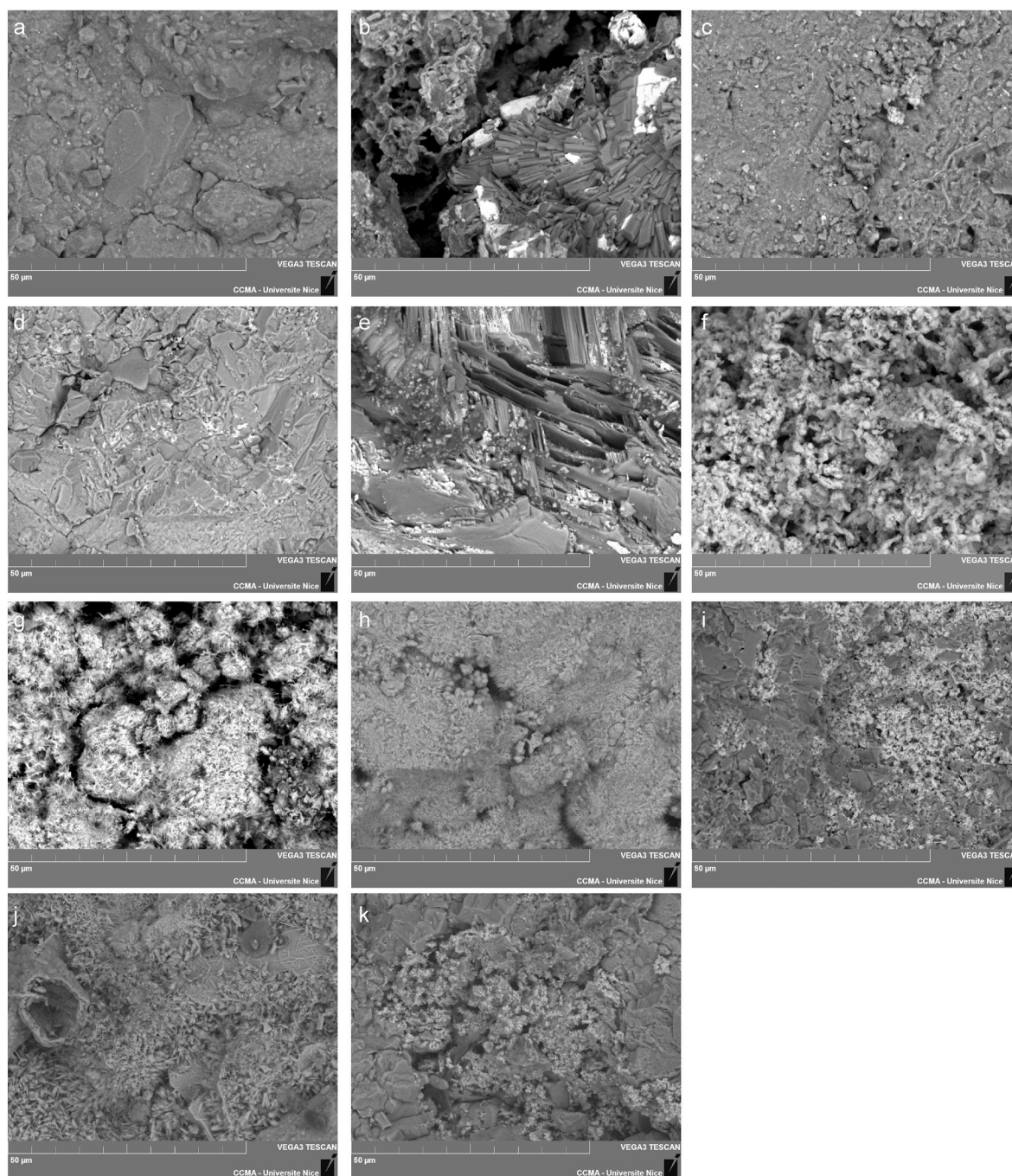


Fig. 8. SEM images of geological ochre pieces. All figures are in backscattered electron (BSE) mode. The letters correspond to the analysis codes given to the individual pieces listed in [Table 1](#).

