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Red Balloon rock shelter Middle Stone Age ochre assemblage and population's adaption to local resources in the Waterberg (Limpopo, South Africa)

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Abstract

Ochre has been found at many Middle Stone Age sites throughout southern Africa. Much work has been done to document these iron-rich raw materials, their modifications and their implications for past communities' behaviours, skills and cognition. However, until recently few works focused on the Middle Stone Age Waterberg ochre assemblages. The paper presents the ochre assemblage recovered at Red Balloon rock shelter, a new Middle Stone Age site on the Waterberg Plateau. The site preserves Middle Stone Age occupations dated around 95,000 years ago. Scanning electron microscopy observations, portable X-ray fluorescence spectroscopy and infrared spectroscopy characterization document the presence of four ochre types. The MSA ochre assemblage recovered is mainly composed of specularite and specular hematite similar to the ones of Olieboomsport and North Brabant. Microscopic observations and infrared analyses of soil sediment and of post-depositional deposits found on the ochre pieces show that this raw material specificity is of anthropic origin and not the result of post-depositional processes. Optical and digital observations of the archaeological assemblage and its comparison with a preliminary exploratory experimental one highlight the use of abrasion and bipolar percussion to process the ochre pieces at the site. The results point to the know-how and skills of the Middle Stone Age populations who inhabited the Waterberg region around 95,000 years ago. This raises the question of whether the specificities of the Waterberg ochre assemblages correspond to populations' adaptation to the local mountainous mineral resources and the existence of a regional ochre processing tradition.

Keywords Ochre sensu lato · Middle Stone Age · Percussion · Abrasion · Waterberg · South Africa

Introduction

Middle Stone Age was a period of important innovations in human populations. Such innovations include heat-treatment of silcrete (Schmidt et al. 2020), and hafting adhesive (Lombard 2006, 2007; Wadley et al. 2009; Charrié-Duhaut et al. 2013). Collected since 500 ka, ochre uses developed during the MSA (Watts et al. 2016; Dapschaskas et al. 2022). In archaeology, ochre is a catch-all term that refers to ferruginous material with strong colouring and covering powers (Wadley 2005a, b; Hodgskiss 2012, 2013, 2014; Roebroeks et al. 2012; Dayet et al. 2013, 2016, 2017; Mauran et al. 2020, 2021; Popelka-Filcoff and Zipkin 2022).

Their symbolical use has long been debated (Dart 1975; McBrearty and Brooks 2000; Watts 2002, 2009; d'Errico 2003, 2008; Henshilwood and Marean 2003; Wadley 2005a, b; Soriano et al. 2009; Rifkin et al. 2015, Rifkin 2015; Dapschaskas et al. 2022). It is nowadays established that such material could have been used for many other purposes than mere pigments or symbolical purposes such as adhesive charge (Wadley 2005b; Lombard 2006, 2007), abrasive agent (Audouin and Plisson 1982), mosquito repellent (Rifkin 2015), hide tanning agent (Rifkin 2011), sun protection agent (Rifkin et al. 2015; Havenga et al. 2022), antibacterial agent (Havenga et al. 2022) and potentially as knapping hammers (Soriano et al. 2009).

Here the term "ochre" is used in its archaeological sense, ochre sensu lato, as suggested by Popelka-Filcoff and Zipkin (2022). They include a large variety of rocks and residues resulting from their exploitation and use. Among

them are the hematite ores which exhibit different textures such as massive, microlamellar or micro-fibular. Specularite, *Sebilo* in Setswana (Robbins 2016), *Qhang-qhang* in Xhosa (How 1962), *//hára* as mentioned by /Xam (Bleek et al. 1911, p.7272–7276), corresponds to one of these kinds of hematite (Thackeray et al. 1983; Kiehn et al. 2007; Robbins 2016; Watts et al. 2016). It might sometimes be referred to as “marron hematite” (Schoonraad and Beaumont 1968). It is a grey metallic hematite with a randomly oriented micro-lamellar texture. When rubbed or crushed into a broad powder it has a purple hue with shiny particles, but ground to a fine powder the hue turns to a dark red with few shiny particles. Well known to southern African communities, it has been extensively mined from the Middle Stone Age up to historical times at various locations in nowadays Botswana, Eswatini, and South Africa (Dart and Beaumont 1969; Beaumont 1973; Thackeray et al. 1983; Kiehn et al. 2007; Robbins 2016).

Excavations of southern African MSA sites have often yielded ferruginous colouring materials (see synthesis in Lombard et al. 2012; Wadley 2015). Though several sites attest the existence of ochre exploitation between 500 and 200 ka, their use became habitual around 160 ka (de Lumley 1966; de Lumley-Woodyear 1969; Yokoyama et al. 1986; Clark and Brown 2001; Barham 2002; Watts 2002; Roebroeks et al. 2012; de Lumley et al. 2016; Watts et al. 2016; Dapschuskas et al. 2022). From about 160 ka, South African Middle Stone Age ochre assemblages are more common (Fig. 1; Watts 1999, 2002, 2009, 2010; Henshilwood et al. 2011, 2018; Hodgskiss 2012, 2013; Bernatchez 2013; Dayet et al. 2013, 2016, 2017; Hodgskiss and Wadley 2017; Dapschuskas et al. 2022). The studies carried out on these assemblages have offered inferences about past population cognitive abilities (Hodgskiss 2014), procurement strategies (Dayet et al. 2016; McGrath et al. 2022) and their uses of these raw materials.

Though archaeological sites have been excavated in both coastal and interior areas of southern Africa, most studies have focused on coastal sites such as Blombos Cave (Henshilwood et al. 2011, 2018) and Klasies River main site (Dayet et al. 2017) or sub-coastal sites such as Sibudu Cave (Hodgskiss 2012, 2013). The scarce MSA ochre provenance studies that have yet been carried out are all from coastal regions (Bernatchez 2013; Dayet et al. 2016, 2017; McGrath et al. 2022). Within these coastal and sub-coastal assemblages, grinding is well documented on around 10% of the ochre pieces, thanks to experimental works such as the one of Hodgskiss (2010).

For long southern African archaeologists focused on identifying abrasion use-wear on ochre pieces and debated their relation to symbolic behaviours (Watts 1999, 2002, 2009; Henshilwood and Marean 2003; Wadley 2005a). Thus, little is known about ochre percussion. It has nonetheless been identified at some rare southern African sites such as Klasies River main site (Dayet et al. 2017), and Blombos Cave (Henshilwood et al. 2009). Unipolar knapping of ochre pieces has been described on some ochre pieces (Henshilwood et al. 2009; Salomon 2009; Rosso et al. 2016). Its identification relied on percussion scars similar to what has been described for lithic tools. Though some experimental studies have been published (Salomon 2009), the identification of percussion suffers from the lack of common terminology and a large predominance of studies focusing on abrasion use-wear. This lack of knowledge hinders our understanding of ochre processing “chaînes opératoires” as defined by Perlès (1987, p. 23) and their evolutions. As both have the potential to interrogate the sociocultural behaviours of the MSA populations, this lack of knowledge hinders our understanding of MSA populations’ cultural choices and traditions in a region and at a period of extreme importance in humankind’s evolution.

The situation in the interior of southern Africa remains understudied despite the recent works carried out in different parts of the interior of southern Africa (e.g. van der Ryst 2007; Backwell et al. 2014; Porraz et al. 2015, 2018; Stewart and Mitchell 2018; de la Peña et al. 2019; Val et al. 2021; Wadley et al. 2021). With these studies, archaeological interest in mountainous regions has waxed in recent years. The Waterberg in the Limpopo province is no exception as summarised by Wadley et al. (2021). Though these geographical biases are about to be fixed, they further hinder our understanding of social-cultural practices and networks.

In the Limpopo region (South Africa) several sites have delivered MSA ochre assemblages of interest: Bushman Rock Shelter, Mwulu’s Cave, Olieboomspoor, and North Brabant (Fig. 1; Tobias 1949; Mason 1962; Schoonraad and Beaumont 1968; Watts 2002; van der Ryst 2007; Porraz et al. 2015; de la Peña et al. 2019; Val et al. 2021). Other MSA sites have been excavated, but the apparent absence of ochre pieces such as at Steenbokfontein or the scarcity of ochre at sites such as Wonderkrater or Cave of Hearths bring little information about ochre exploitation in this region of southern Africa (Backwell et al. 2014; Wadley et al. 2016; Dapschuskas et al. 2022).

While Bushman Rock Shelter and Mwulu's Cave present a wide diversity of ochre raw materials, the sites of the Waterberg strike out from the rest of southern African sites with a large predominance of "maroon hematite" or specularite (Schoonraad and Beaumont 1968; Watts 2002; van der Ryst 2007; Val et al. 2021). As the Waterberg region might have experienced wetter conditions for a long period, one can wonder if the specificities of the ochre assemblage of this region are not to be related to post-depositional processes that can prevent the preservation of the softest raw materials or if it is the result of anthropogenic choices (Wadley et al. 2021).

This paper presents the ochre assemblage from the MSA site of Red Balloon rock shelter (Waterberg, Limpopo, South Africa). Though the site does not present a long cultural sequence, it is of importance as it is one of the two dated MSA sites from the Waterberg plateau which has received lesser attention than other South African regions. The present paper aims at (1) documenting the Red Balloon rock shelter ochre assemblage and (2) questioning its specificities within southern African MSA contexts. Documenting the ochre assemblage requires interrogating its representativity and its potential alteration by post-depositional processes, as well as documenting the way the ochre pieces were worked at the site. In the present paper, this relies on the comparison of the archaeological assemblage with preliminary exploratory experimentation presented in SI 2.

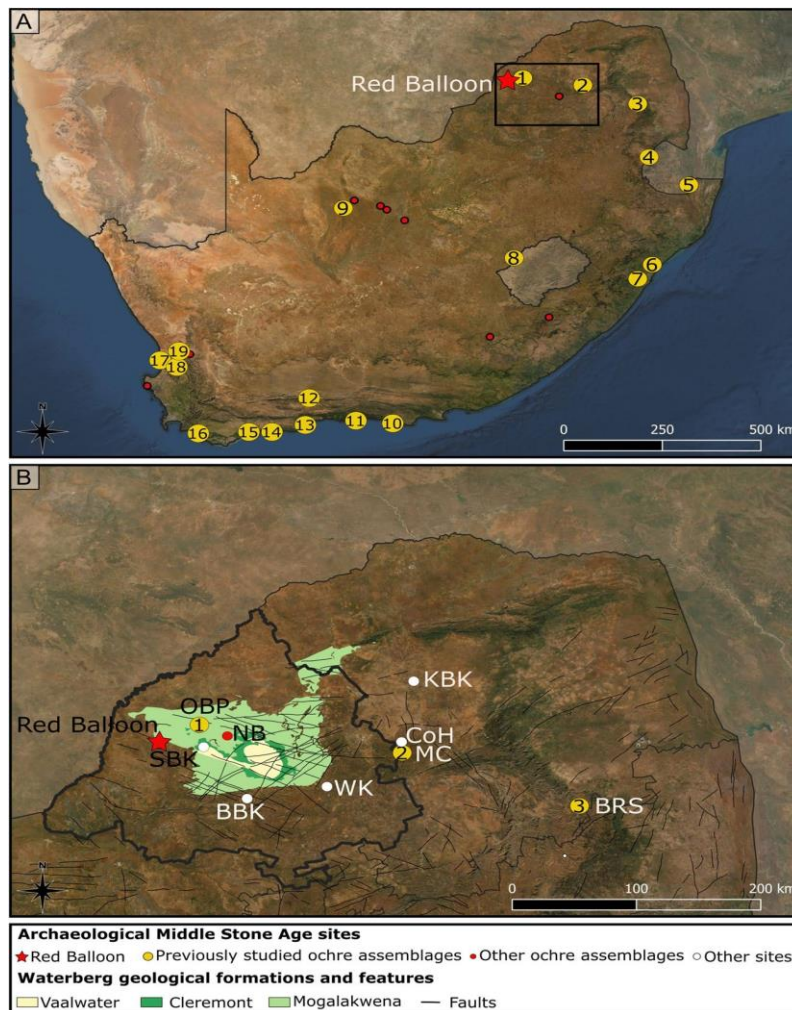
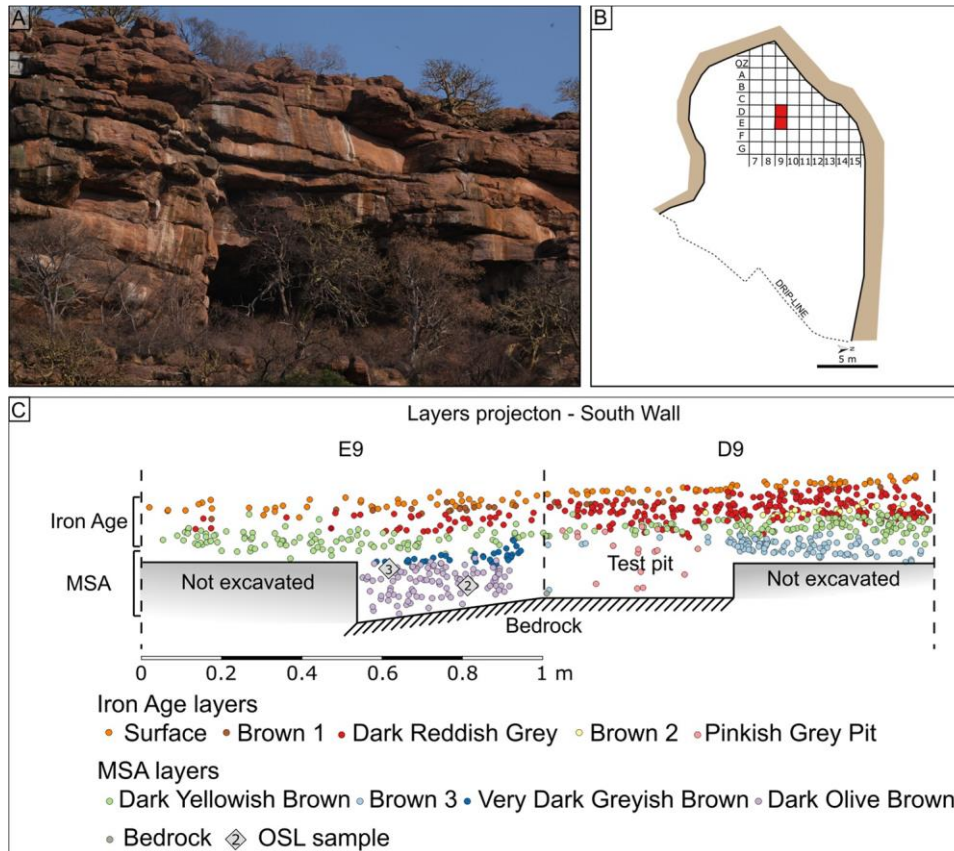


Fig. 1 A Map of south African sites mentioned in the article. B Archaeological and geological map of Waterberg region. 1, Olieboomspoor; 2, Mwulu's Cave; 3, Bushman Rock Shelter; 4, Lion Cavern; 5, Border Cave; 6, Sibudu Cave; 7, Umhlatuzana; 8, Rose Cottage; 9, Kathu Pan; 10, Klasies River Mouth; 11, Nelson Bay Cave; 12, Pinnacle Point; 13, Boomplaas; 14, Blombos Cave; 15, Klipdrift Shelter; 16, Die Kelders; 17, Hoedjiespunt; 18, Elands Bay Cave; 19, Diepkloof Rock Shelter. Maps G.M. Geographical coordinates are provided in SI 1.

1 **Red Balloon rock shelter**

2
3 Red Balloon rock shelter (Fig. 2) is on the north-western edge of the Waterberg plateau, close to Vaalwater (Ellisras, Limpopo), about 15 km south of
4 Olieboomspoor and 100 km east of Mwulu's Cave (Fig. 1). Red Balloon rock shelter is at an altitude of about 1200 m above mean sea level on an east-facing
5 sandstone cliff. It lies within the mid-Proterozoic ferruginous sandstone of the Mogalakwena Formation which overlies high-grade rocks of the Limpopo Belt
6 (Ericsson et al. 1997; Distler et al. 2000). The granular sandstone, siltstone and lenses of quartz-conglomerate of the Mogalakwena Formation are cut through
7 quartz and hematite hydrothermal veins (Corcoran et al. 2013; Oberthür et al. 2018).
8

9 **Fig. 2** The site of Red Balloon rock shelter. A Photograph of the entrance of the site. B Map of the site with location of the excavated squares in red. C Projection of the find



10 recorded with EDM according to the layer they belong. Photo, map and projection GM.

11
12 The shelter is large with a dry living area of greater than 20 × 12 m (Fig. 2). Red Balloon rock shelter lacks rock art, but an adjacent shelter has a few red fine-
13 line paintings and white finger paintings, two of which appear to be spread hides (Laue 2001). Red Balloon rock shelter was excavated in 2020 (Wadley et al.
14 2021). Excavations conducted on less than 2 m² revealed a rather shallow deposit of 30 cm comprising a total of 325 L of sediment excavated.

15
16 The sequence registers occupations phases of the Iron Age and Middle Stone Age (Fig. 2), as observed elsewhere on the high plateau (van der Ryst 2007). IA
17 layers comprise a pit (Pinkish Grey Pit) that disturbed the MSA underlayers (Fig. 2; Wadley et al. 2021). MSA occupations were OSL dated around 92–100 ka
18 ago during MIS 5 (Wadley et al. 2021). While IA layers delivered organic materials, one glass bead, 33 ostrich eggshell beads and 154 pottery shards, MSA
19 layers have only delivered lithic tools and ochre pieces but no organic remains (Wadley et al. 2021). The lithic assemblage recovered at Red Balloon rock
20 shelter is made of rhyolite, siltstone, quartz and quartzite. All these raw materials can be found in the Waterberg range. A majority of the lithic core assemblage
21 (N = 119) is made of bipolar cores (N = 54). Twenty Levallois cores and some points have also been found.
22

23 **Materials**

24
25
26 Materials analysed here include the whole 2020 Red Balloon rock shelter's archaeological ochre assemblage. Each piece of length higher than 10 mm (fragment,
27 flake or nodule) was labelled separately, measured and examined. Colour, hardness, cohesion, magnetic properties, presence of patina, lustre, cement or
28 matrix composition, structure and texture as well as inclusions nature, distribution and structure were described for each ochre piece longer than 10 mm.
29 Description of the different ochre pieces are the results from these observations and are thus presented in the results section (see Results - Raw material
30 identification). Identification of small ochre fragments can be difficult due to the heterogeneity of ochre fragments. Thus, pieces smaller than 10 mm were
31 counted, and weighed by bucket point, but were not labelled separately. These small pieces were classified according to macroscopic observations and were
32 not described with as much detail as the larger ochre pieces. Refitting of small and large pieces has been attempted over the whole excavation. Only two pieces
33 were refitted and labelled as one.

34 Most MSA ochre pieces had a brown crust on their surface. After photographing the ochre pieces with these crusts, analyses and sampling of these crusts,
35 ochre samples were cleaned in deionised water using ultra-sonification for 2 min. Soil samples from the different layers that have delivered ochre pieces were
36 also analysed to understand the post-depositional processes that affected the assemblage. Experimental material is presented in SI 2.
37

38 **Methods**

39 **Macroscopic observations and digital microscopy**

40
41 Observations were carried out with a binocular (1 to 40 × magnification) and a digital microscope Dino-Lite (20 to 200 × magnification). Pieces bigger than 10
42 mm were all photographed with a Fujifilm XT-3 Camera with a Fujifilm XF 80 mm aspherical macro lens. Microscopic pictures were taken of worked and
43 representatives unworked pieces with the Dino-Lite digital microscope and the DinoCapture 2.0 software.

44 **Percussion marks terminology**

45
46

47 Techno-typological features consisted of both abrasion use-wear and percussion marks. Use-wear traces were characterised according to Hodgskiss's (2010)
48 protocol. Macroscopic characteristics of the percussion marks follow Vettese et al. (2020) work on bone percussion and breakage. Described features
49 consisted of percussion notch, crushing marks, adhering flakes, micro-striations, flake and fracturing. The definitions of these different percussion marks are
50 presented here thanks to observations carried out on the experimental material presented in SI 2.

51 Fracturing edges vary from rough to sharp edges of a "fresh surface" that does not exhibit any patina (Fig. 3). They do not constitute a diagnostic feature
52 of anthropic percussion as they could form naturally.

53 Crushing marks are percussion scars visible thanks to the presence of a dense patina on some of the ochre pieces. The patina absorbs the shock of the
54 impact and reduces its impact on the rest of the piece. They often form roundish patches where the patina is of lesser thickness than the rest of the piece
55 (Fig. 3A–C). They appear where repeated blows were performed.

56 Percussion of some of the ochre pieces leads to the creation of flakes, similar to the ones produced by stone tool manufacturing. The most characteristic
57 features are the presence of a platform at the impact point and the presence of ripples below the platform, where a bulb can sometimes be seen (Fig. 3D–F,
58 H and J).

59 Percussion notching is found on a fracturing edge and it corresponds to a negative flake scar. It consists in a small negative flake scar that forms
60 perpendicular to the main negative flake scar where the impact point lies. Percussion notches can appear isolated or in opposite pairs, complete (two changes
61 of curvature) or incomplete (one change of curvature). Double opposite notches correspond to two percussion notches on the opposite part of a fragment.
62 Double opposite notches are typical of bipolar percussion (Fig. 3A–C, H–I, K and M).

63 An adhering flake/fragment is a flake or a micro-fragment still attached to the ochre piece (Fig. 3K–L). They usually occur next to percussion pits or crushing
64 marks.

65 Randomly oriented micro-striations are sometimes visible on the surface facing the anvil. They form due to the roughness of the anvil, due to scrubbing
66 between the anvil and the ochre piece being crushed. The sole presence of these randomly orientated micro-striations does not allow one to determine the
67 use of percussion. These micro-striations must be associated with the presence of other percussion characteristics.

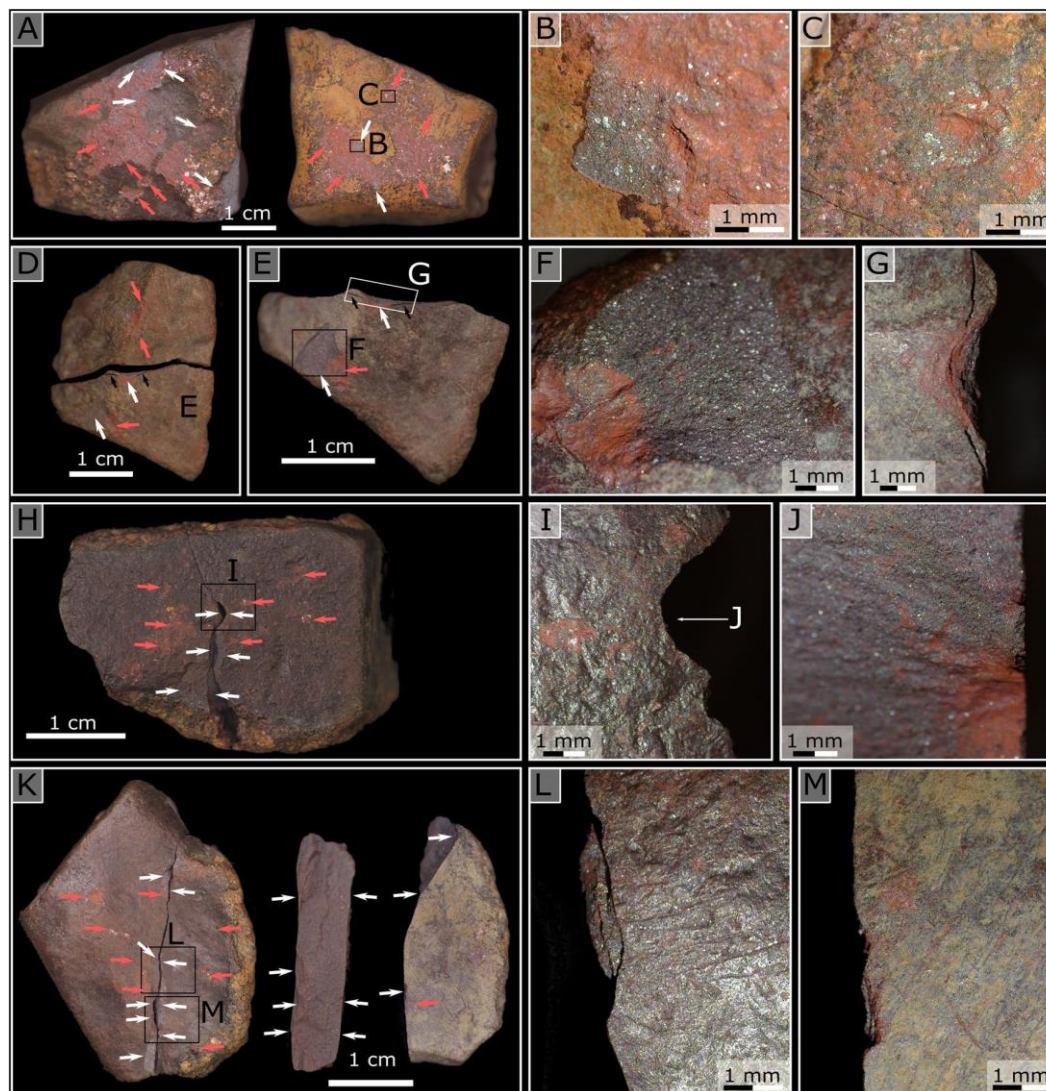
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70 **Scanning electron microscopy**

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72 Microscopic observations of selected representative pieces and sediment samples were carried out with scanning electron microscopy. Selected samples
73 were mounted on double-sided carbon tape and cleaned with compressed air. Carbon tape was used as there is no organic preservation and will thus not
74 bias future non-invasive analyses. Observations were performed with a Phenom Pure desktop SEM (Thermo Fisher Scientific) located at the Evolutionary
75 Studies Institute (ESI, University of the Witwatersrand, Johannesburg). They were carried out in low-vacuum mode in Backscatter Electron mode, with a
76 field-emission source of 10 keV. The equipment was not coupled with an EDS detector. The COVID-19 pandemic prevented access to an SEM with an EDS
77 detector.