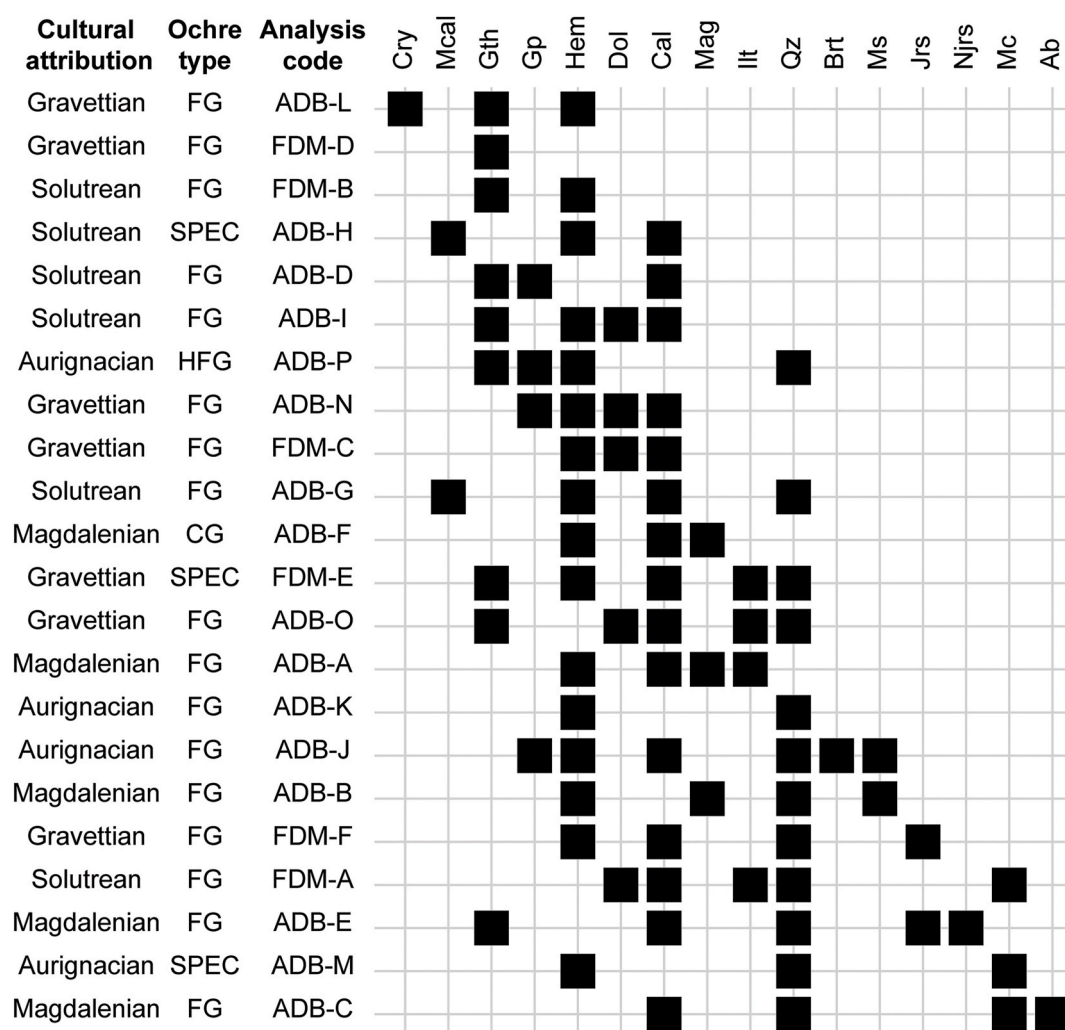


Fig. 9. Seriation analysis of mineral phases identified by XRD on colouring materials from Abrigo de la Boja and Finca Doña Martina. FG: fine-grained ochre type, SPEC: specularite ochre type, HFG: hard fine-grained ochre type, CG: coarse-grained ochre type. Crystalline phases (according to [Whitney and Evans, 2010](#)): Ab: albite; Br: barite; Cal: calcite; Mcal: magnesian calcite; Cry: cryptomelane; Dol: dolomite; Gth: goethite; Gp: gypsum; Hem: hematite; Ill: illite; Jrs: jarosite; Mag: magnetite; Mc: microcline; Ms: muscovite; Njrs: natrojarosite; Qz: quartz.