78 These observations enabled the identification of both petrographic and techno-typological features. Petrographic features documented corresponded to
79 the texture, nature, morphology and abundance of matrix, presence, nature and abundance of inclusions. After analyses of all archaeological and geological
80 samples, all samples were classified according to rock types and petrographic features.



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Fig. 3 Selected percussed experimental ochre (A, D with zoom E, H and K) and percussion marks identified during the percussion experiment: crushing mark (B), crushing mark (C), negative flake with ripples (F), complete notch (G), double incomplete notches (I), negative flake with ripples (J), adhering flake (L), double complete notches (M). Arrows point to different percussion marks.

85 **Fourier transform infrared spectroscopy**

86

87 Fourier transform infrared (FTIR) spectroscopy was carried out on the ochre pieces and the crust covering it with an Alpha FTIR spectrometer (Bruker) at ESI
88 (University of the Witwatersrand, Johannesburg). Non-invasive analyses were performed on the ochre pieces with a DRIFT module. Spectra were acquired in
89 the 400–4000 cm^{-1} mid-IR range with 32 scans and a resolution of 4 cm^{-1} .

90 Micro-samples of the crust covering the ochre pieces and sediment samples were crushed in an agate mortar and analysed with an attenuated total reflectance
91 (ATR) module equipped with a diamond crystal. These ATR FTIR spectra were also acquired in the mid-IR between 400 and 4000 cm^{-1} but with 64 scans and
92 a resolution of 4 cm^{-1} . Between two ATR-FTIR analyses, the FTIR was cleaned with ethanol.

93 For both procedures, background spectra were recorded before each sample and the spectra were corrected with an atmospheric compensation using software
94 OPUS 7.5 software.

95

96 **Portable X-ray fluorescence spectrometry**

97

98 Representative pieces of the archaeological and modern assemblage that were bigger than 8 mm and with a flat surface were analysed using a Thermo
99 Scientific Niton XL3t 950 portable XRF at the Geosciences Department of the University of the Witwatersrand (Johannesburg). The pXRF equipment is fitted
100 with a GOLDD + drift detector and a miniaturised X-ray tube with a 50 kV excitation source. Each piece was tested with two programs. The first program,
101 “mining” lasting 180 s, is designed for major and minor elements semi-quantification (Mg, Al, Si, P, Cl, Fe, Ca, S, K, Ti, Cr, Mn) while the second one, “soil”
102 lasting 90 s, is designed for trace metal elements (Fe, Ca, S, K, Ti, V, Cr, Mn, Co, Ni, Cu, W, Zn, As, Rb, Sr, Zr, Mo, Sc).

103

104 Each ochre piece was analysed six times. Every morning BHVO2 (basalt), NIM-P (pyroxenite), and NIM-D (dunite) standards were analysed to ensure proper
105 calibration of the XRF. These standards were the three standards with the highest iron concentration at the Geosciences laboratory. The pXRF measurements
106 were performed to confirm the qualitative chemical similarity between the modern and archaeological collections. Quantitative pXRF is out of the scope of the
107 present article.

108

109 Six soil samples were analysed by pXRF using the “soil” 90 s analyses. Soil samples were placed between two Mylar films before analyses. These analyses
110 aimed to investigate the possible post-depositional heavy elements contamination.

111

112 As the pXRF was not calibrated to analyse the ferruginous matrix, the results should be seen as qualitative. All results are presented in the supplementary
113 information (SI 3).

114

115 **Stratigraphic and spatial distributions**

116

117 Stratigraphic and spatial distributions were investigated through ochre piece numbers and mass per layers identified at Red Balloon. To prevent bias due to
118 layer volume, stratigraphic number and mass distributions were normalised to layers' volume to provide stratigraphic density and mass density of ochre pieces

119 at Red Balloon rock shelter. Stratigraphic and spatial distributions were investigated through recorded pieces thanks to the QGIS software and the stratigraphic
120 densities graphs thanks to Excel.

121

122 **Results**

123 The Red Balloon rock shelter ochre assemblage comprises 459 pieces for a total weight of 2.9 kg. Two hundred fiftyfour fragments (55.3%) representing a
124 weight of 0.2 kg (8.7% in weight) are smaller than 10 mm. These small ochre pieces were counted and included in considerations about ochre stratigraphic
125 distributions.

126

127 **Post-depositional alteration of the ochre pieces**

128

129 A brown crust was covering 65% and 95% of the IA and MSA samples, respectively. The ultra-sonification helped to remove some of the crust, allowing
130 observation of non-patinated surfaces to identify the raw material. It did not completely remove the crust. The presence of such crust on the pieces might have
131 prevented the identification of some percussion marks.

132

133 IR spectra of the crust exhibit strong characteristic bands of quartz at 693, 778, 795 and 1162 cm^{-1} and kaolinite at 3621, 3658 and 3696 cm^{-1} (Fig. 4A). Bulk
134 IR analyses of the sediment presented the same composition. Hearth sediments present strong calcite features (Fig. 4). Sediment single-grain binocular
135 observations coupled with IR analyses allowed the identification of the following phases: altered bone, micro-fragments of black hematite, mica, ash, clay,
136 rootlets and quartz. The micro-fragments of hematite correspond to angular grains that could result from the exploitation of ochre on the site. Due to their
137 morphology, there is a little risk they result from the post-depositional chemical alteration of the archaeological ochre pieces.

138

139 SEM observations of the crust covering the ochre pieces confirm the crust to be a clay-rich mixture tending to be of higher porosity towards its outer part (Fig.
140 4B, C).

141

142 Though pXRF analyses performed in this study should be seen as qualitative, pXRF analyses of soil samples highlighted some compositional differences
143 between the IA and MSA layers (SI 3). Surface and DRG exhibited a CaO concentration of around 3.5%, while MSA layers (Brown 3 and Brown 3 indurated)
144 had a CaO concentration of around 1.3%. Naturally, combustion features DRG Hearth, DRG Hearth Ash and Greyish Brown exhibited higher CaO, respectively,
145 of 19.2%, 5.4% and 5.5%. Sr concentration presented a similar trend (SI 3).

146

147 While K_2O concentrations appear similar between the IA and MSA layers, MSA layers presented higher Fe_2O_3 contents (around 2.2%) than the IA layers
148 (around 1.2%) (SI 3). A similar trend appears for Cr contents, this might be due to the pXRF iron escape lines. Zn concentrations are slightly lower in the MSA
149 layers (around 80 ppm) than in the IA layers (around 120 ppm) (SI 3). The analyses did not evidence the existence of elemental contamination detectable with
150 pXRF within the Red Balloon rock shelter stratigraphic sequence.

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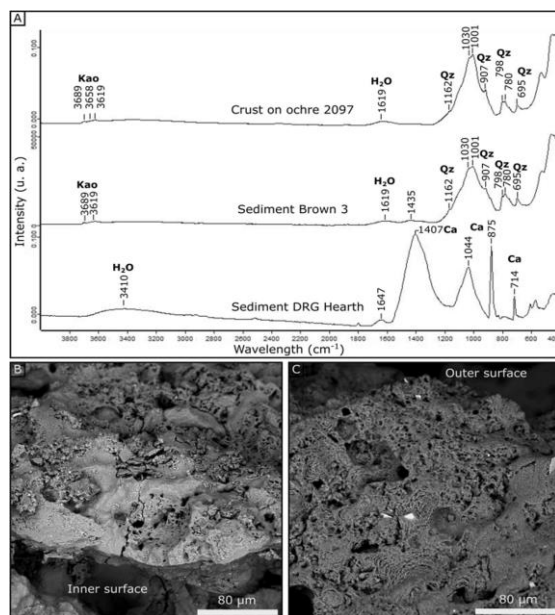


Fig. 4 A FTIR analyses of crust on ochre piece 2097 and of sediments from Brown 3 and Dark Reddish Grey – Hearth layers (made with OPUS software). B SEM pictures of a micro-sample of post-depositional crust covering ochre 2097 retrieved in layer Brown 3.

Raw material identification

Within the 205 pieces larger than 10 mm, four different categories of ochre were identified: grey hematite, specularite, sandstone and breccia (Fig. 5, Table 1). Some of these ochre fragments exhibited magnetic properties ($N = 24$, 11.7%).

Table 1 Summary of the ochre pieces recovered at Red Balloon rock shelter (Limpopo, South Africa). N: number of pieces, M: mass in g.

Layer	Grey hematite				Specularite				Ferruginous sandstone				Ferruginised breccia				Total – N(M)		Mica
	Total pieces >10 mm– N(M)	Utilized		Magnetic	Total pieces >10 mm– N(M)	Utilized		Magnetic	Total pieces >10 mm– N(M)	Utilized		Magnetic	Total pieces >10 mm– N(M)	Utilized		Magnetic	Total pieces >10 mm– N(M)	Total pieces <10 mm– N(M)	Total
		Ground - N	Percussed - N			Of which N core-shaped	Ground - N			Percussed - N	Of which N core-shaped			Ground - N	Percussed - N				
<i>Iron Age layers (disturbed MSA material)</i>																			
Surface	7 (55.3)	2			1 (24.2)				1 (8.3)	1			0				9 (85.8)	9 (7.1)	0
Dark Reddish Grey	16 (193)	6		1	3 (76)	1	2	1	7 (45.9)	4		2	0				26 (314.9)	16 (25.2)	3
Pinkish Grey Pit	4 (21.3)	3			2 (8.8)		2		0				0				6 (30.1)	16 (12)	0
Total IA	27 (267.6)	11		1	6 (109)	1	4	1	8 (54.2)	5		2	0				41 (430.8)	41 (44.3)	3
<i>Middle Stone Age layers</i>																			
Dark Yellowish Brown Hearth	14 (191.2)	9			4 (44.3)		3		2 (16.8)	2		1	0				20 (252.3)	27 (20.5)	3
Dark Yellowish Brown	37 (401.1)	22	2	3	8 (124.3)		5	1	1	3 (30.1)		2	1	1 (16.3)			49 (571.8)	62 (66.9)	0
Brown 3 Hearth	14 (123.5)	2	12		2		1 (15)			4 (38.7)		4	4	0			19 (177.2)	52 (43.7)	0
Brown 3	31 (367.2)	2	20		3		11 (252.4)		2	7	1		3 (58.5)		1	1 (4.4)	46 (682.5)	38 (42.6)	3

Dark Olive	16 (247.9)		10		1	9 (128.9)		6			2 (28.2)			2	0			27 (405)	22 (23.5)	2
Brown																				
Very Dark Greyish	3 (111.7)		2	1		0					0				0			3 (11.7)	12 (10)	0
Brown																				
Total MSA	115 (1442.6)	4	74	3	9	33 (564.9)	2	20	2	1	14 (172.3)	8		11	2 (20.7)			164 (2200.5)	213 (207.2)	8
TOTAL	142 (1710.2)	4	84	3	10	39 (673.9)	3	24	3	1	22 (226.5)	13		13	2 (20.7)			205 (2631.3)	254 (251.5)	11

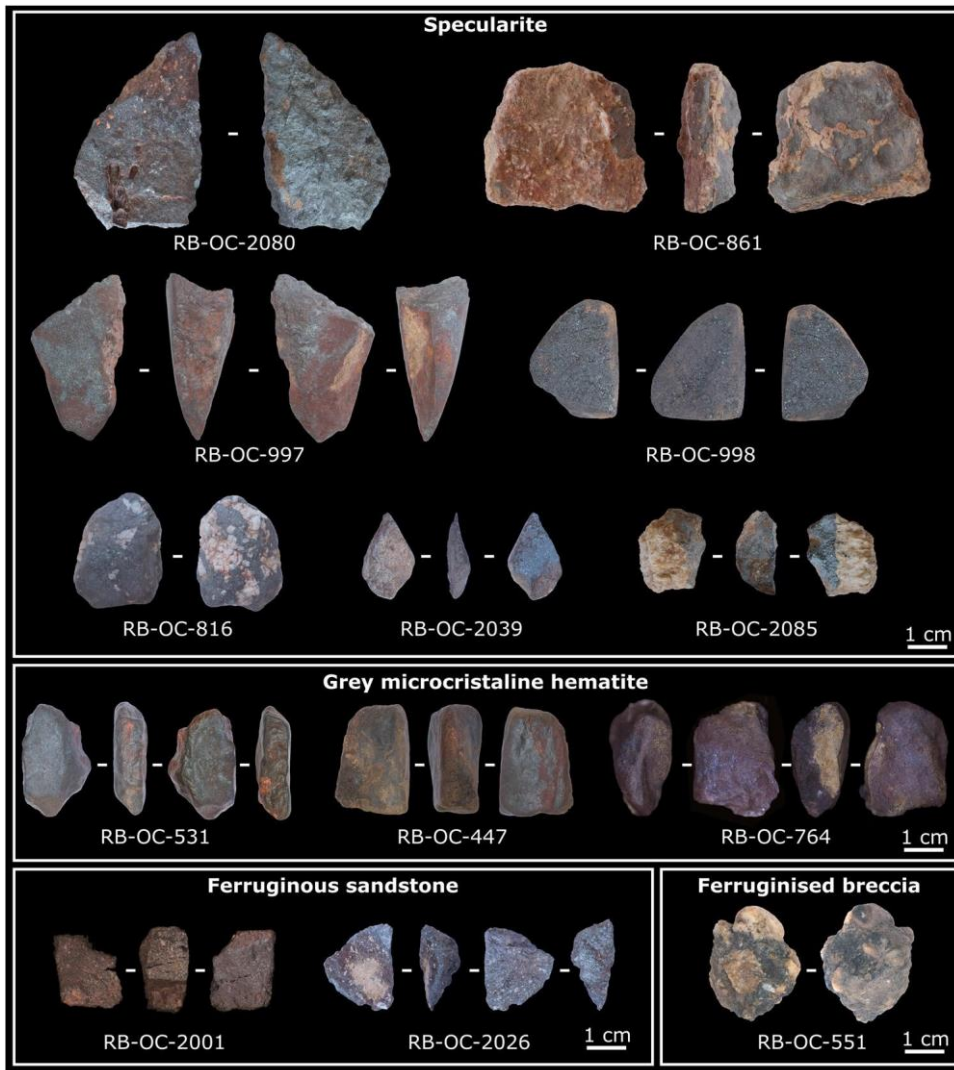


Fig. 5 Selected undisturbed MSA ochre pieces recovered at Red Balloon rock shelter (Limpopo, South Africa).

Grey hematite samples present a heterogeneous metallic to sub-metallic lustre. Among the hematite samples of Red Balloon rock shelter, there are two different types: microcrystalline platy specular hematite and microcrystalline hard hematite (Fig. 5). Microcrystalline platy specular hematite samples have a fluid-like structure with locally oriented euhedral sutured micro-platy fine crystals of 10 μm length and a thickness of less than 1 μm (Fig. 6). Microcrystalline hard hematite samples are composed of randomly oriented sutured anhedral micro-crystals of 5 μm size (Fig. 6). Both types present some fine to coarse white to reddish quartz inclusions representing between 5 and 15% of the samples' composition. According to the pXRF analyses, the two hematite types are mainly composed of iron oxide (Fe_2O_3 above 80%) with minor CaO and K_2O content (1–2%), possibly contaminated by the sediment in which they lay (SI 3).

Specularite samples are grey with a non-uniform metallic lustre. They consist of randomly oriented sutured platy euhedral hematite crystals of at least 100 μm length. The thickness of these platy crystals varies from one sample to another between 2 and 20 μm (Fig. 6). Samples with thicker crystals are the hardest. Specularite samples also present some large white to reddish rounded quartz inclusions, representing up to 45%. pXRF analyses of specularite samples confirmed these to be rich in iron oxide (Fe_2O_3 content above 73%, SI 3). Analyses of sample RB-OC-0057 present higher SiO_2 content than the other samples. This could be due to quartz inclusions.

Ferruginous sandstones are grey to red with heterogeneous sub-metallic lustre. They consist of a ferruginous matrix made of micro-aggregates of fine, randomly oriented micro-platy hematite crystals cementing subangular, altered quartz inclusions (Fig. 6). Quartz inclusions represent between 45 and 60% of the ferruginous sandstone samples'

composition. The quartz grains are comprised between 62 and 2 mm. They are packed and cemented by a secondary microcrystalline ferruginous matrix. The ferruginous sandstones differ from the altered ferruginous sandstone slabs coming from the roof due to their iron oxide content and petrographic features.

Ferruginised breccia samples are scarce at Red Balloon rock shelter (Table 1). They are grey with no lustre. They were identified due to the presence of non-sorted large to fine quartz and calcite-rich phase cemented in a ferruginous matrix made up of randomly oriented micro-platy hematite. Specular hematite represents 69.2% of the assemblage ($N=142$), specularite 19% ($N=39$), ferruginous sandstones 10.7% ($N=22$) and ferruginised breccia 1% ($N=2$). No ferruginous shale, mudstone, siltstone or chert was identified within the Red Balloon rock shelter assemblage.

Utilised pieces

At Red Balloon rock shelter, nine ochre pieces present abrasion marks consisting of use-wear on faceted or non-faceted surfaces (Fig. 7).

Use-wear were identified on four hematite and three specularite pieces (Table 1), bigger than 10 mm and two specularite pieces smaller than 10 mm. Use-wear either covered large areas of faceted surfaces (Fig. 7A–D) or small areas of non-faceted surfaces (Fig. 7E–F). In most cases, use-wear was well oriented with parallel striations most easily visible close to fresh edges. Within the samples with use-wear patterns, some presented juxtaposed facets producing a pointed “crayon-like” shape (Fig. 7G and H). Two specularite samples bigger than 10 mm and two specularite pieces smaller than 10 mm presented a crayon-like morphology. Their facets are flat and striations are oriented in the direction of the main axis of the facet. The largest crayon-shaped ochre RB-OC-0998 presented use-wear at the top of tips (Fig. 7H). These crayon-like ochre pieces all come from layer Brown 3 (Table 1). Four ochre pieces with use-wear with only one facet come from layer Brown 3 and its associated hearth, one comes from Dark Reddish Grey (Table 1). In layer Brown 3 and its associated hearth, abrasion is thus present on 6 pieces bigger than 10 mm (9.2%). Polished and smoothed surfaces are present on two specularite pieces bigger than 10 mm. They were retrieved in layers Dark Yellowish Brown and Surface. However, they were not counted as bearing abrasion scars as they could well be of natural origin.

Most of the analysed archaeological samples ($N=121$, 59%) present percussion marks (Table 1). Most pieces only bear one type of modification ($N=124$), three samples bigger than 10 mm and two smaller than 10 mm (two crayon-shaped) presented both abrasion use-wears and percussion marks. The presence of use-wear grooves interrupted by a fractured edge with percussion marks and notches on samples RB-OC-997 and RB-OC-764 confirmed that the samples have been first abraded and then percussed (Fig. 7A, E and F and Fig. 8A and B).

Percussion marks consist of crushing marks close to fresh fractured edges, notches on the fractured edges and negative flakes scars (Fig. 8). Though most ochre samples found at Red Balloon are thinner than 2 cm, bipolar knapping was identified through the presence of opposite notches, percussion pits and negative flakes as evidenced for samples RB-OC-0997, RB-OC-2001 and RB-OC-0428 (Fig. 8A–G).

Percussion scars and bipolar percussion scars were identified on hematite, specularite and ferruginous sandstones from undisturbed MSA layers.

A few hard massive hematite bear percussion scars close to natural edges. These scars correspond to fresh edges, percussion pits, adhering flakes and notches (Fig. 8H). They could correspond to scars on a hammer or intentional percussion to knap small flakes or chunks.

Six ochre pieces, three hematite pieces and three specularite pieces might correspond to chunks with a shape similar to lithic cores (Fig. 9A, Table 1). Though some of them are still heavily coated with the post-depositional crust some negative flakes are still visible on several faces of these samples (Fig. 9A). One of these core-shaped ochre chunks was recovered in the IA layer Dark Reddish Grey, while the five others were recovered in MSA layers Dark Yellowish Brown ($N=3$), Brown 3 ($N=1$) and Very Dark Greyish Brown ($N=1$).

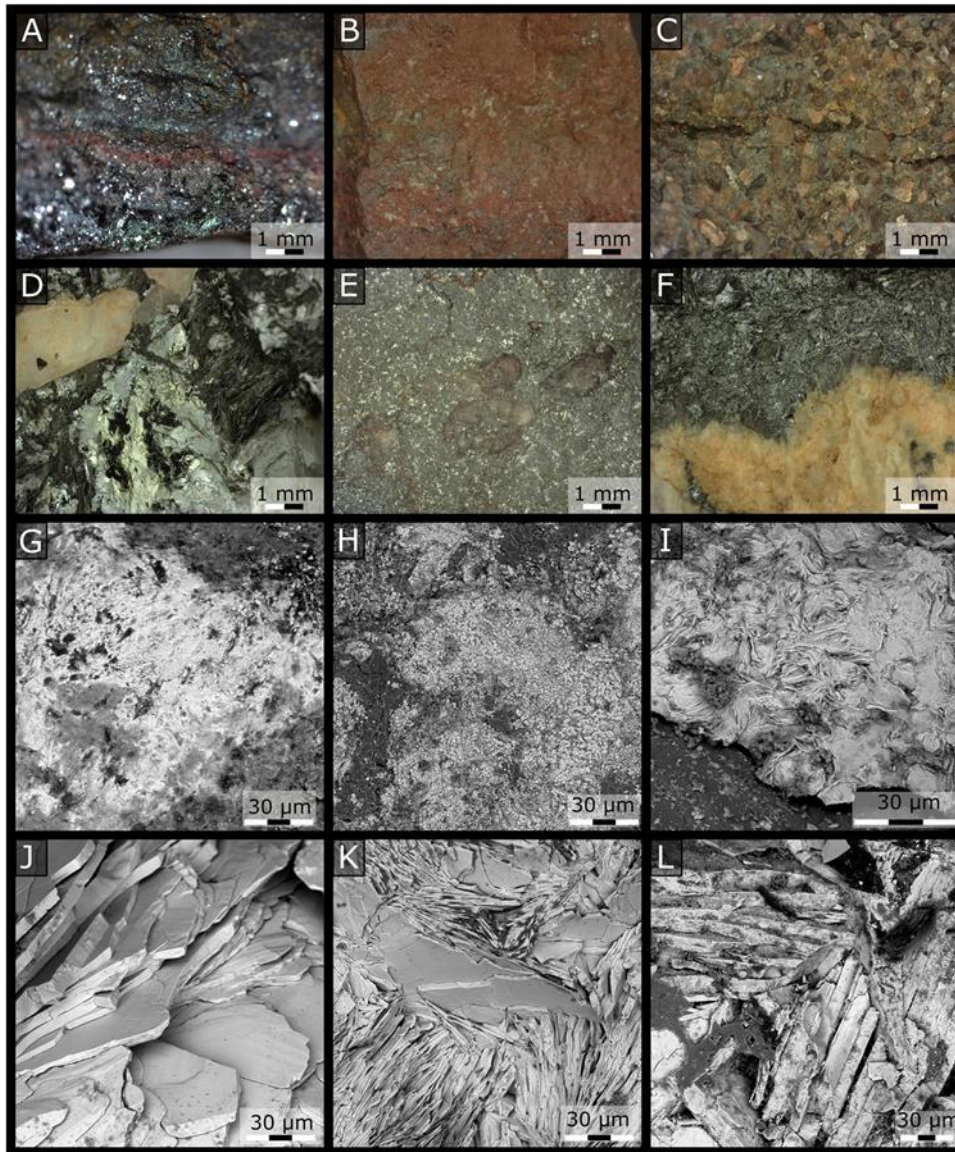


Fig. 6 The different MSA ochre types of Red Balloon rock shelter. Dinolite (A–F) and SEM (G–M) pictures of the different ochre found at Red Balloon. A, G Massive grey hematite RB-OC-797. B, H Microplaty grey hematite RB-OC-2121. C, I Ferruginous sandstone RB-OC-2001. D, J Specular hematite RB-OC-2028. E, K Fine specularite RB-OC- 2092. F, L Coarse specularite RB-OC-2085. All pictures were taken on fresh surfaces.