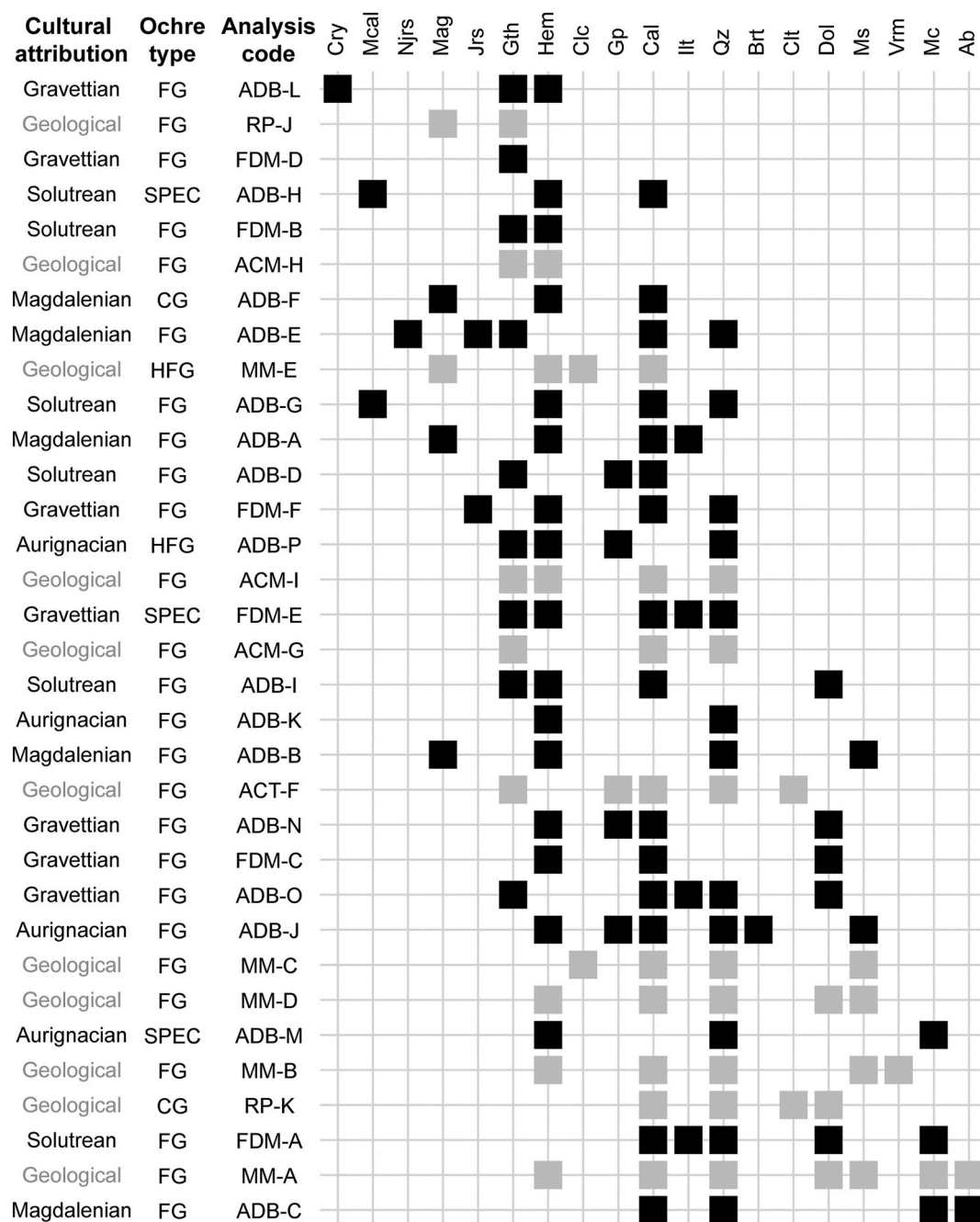


Fig. 10. Seriation analysis of mineral phases identified by XRD on colouring materials from Abrigo de la Boja, Finca Doña Martina and on geological samples. FG: fine-grained ochre type, SPEC: specularite ochre type, HFG: hard fine-grained ochre type, CG: coarse-grained ochre type. Crystalline phases (according to [Whitney and Evans, 2010](#)): Ab: albite; Brt: barite; Cal: calcite; Mcal: magnesian calcite; Clc: clinocllore; Clt: celestine; Cry: cryptomelane; Dol: dolomite; Gth: goethite; Gp: gypsum; Hem: hematite; Ill: illite; Jrs: jarosite; Mag: magnetite; Mc: microcline; Ms: muscovite; Njrs: natrojarosite; Qz: quartz; Vrm: vermiculite.