Together with these core-shaped ochre chunks, some ochre flakes were also recovered (Fig. 9B, C). These correspond to the same raw materials as the chunks: ferruginous sandstone, grey hematite, and specularite. Though the 254 pieces smaller than 10 mm were not classified according to their nature due to the heterogeneity of the ochre fragments, they all presented flaking scars consisting of flakes with the presence of a platform and ripples characteristic of flakes, or the presence of percussion pits and notches close to fresh edges.

Within MSA layers Brown 3 and Dark Yellowish Brown and their associated hearths, percussion was identified on more than 80% of the ochre pieces, while it is present on 77% of ochre pieces in Dark Olive Brown. Although percussion scars were identified on 121 ochre pieces, with some bearing some evidence pointing towards the use of bipolar percussion, no anvil and no hammer were retrieved within the excavated area of Red Balloon rock shelter.

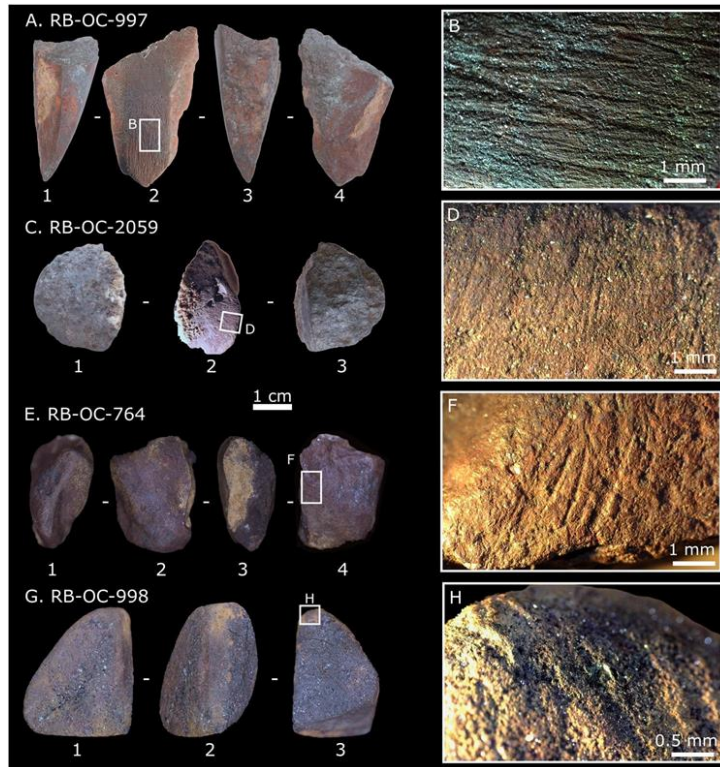


Fig. 7 Selected MSA ochre pieces from Red Balloon rock shelter with use-wear. A, C, E Ground and percussed pieces. G Crayon-like piece. B Use-wear on face 2 of A. D Use-wear on face 2 of C. F Use-wear on face 4 of E. H Use-wear on point of G.

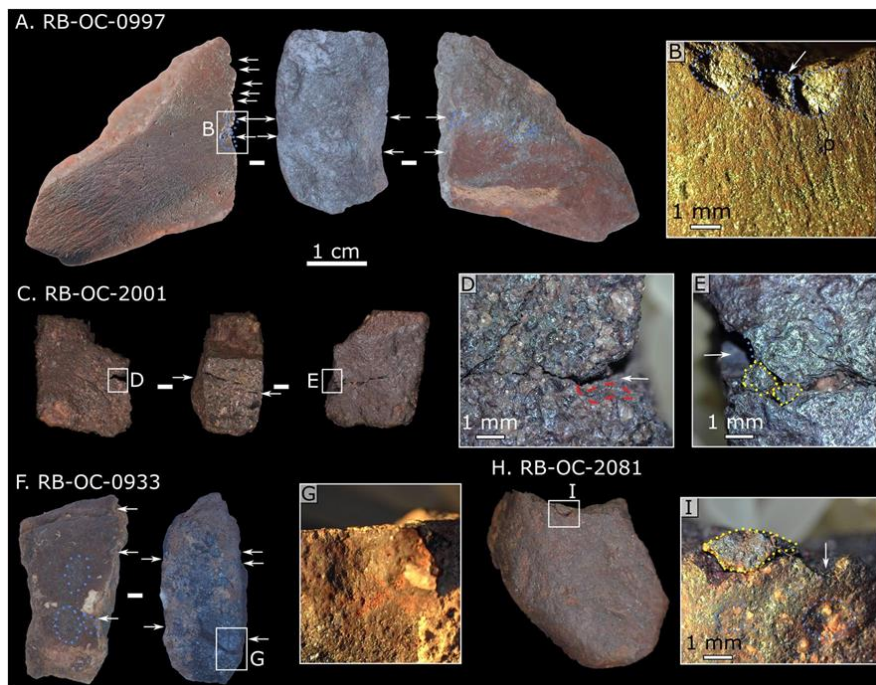


Fig. 8 Selected percussion marks on Red Balloon rock shelter ochre assemblage. Arrows point to notches, red lines mark flaking scar, blue ones pits, yellow ones adhering flakes. A, C and F present scars of bipolar percussion.

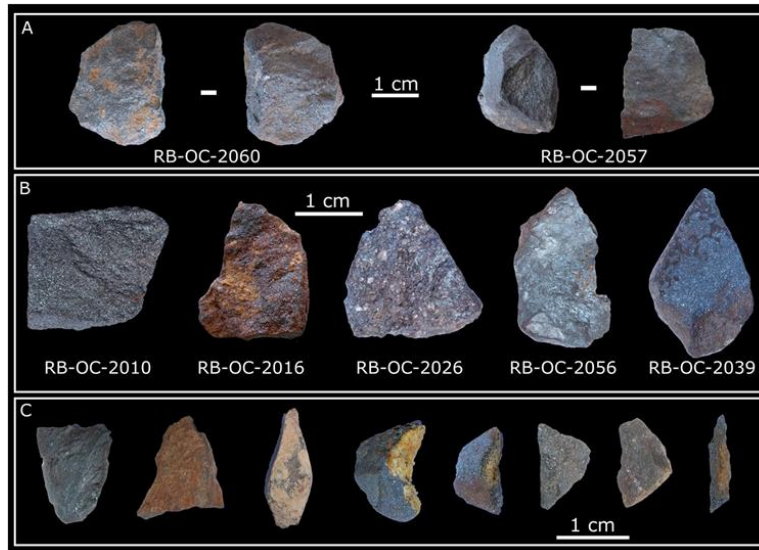


Fig. 9 A Selected core-shaped ochre chunks from Red Balloon rock shelter. B Selected ochre flakes from Redd Balloon Rock Shelter. C Unlabelled flakes smaller than 10 mm.

Stratigraphic and spatial distribution

Figure 10A and Table 1 sum up the stratigraphic distribution of the plotted ochre pieces along the Red Balloon rock shelter sequence. Though ochre appears all along the stratigraphy, they are more abundant in MSA layers (Fig. 10, Table 1).

IA layers yielded 82 (14.7% in number) ochre pieces (41 bigger than 10 mm and 41 smaller than 10 mm) weighing 475.1 g (16.4% in mass, Table 1, Fig. 10). Both the density and the mass density of the ochre pieces in IA layers are lower than in MSA layers due to the small numbers of ochre pieces and the large volume excavated.

MSA layers yielded 377 (82%) ochre pieces weighing around 2407 g (83.5%, Table 1; Fig. 10). Within these MSA layers, the distribution of the ochre pieces is not uniform. Dark Yellowish Brown, Brown 3 and Brown 3 hearth yielded the highest numbers and density of ochre fragments, while Very Dark Greyish Brown yielded the lowest number and density. This last layer yielded only a small volume (6 L of sediment). Thus, the mass density is the highest but cannot be compared directly to the others.

In terms of mass, Brown 3 and Dark Yellowish Brown presented the highest ochre mass with respectively 725.1 g and 637.8 g (Fig. 10). Mass density is the highest for Brown 3 and Brown 3 hearth around 25 g/L, the ochre mass density is lower for Dark Yellowish Brown and Dark Olive Brown respectively at 16.5 g/L and 16 g/L. For all IA layers, ochre mass density is lower than 5 g/L.

Small pieces (< 10 mm) constituted more than 50% of the assemblage for most MSA layers. In layers Dark Yellowish Brown Hearth, Dark Yellowish Brown, Brown 3 Hearth and Very Dark Olive Brown small pieces represent respectively 57.5%, 55.9%, 73.2% and 80.0% (Fig. 10, Table 1). In Brown 3 and Dark Olive Brown, they represent respectively 45.2% and 44.9% of the ochre assemblage recovered in these layers.

Both types of grey hematite, the most abundant ochre recovered at Red Balloon rock shelter, are the most abundant in Dark Yellowish Brown (N = 37) and Brown 3 (N = 31).

It is present in hearths associated with these layers and in Dark Olive Brown. In terms of density, it is most abundant in Brown 3 hearth. The three pieces of hematite recovered in Very Dark Greyish Brown account for 111 g, due to the presence of a large piece of hematite that has been worked. Specularite is the most abundant in Brown 3 (N = 11). It is also present in other MSA layers, except in Very Dark Greyish Brown. Ferruginous sandstones were recovered in all MSA layers but it is the most abundant in the Dark Reddish Grey Iron Age layer (N = 7). Breccia pieces were both recovered in MSA layers Dark Yellowish Brown and Brown 3 where ochre pieces are the most abundant.

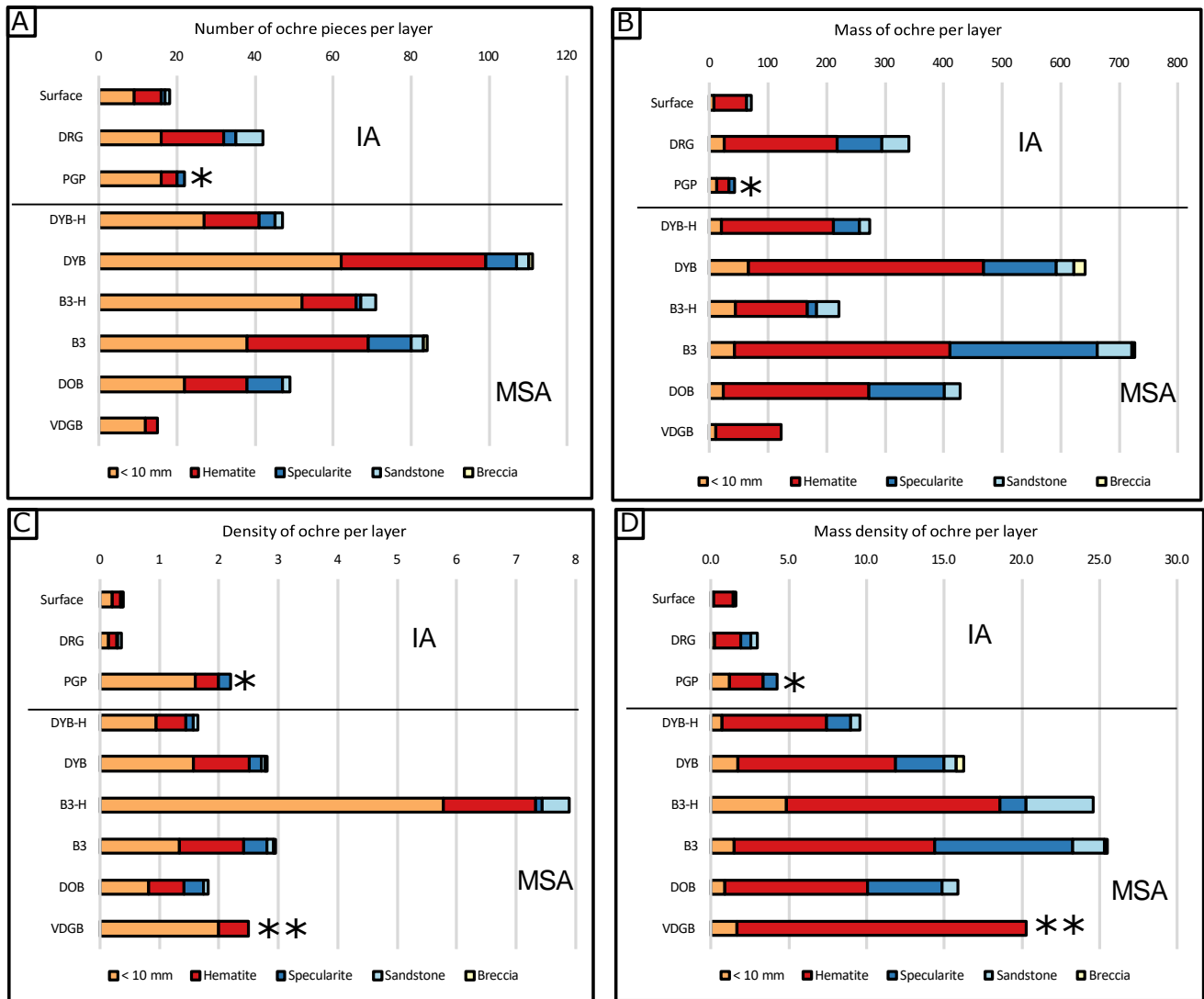


Fig. 10 Red Balloon ochre stratigraphic distribution. **A, B** Ochre number and mass stratigraphic distribution. **C, D** Ochre density and mass density in the stratigraphy. *Intrusive layers, **layer little excavated. DRG: Dark Reddish Grey, PGP: Pinkish Grey Pit, DYB-H: Dark Yellowish Brown Hearth, DYB: Dark Yellowish Brown, B3-H: Brown 3 Hearth, B3: Brown 3, DOB: Dark Olive Brown, VDGB: Very Dark Greyish Brown.

Thanks to the 3D recording of artefacts during the excavations, it was possible to map the ochre distribution within layers as illustrated in Fig. 11. Figure 11A plots the ochre distribution within all IA layers, and Fig. 11B and C plot the ochre distribution within the sole layers Dark Greyish Brown and Brown 3 respectively. SI 4 sums up the number of ochre pieces, their mean size, mean mass, mean number and frequency of percussed pieces in MSA layers Dark Greyish Brown and Brown 3.

Plotted IA ochre pieces appear to be scattered within all areas excavated with a slight concentration towards the IA pit (Pinkish Grey Pit, Fig. 11A). Thus, there is no evidence of intense ochre processing activity for this period.

Ochre pieces from the MSA layer Dark Yellowish Brown in D9 are highly concentrated in the northwestern corner of the excavation, next to the DYB hearth, while DYB ochre pieces recovered in E9 were scattered within the whole square (Fig. 11B). Though a similar number of ochre pieces larger than 10 mm was recovered within D9 (N = 29) and E9 (N = 23), a much greater number of ochre fragments smaller than 10 mm were recovered in D9 (N = 42) than E9 (N = 18, SI 4). 61% of ochre pieces larger than 10 mm recovered in E9 (N = 14) exhibit percussion marks, while there are only 42% of ochre pieces with percussion marks in D9 (N = 14). Few pieces were recovered in DYB Hearth, 20 bigger than 10 mm and 26 lower than 10 mm. Most of these pieces present percussion marks (70%, SI 4). Only one ochre piece from DYB was abraded. It was recovered within an area of ochre concentration next to the fire. Thus, it seems an ochre processing activity took place next to DYB hearth in the northwestern corner of the excavation. Pieces recovered in the hearth and E9 might thus correspond to percussion wastes. No ochre pieces

from this layer presented some striking evidence.

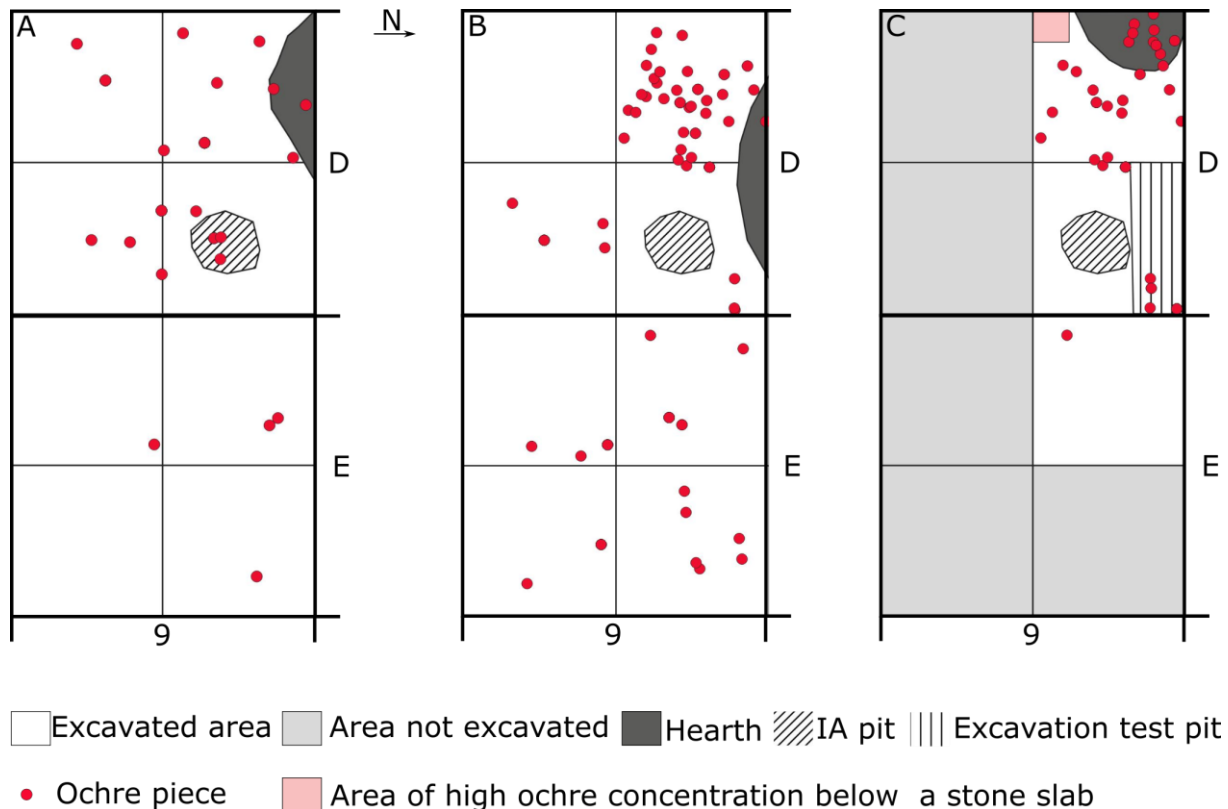


Fig.11 Red Balloon rock shelter ochre distribution map within Iron Age layers (A), MSA layer Dark Yellowish Brown (B) and MSA layer Brown 3 (C). Figure made with QGIS software.

Ochre pieces recovered from MSA layer Brown 3 also presented a high concentration towards the northwestern corner of the excavation next to the Brown 3 Hearth (Fig. 11C). A total of 114 ochre pieces were recovered from the area next to the fire (50 bigger than 10 mm, 64 smaller than 10 mm - SI 4). A total of 42 ochre pieces were recovered from the hearth. Ochre pieces recovered from the hearth are on average lighter than the ones recovered from Brown 3 (mean mass 7.8 g for hearth, 14.9 g for Brown 3 - SI 4).

Moreover, most ochre pieces recovered from the hearth exhibit percussion scars (93%, N = 14 from the 15 ochre pieces bigger than 10 mm). Ochre with percussion marks were also abundant from Brown 3 layers (62%, N = 31 from the 50 ochre pieces bigger than 10 mm). The six ochre pieces exhibiting abrasion use-wear and facets were all recovered from Brown 3 layer, next or under a flat stone under which was recovered a high concentration of ochre pieces. Pieces recovered in the vicinity of the hearth point towards the existence of an intense ochre processing activity towards the northwestern corner of the area excavated. There, the ochre pieces evidence the use of two different ways to process ochre were performed: abrasion and percussion. Due to the taphonomical crust that was covering all lithic pieces, it was not possible to ensure that lithic pieces presented any red residue on their surface.

Discussion

Taphonomy and representativity of the ochre assemblage

Most of Red Balloon rock shelter Middle Stone Age ochre assemblage was covered by a brown crust. Infrared analyses and SEM observations of this crust highlighted that it is a mixture of kaolinite clay and quartz (Fig. 4). The kaolinite clay and quartz mixture might be the result of the alteration of the sandstone cavity during a humid

period. These conditions might have then soaked the sediment deposit (Wadley et al. 2021). pXRF analyses of soil samples highlighted some compositional differences between layers. Naturally, combustion features exhibited higher Ca and Sr content from the heating of bone fragments (SI 2). pXRF analyses did not evidence the existence of elemental contamination within the Red Balloon rock shelter stratigraphic sequence. MSA layers also presented higher Fe contents than the IA layers. This higher Fe content might be due to the abundance of microscopic hematite fragments and clays in the MSA layers. Naturally, if a wet period existed, the clay and iron fragments resulting from previous ochre processing activities content would have been mixed. This is consistent with the identification of some small iron-rich fragments within the clayish matrix of the brown crust (Fig. 4).

The ochre pieces recovered at Red Balloon rock shelter are all grey materials producing a red to purple powder. No red chert, shale or any soft red ochre has been recovered at Red Balloon rock shelter. These materials are frequent in other South African MSA sites such as Sibudu Cave (Hodgskiss 2014), Rose Cottage (Hodgskiss 2013), Bushman Rock Shelter (Watts 2002) or Mwulu's Cave (Watts 2002; de la Peña et al. 2019). If soft red pieces would have been exploited at Red Balloon rock shelter, they most probably would have been washed out during the wet conditions and mixed with the clays within the brown crust. Thus, the brown crust would contain a large number of small red iron-rich fragments. Though we identified some iron-rich fragments mixed within the clay, they are not red but black. Moreover, microscopic observations of soil sediment only identified small black iron-rich fragments. Thus, we do not have any trace of the potential exploitation of red soft ochre materials at Red Balloon rock shelter. The absence of such materials can thus not be imputed to post-depositional processes. Red soft materials were not exploited by Middle Stone Age populations at Red Balloon rock shelter.

Specularite and specular hematite

Red Balloon rock shelter hematites and specularites are close one to another and are both high metallic hematitic ochre (Fig. 5). In his study of Oliebomspoort ochres, Watts (2002) identified specularite thanks to the identification of small "micaceous" particles randomly oriented, here I defined specularite as samples exhibiting a structure made of randomly oriented sutured platy euhedral hematite crystals of at least 100 μm length (Fig. 5). The randomly platy structure of these samples provides them with their typical small mirror-like particles.

It is here noteworthy to mention that though Red Balloon rock shelter specularite exhibits some mica-like crystals, there is no mica within Red Balloon specularite. The mica-like crystals are due to micro-crystalline platy hematite. As Watts did not carry out petrographic nor SEM observations, there is a high possibility that what he described as mica was micro-crystalline platy hematite randomly oriented as observed on Red Balloon rock shelter ochre.