6. Conclusion

Although the use of ochre is widespread during the Upper Palaeolithic, it remains poorly documented, particularly in contexts apparently unrelated to art.

Due to the high concentration of Palaeolithic art in northern Iberia, ochre assemblages from Upper Palaeolithic contexts have been more extensively studied in this region, at sites such as Santa Catalina ([Pastor and Gochi, 2022](#)), Lumentxa ([Arribas Pastor et al., 2021](#)), Praileaitz ([Queffelec et al., 2017](#)), or Coímbre ([García et al., 2016](#)), among others. These studies reveal a complex use of this material, combining different processing techniques, such as flaking, grinding and scraping or scoring,

not only for the production of art, but also for other functional purposes.

In SE Iberia, however, a significant gap remains in the study of ochre assemblages from Upper Palaeolithic contexts. Evidence for ochre use has been reported at different sites (for the Region of Murcia, for instance, see [Andreu, 1992](#)) but systematic studies of these assemblages are rare.

Ochre used for the production of painted portable art was analysed at Parpalló Cave, revealing the use of hematite and goethite-based pigments, as well as manganese and charcoal, for the production of painted portable art, in occupations dated between 30 and 14.6 ka BP in calibrated ages (estimation based on the regional sequence, [Aura Tortosa and Villaverde Bonilla, 2014](#); [Roldán et al., 2013](#); [Roldán García et al.,](#)

2016; Villaverde Bonilla, 1994). Ochre pieces and ochre processing tools recovered from the same levels are the object of ongoing studies by one of us (DER). Preliminary results show that a variety of techniques, including pounding and grinding, were used throughout the sequence to produce different types of ochre powder for artistic purposes, but likely also for other activities (Rosso et al., 2024).

The cases of FDM and ADB appear distinctive when compared to the few Upper Palaeolithic ochre assemblages that have been systematically studied. Although the raw materials at ADB and FDM vary in colour, texture, and composition, processing techniques are less diverse, consisting of crushing ochre to exhaustion, with virtually no evidence of grinding throughout the sequence. This pattern contrasts with what is typically observed at other Upper Palaeolithic sites, where crushing, flaking, grinding and scoring are generally combined. Further studies of ochre use in residential sites across SE Iberia may, in the future, allow to determine whether this pattern is also found in nearby localities and provide insights into the underlying causes of these differences, and potentially link them to specific activities.

Another systematically studied ochre assemblage from a residential site in SE Iberia is that of Cova de Santa Maira (Aura Tortosa et al., 2021). This site, which features more recent occupations than ADB and FDM, yielded an ochre assemblage from layers dated between 15 and 6 ka cal BP. More than 50 % (by weight) of the ochre was recovered from Mesolithic layers, while earlier occupations (attributed to the Final Magdalenian, Epipalaeolithic and Sauveterroid Epipalaeolithic, 15–10.2 ka cal BP) reflect a less frequent use of this material. This aligns with our findings, which point to a decline in ochre-related practices in the final stages of the Upper Palaeolithic in the region. Future research in Upper Palaeolithic sites across SE Iberia will be essential to determine whether this change is reflected in other sites.

Abrigo de la Boja and Finca Doña Martina rock-shelters represent a first step to systematically documenting a key cultural feature of SE Iberia's Upper Palaeolithic. Evidence from these sites reveals complex and shifting patterns of raw material acquisition, coupled with a technical system that persisted throughout the Upper Palaeolithic. A possible cultural shift indicating a decline in ochre-related activities during the final phases of the Upper Palaeolithic was also identified. These findings not only expand our understanding of ochre-related practices in residential contexts but also offer valuable insights into the mechanisms involved in the transmission and evolution of cultural practices. Further research at other sites across the region will be crucial to assess the significance of these patterns in SE Iberia and to better understand regional variability in ochre use during the Upper Palaeolithic.

CRediT authorship contribution statement

Daniela Eugenia Rosso: Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Àfrica Pitarch Martí:** Formal analysis, Conceptualization, Writing – review & editing, Writing – original draft, Methodology, Investigation. **François Orange:** Formal analysis. **Josefina Zapata:** Investigation, Funding acquisition. **João Zilhão:** Writing – review & editing, Writing – original draft, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization.

Data availability statement

The data that support the findings of this study are included as part of the paper and as supplementary materials.

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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.jas.2025.106342>.

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