Moreover, according to Watts (2002):

"Specularite sometimes grades into what we have designated "specular hematite," a more finely specular expression that cannot be described as glittery, although fresh exposures glisten in bright light."

As Watts did not identify specularite and specular hematite according to their structure but according to their surface aspect, and that patina can render their identification complex, the specularite and specular hematite identified by Watts (2002) and in the present collection do not concur. However, both Red Balloon rock shelter and Oliebomspoort assemblages are dominated by specularite and specular hematite which are both grey hematite nodules that glitter in sunlight. They could correspond to variations of the same material. As they exhibit different structures, their distinction is interesting for provenance studies, as ochre structures can be an important criterion for ochre sourcing (Pradeau et al. 2016).

Ochre processing at Red Balloon rock shelter

Although some of the Red Balloon ochre pieces were recovered in IA layers, the presence of an IA pit (Pinkish Grey Pit) that disturbed the MSA layers, the low weight and numbers of ochre pieces retrieved in the IA layers and their large scattering within these layers tend to support that ochre recovered in these layers are disturbed MSA materials (Figs. 10 and 11, Table 1).

Within Red Balloon rock shelter's undisturbed MSA ochre assemblage, there is no significative change in the ochre raw materials exploited, but it seems the way the ochre pieces were processed did not remain identical along the

MIS 5 occupations documented by the materials recovered in Dark Yellowish Brown, Brown 3 and Dark Olive Brown layers. Layers Dark Yellowish Brown and Brown 3 yielded numerous ochre pieces with percussion or abrasion evidence (Table 1, Figs. 6, 7 and 8). The spatial concentration of these worked ochre fragments within the northwest corner of the excavations supports an intense ochre processing activity at the site (Fig. 11). Two main techniques were used to pulverise ochre pieces, crushing and pounding by bipolar percussion and grinding, the latter being applied before the former when they took place within the same processing sequence.

Abrasion

Abrasion of ochre pieces is rare at Red Balloon rock shelter, as only nine pieces exhibit clear abrasion marks (Table 1, Fig. 6). Eight of the pieces are found in layer Brown 3 or its associated hearth, concentrating 89% of the abraded ochre pieces. Other layers are dominated by percussion. In Brown 3 layer of Red Balloon rock shelter, abrasion was identified on two specularite and four specular hematite pieces, representing 12% of the ochre from this layer. This is the usual percentage of abraded ochre pieces in MSA southern Africa sites (Hodgskiss 2012, 2013; Dayet et al. 2013, 2017).

Among the abraded pieces, one ochre has a crayon shape bearing use-wear traces on its tips, thus demonstrating its use as a crayon (Fig. 6). Though this ochre piece might have been used in symbolic activities as it has been suggested for a long time (Henshilwood et al. 2001; Watts 2002; d'Errico 2003), there is no evidence of such activities at Red Balloon rock shelter. Thus, the crayon-shaped and faceted ochre piece found at Red Balloon rock shelter can only be seen as the result of intense grinding activities as advocated by Wadley (2005a, b) for other MSA crayon-shaped ochres.

Although there is no direct trace of the use of ochre powder at the site, all these pieces with use-wear are indirect evidence of ochre processing to produce a colouring powder at Red Balloon rock shelter around 95 ka. As we do not have direct evidence of the use of these powders their use remains unknown. The use of time-costly abrasion and the production of crayon-shaped ochre reflect a need to save up some of the raw materials either for economical, technical or symbolic reasons. Such ochre raw materials might have been considered precious either because rare in the region, or hard to collect because of the accessibility of their outcrop or for the beliefs linked to its outcrop. It is also possible that the use the production of the powder had to be performed in a certain way for technical or social reasons. If such abraded and crayon-shaped would have been precious, it would be interesting to investigate whether they were procured from different outcrops than the percussed pieces.

The almost absence of abraded ochre pieces in other layers might be due to the occupation of the site by different populations or the existence of contact between populations of Red Balloon rock shelter with other sites out of the period at the time of deposition of layer Brown 3.

Percussion

Most use marks on ochre pieces from Red Balloon rock shelter correspond to percussion marks as illustrated in Fig. 7. Together with the chunks, flakes and the small size of the ochre pieces retrieved at Red Balloon rock shelter (Figs. 8 and 9), it supports an intensive processing of ochre pieces through percussion at the site. It is possible that different percussion modes were used, such as knapping or bipolar percussion. The use of different percussion modes is difficult to test as most ochre pieces are thinner than 2 cm. As evidenced in the exploratory experimentation, such small samples can easily be pulverised without leaving evident percussion stigmata on the remaining flakes and chunks (SI 2).

Bipolar percussion was identified thanks to the presence of opposite percussion pits and notches on fresh fracture surfaces (Fig. 7). Its identification is only possible on ochre pieces preserving scars produced by the hammer and the anvil. Bipolar percussion was probably intensively used but only rare fragments have preserved diagnostic stigmata.

Some of the hard hematite pieces could also have been used as hard hammers to percuss other ochre pieces. In their study of the Sibudu backed piece, Soriano et al. (2009) suggested that the red residues present on the lithic tools might correspond to the use of ochre as a soft hammer. At Red Balloon, it is not possible to investigate if the lithic bear red residues on their surface that might point toward a similar use of an ochre hammer. But some of the hard hematite fragments present percussion marks that could be coherent with their use as hard hammers to either knap some lithic tools or percuss some softer ochre.

Provenance considerations

In the Waterberg, there are numerous potential ochre sources: (1) faults, (2) iron nodules of the Mogalakwena Formation, (3) Thabazimbi formations, (4) contact between diabase and sandstones in the Eastern part of the Waterberg. Materials recovered at faults are of hydrothermal origin. The iron nodules entrapped in the Mogalakwena correspond to the modern ochre used in the experiments presented in this article. They are massive hematite samples with some rare quartz inclusions. Thabazimbi hematite is currently being mined extensively. It is massive with no microcrystals. Finally, though the contact between the diabase and the sandstone in the eastern part of the Waterberg could be a source of ochre, no hematite nor specularite has yet been reported there.

Most of the ochre retrieved at Red Balloon could be of hydrothermal origin and could be coming from the faults in the Waterberg. According to Maria van der Ryst (2007), hematite and specularite are commonly found at such sources in the Waterberg. Such faults are found less than 5 km from the site of Red Balloon rock shelter. Future works will need to investigate Red Balloon rock shelter ochre provenance.

Recent ochre studies have also suggested the existence of long-distance ochre procurement during the European Palaeolithic (Velliky et al. 2021) and southern African Later Stone Age (Mauran et al. 2021). Such provenance studies would also contribute to improving our understanding of why some ochre pieces have been intensively abraded while most of the fragments have been percussed. If abraded ochre pieces had been circulated over long distances, sourcing them might even open a new horizon to ochre provenance studies to question whether material circulated together thanks to populations mobility or trade networks as it sometimes performed with lithic raw materials (e.g. Tomasso 2018).

Population adaptation to local resources?

Though grinding is an easy processing technique for soft ochre materials, the preliminary experiments presented in SI 2 support that percussion is an effective expeditive way to pulverise hard ochre pieces such as hard specular hematite (Dayet et al. 2013; Hodgskiss 2014; Rosso et al. 2016; Mauran et al. 2020). The abundance of percussion marks, the presence of expeditious chunks, and the numerous ochre flakes document past population know-how to process hard hematite and specularite effectively (Table 1, Figs. 7 and 8). Percussion marks are so far little investigated on ochre pieces. Bipolar percussion was identified at Klasies River main site (Dayet et al. 2017) and in Ethiopia at Porc-Epic (Rosso et al. 2016) or in younger LSA assemblages in Namibia (Mauran et al. 2020). At Diepkloof Rock Shelter, Dayet and colleagues (Dayet et al. 2013) record little use of percussion to fracture ochre pieces. They impute this to the selection of small ochre nodules. These ochre pieces are shales and ferricretes, softer than the specular and some of the specularite found at Red Balloon rock shelter.

Interestingly, most ochre pieces found at the site are thinner than 2 cm. As documented during the experimentation presented in this study, bipolar percussion of such ochre pieces leaves scarce traces as fragmentation is immediate and the powder is similar to the one obtained through abrasion, making bipolar percussion of such ochre pieces a very efficient processing way to produce ochre powder from hard material as hematite. It seems MSA populations who occupied the site around 95 ka had this knowledge and selected preferentially relatively thin ochre pieces. Such an ochre selection could correspond to a cultural choice to procure preferentially small ochre pieces.

The percussion of numerous hard specular hematite and specularite of potential local origin seems to point out that MSA populations who occupied Red Balloon rock shelter adapted to the local material. If local ochre sources are abundant and provide numerous specular hematite and specularite, as nowadays (van der Ryst 2007), then past populations did not have to bother about the accessibility of ochre raw materials. They did not require optimizing ochre exploitation and could even produce more ochre powder than required. In such conditions, past populations would not need to spend time and energy to ensure the reusability of ochre pieces. The use of percussion would then be efficient to produce ochre powder for everyday life. The loss of small ochre fragments would not constitute a big loss as raw materials would be of high accessibility. Thus, the use of bipolar percussion on local materials could testify to a period of ochre abundance with little exchange and interactions with populations out of the Waterberg.

Specificities of the assemblages from the Waterberg within the southern African MSA context

In the Waterberg, other MSA archaeological ochre assemblages are similar to what has been found at Red Balloon rock shelter. They all correspond to highly metallic grey to black hematite samples producing a red-to-purple powder. Two MSA sites in the Waterberg yielded some ochre assemblages: North Brabant and Olieboomspoot. North Brabant was excavated by Schoonraad and Beaumont (1968). A large part of the MSA assemblage was mixed with IA materials. But all ochre retrieved at North Brabant is referred to by the authors as “maroon hematite”, or specularite. Due to the mixing of the MSA and IA layers, the MSA deposit was not dated. The most well-known site of the Waterberg is Olieboomspoot, 10 km north of Red Balloon rock shelter. There, LSA ($N = 9474$) and MSA ($N = 756$) ochre assemblages are composed of heavy, hard specularite or haematite similar to what has been found at Red Balloon rock shelter (Watts 1998, 2002; Val et al. 2021). The MSA deposit was dated around 150 ka (Val et al. 2021). Ian Watts who studied the MSA ochre recovered by Mason ($N = 304$), suggested Olieboomspoot to be a primary processing site. According to him, the local raw materials were processed there and later transported thanks to regional exchange networks (Watts 1998: p.706). There is yet no evidence of such regional networks. Several specular hematites and specularite prehistoric mines have been documented in southern Africa (Thackeray et al. 1983; Kiehn et al. 2007; Robbins 2016; Watts et al. 2016). Though most are dated to LSA and IA, they document the existence of specular hematite and specularite in different parts of southern Africa.

These assemblages strike out of the rest of the South African MSA ochre assemblages. In the Waterberg, the ochre assemblages are composed of hard metallic specularite and hematite while outside of the Waterberg, in the Limpopo region, the sites of Mwulu’s Cave and Bushman Rock Shelter yielded very different ochre assemblages. At Bushman Rock Shelter, the ochre assemblage is dominated by shales, mudstones and silts. Hematite and specularite represent 32.7% of the ochre assemblage described by Watts (2002) but the main ochre raw materials are red ferruginous shale. Recent re-examinations of the Bushman Rock Shelter collection and examination of ochre recovered during recent excavations have been performed by Porraz and colleagues (Porraz et al. 2015). They mention the presence of massive hematite, lateritic crusts, shale, mudstone and siltstone. This new examination of the Bushman Rock Shelter ochre assemblage might bring some further understanding of the raw materials selected by past populations who occupied the site. Percentages provided by Watts (2002) are thus to be considered as preliminary results, as new trends could be identified.

At Mwulu’s Cave, dated around 90 ka (Feathers et al. 2020), the ochre assemblage is rather small as only 200 ochre pieces have been recovered. Moreover, there is a striking difference between the ochre assemblage recovered by Tobias and the one recovered by de la Peña. While Tobias recovered 13 ochre pieces with specularite and hematite, de la Peña only recovered 4 hematite and no specularite but recovered 137 small fragments of ferruginous shale (de la Peña et al. 2019). Tobias’ ochres are larger and heavier than de la Peña’s ochre pieces. Thus, it seems Tobias only collected the most impressive ochre pieces leaving aside most of the smaller fragments, creating a major bias in the ochre assemblage he recovered (de la Peña et al. 2019). Thus, specularite and hematite are present but they are not predominant at Mwulu’s Cave. These specularites and hematites are polished, faceted or even crayon-shaped they might represent valuable scarce resources possibly coming from the Waterberg.

Out of the Waterberg, in the East, one piece of extensively ground specularite was present in the MSA context of Rhino Cave, Tsodilo Hills, Botswana (Robbins et al. 1998; Robbins and Murphy 2011). There, specularite was mined extensively during the Later Stone Age and Iron Age. Though only one piece was found in the MSA context, Robbins and Murphy (2011) advocate for an early exploitation of specularite around 95 ka.

Outside the Waterberg, hard iron oxides, including hematite, only represent 5.9% ($N = 319$) of all ochres at Sibudu Cave (Hodgskiss 2012), 4.3% ($N = 21$) of all ochres at Rose Cottage (Hodgskiss and Wadley 2017), 1.7% Umhlatuzana (Watts 2002).

These differences in the type of ochre exploited in the Waterberg compared to other South African sites is imputable to the population’s adaptation to the local Waterberg ecosystem and geology. This region is highly metalliferous, hard grey hematite and specularite are common (Beukes et al. 2003). However, it is interesting to see that Mwulu’s Cave and Bushman Rock shelter, also highly metalliferous yielded some hematite and specularite but in significantly lower proportions than the sites of the Waterberg. This might be due to the presence of more

diverse sources close to these sites. Materials from these other sources were not transported to the MSA sites of the Waterberg. Such regional conventions existed during the Howiesons Poort close to the coast as demonstrated by Dayet and colleagues (Dayet et al. 2017). It seems populations who occupied the Waterberg found specular hematite and specularite, common in local geology, as good ochre raw materials, pointing to the existence of a regional Waterberg convention to exploit local materials possibly of hydrothermal origin.

A preliminary study highlighted that at Red Balloon rock shelter among the 119 MSA cores, 54 are bipolar. According to Mason (1962: 273), bipolar knapping is well represented in Bed 2 of Olieboomspoort through the identification of *outil écaillé*. Bipolar knapping is not reported in contemporary layers in the East of the Waterberg at Cave of Hearths, Mwulu's Cave or Bushman Rock Shelter (Porráz et al. 2018; de la Peña et al. 2019). The extensive use of bipolar knapping and the exploitation of similar specular hematite and specularite could point to the existence of regional mineral preferences during Marine Isotope Stage 5. This could be imputed to the populations' adaptations to local raw materials and the development of a local tradition in the techniques used to work mineral resources as suggested by Wadley et al. (2021).

The preliminary study of the lithic assemblage recovered at Red Balloon rock shelter also recorded the presence of Levallois points (Wadley et al. 2021). Such points are found at the contemporary site of Mwulu's Cave (de la Peña et al. 2019). However, they are absent from the Cave of Hearths assemblage and the 90-100 ka layers of Bushman Rock Shelter (Porráz et al. 2015, 2018). At this last site, Levallois technology is only identified in the deepest layers. The presence of such Levallois points and specularite crayons at both Red Balloon rock shelter and Mwulu's Cave might be the result of some punctual interactions between populations who occupied the Waterberg and Makapan valley. Future ochre studies of Waterberg and surrounding sites should investigate the provenance of these raw materials to investigate whether some interactions existed or not. Ochre studies in the Waterberg should also question whether MSA populations of the Waterberg exploited local ochre materials and if the different potential sources of the Waterberg were all exploited. Moreover, they should question whether bipolar percussion was used extensively in this region of southern Africa. If so, it seems MSA populations of the Waterberg might have developed some technological traditions adapted to the local resources of the Waterberg more than 95 ka.

Conclusion

In this article, a terminology for percussion marks found on these materials was suggested. The description of the ochre assemblage recovered at Red Balloon rock shelter (Waterberg, South Africa) includes a description of the post-depositional processes it suffered from, the nature of the ochre assemblage and the traces of exploitation present on the ochre assemblage. Finally, the implications of the ochre assemblage for past populations who inhabited the Waterberg during the Marine Isotope Stage 5 are discussed.

The ochre percussion experimentation should be repeated with systematic experiments investigating the variability of percussion marks on different ochre raw materials and the influence of the percutor and operator processing the ochre material.

Future studies should also include more percussion modes. But the experiments presented here, allowed to suggest a terminology to refer to percussion marks found on archaeological ochre assemblages. This terminology based on the one suggested by Vettese and colleagues (Vettese et al. 2020) for bones, paves the way for further studies aiming at better documenting ochre percussion on ochre assemblages and the potential existence of technical traditions in the way these materials were processed. This terminology was first used in the present study on the ochre assemblage of Red Balloon rock shelter.

Red Balloon Rock Shelter ochre assemblage is an MIS 5 Middle Stone Age assemblage composed of four ochre types: hematite, specularite, ferruginous sandstone and ferruginous breccia, all grey materials producing a red to purple powder. Traces of anthropic modifications take the form of use-wear patterns, faceted pieces, and percussion marks. These percussion marks are similar to the ones observed on similar modern ochre pieces percussed by bipolar percussion. Though there is no direct evidence of ochre powder use at Red Balloon rock

shelter, the presence of abraded and percussed ochre pieces constitutes indirect evidence of the production of ochre powder around 95 ka. The concentration of the ochre processing activity in the northwestern corner of the excavation seems to point to the spatial organization of the site at this period. The use of both time-costly abrasion and expeditious percussion to process the ochre pieces might point to a difference in the economic and cultural value of the ochre raw material. Abraded pieces might have been considered precious material. Thus, future work will have to investigate the provenance of the ochre pieces to investigate whether the ochre pieces are of local provenance or not and if abraded pieces might have been procured from different outcrops than the percussed ones.

The exploitation of specular hematite and specularite and their intensive exploitation by percussion, possibly bipolar percussion, is similar to what has been identified at the nearby site of Olieboomspoor but considerably differs from what has been found in other MSA South African sites. It thus seems to point to the adaptation of MSA populations to local resources. This paves the way to question whether a tradition adapted to local resources developed in the Waterberg during Marine Isotope Stage 5. The to-be-confirmed existence of such a tradition in the interior of southern Africa around 95 ka raises multiple questions regarding the emergence of multiple key innovations of the MSA. With the existence of different traditions in coastal and interior areas, one can wonder whether MSA innovations are the results of an increase in social-cultural interactions between different cultural groups.

Supplementary information

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Author contributions

G.M. designed the study, wrote the main manuscript, prepared all figures and reviewed the manuscript.

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Data availability

The data that support the findings of this study are available from the corresponding author, GM, upon request.

Declarations

Ethics approval Not applicable.

Consent to participate All authors consented to participate.

Consent for publication All authors consented to the publication.

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References

- Audouin F, Plisson H (1982) Les ochres et leurs témoins au Paléolithique en France: Enquête et expériences sur leur validité archéologique. *Cahiers Du Centre De Recherches Préhistoriques* 8:33–80
- Backwell LR, McCarthy TS, Wadley L, Henderson Z, Steininger CM, de Klerk B, Barré M, Lamothe M, Chase BC, Woodborne S, Susino GJ, Bamford MK, Sievers C, Brink JS, Rossouw L, Pollarolo L, Trower G, Scott L, d'Errico F (2014) Multiproxy record of late Quaternary climate change and Middle Stone Age occupation at Wonderkrater. *South Africa Quat Sci Rev* 99:42–59. <https://doi.org/10.1016/j.quascirev.2014.06.017>
- Barham L (2002) Systematic pigment use in the Middle Pleistocene of south central Africa. *Curr Anthropol* 43:181–190. <https://doi.org/10.1086/338292>
- Beaumont P (1973) The ancient pigment mines of Southern Africa. *S Afr J Sci* 69:140–146
- Bernatchez J (2013) The role of ochre in the development of modern human behaviour: a case study from South Africa. *Azania: Archaeol Res Africa* 48(1) 147–147
- Beukes N, Gutzmer J, Mukhopadhyay J (2003) The geology and genesis of high-grade hematite iron ore deposits. *Appl Earth Sci* 112:18–25. <https://doi.org/10.1179/037174503225011243>
- Bleek WHI, Lloyd L, Theal GM (1911) Specimens of Bushman folklore. G Allen, Limited
- Charrié-Duhaut A, Porraz G, Cartwright CR, Igreja M, Connan J, Poggenpoel C, Texier P-J (2013) First molecular identification of a hafting adhesive in the Late Howiesons Poort at Diepkloof Rock Shelter (Western Cape South Africa). *J Archaeol Sci* 40(9):3506–3518. <https://doi.org/10.1016/j.jas.2012.12.026>
- Clark DJ, Brown KS (2001) The Twin Rivers Kopje, Zambia: stratigraphy, fauna, and artefact assemblages from the 1954 and 1956 excavations. *J Archaeol Sci* 28:305–330. <https://doi.org/10.1006/jasc.2000.0563>
- Corcoran PL, Bumby AJ, Davis DW (2013) The Paleoproterozoic Waterberg Group, South Africa: provenance and its relation to the timing of the Limpopo orogeny. *Precambr Res* 230:45–60. <https://doi.org/10.1016/j.precamres.2012.12.009>
- d'Errico F (2003) The invisible frontier: a multiple species model for the origin of behavioral modernity. *Evol Anthropol* 12:188–202. <https://doi.org/10.1002/evan.10113>
- d'Errico F (2008) Le rouge et le noir: Implications of early pigment use in Africa, the Near East and Europe for the origin of cultural modernity. *South African Archaeological Society Goodwin Series* 10:168–174. <https://www.jstor.org/stable/40650028>. Accessed 4 May 2023
- Dapschaskas R, Göden MB, Sommer C, Kandel AW (2022) The emergence of habitual ochre use in Africa and its significance for the development of ritual behavior during the Middle Stone Age. *J World Prehist* 35:233–319. <https://doi.org/10.1007/s10963-022-09170-2>
- Dart RA (1975) The debt of palaeontology to haematite. *J Palaeontol Soc India* 20:205–210
- Dart RA, Beaumont P (1969) Evidence of iron ore mining in southern African in the Middle Stone Age. *Curr Anthropol* 10(1):127–128. <https://doi.org/10.1086/201014>
- Dayet L, Texier P-J, Daniel F, Porraz G (2013) Ochre resources from the Middle Stone Age sequence of Diepkloof Rock Shelter, Western Cape, South Africa. *J Archaeol Sci* 40(9):3492–3505. <https://doi.org/10.1016/j.jas.2013.01.025>
- Dayet L, Le Bourdonnec F-X, Daniel F, Porraz G, Texier P-J (2016) Ochre provenance and procurement strategies during the middle stone age at Diepkloof Rock Shelter. *South Africa Archaeometry* 58(5):807–829. <https://doi.org/10.1111/arcm.12202>
- Dayet L, Wurz S, Daniel F (2017) Ochre resources, behavioral complexity and regional patterns in the Howiesons Poort: new insights from Klasies River Main Site, South Africa. *J Afr Archaeol* 15(1):20–41. <https://doi.org/10.1163/21915784-12340002>
- Dayet L (2012) Matériaux, transformations et fonctions de l'ocre au Middle Stone Age: le cas de Diepkloof Rock Shelter dans le contexte de l'Afrique australe. Ph.D. dissertation. Université de Bordeaux I, Bordeaux
- de la Peña P, Val A, Stratford D et al (2019) Revisiting Mwulu's Cave: new insights into the Middle Stone Age in the Southern African savanna biome. *Archaeol Anthropol Sci* 11:3239–3266. <https://doi.org/10.1007/s12520-018-0749-9>
- de Lumley H (1966) Les fouilles de Terra Amata à Nice: premiers résultats. *Bulletin Du Musée D'anthropologie Préhistorique De Monaco* 13:29–51
- Distler VV, Yudovskaya MA, Prokofev VY et al (2000) Hydrothermal platinum mineralization of the Waterberg deposit (Transvaal, South Africa). *Geol Ore Deposits* 42(4):328–339
- Ericsson PG, Reczko BFF, Callaghan CC (1997) The economic mineral potential of the mid-Proterozoic Waterberg Group, northwestern Kaapvaal Craton, South Africa. *Miner Deposita* 32:401–409. <https://doi.org/10.1007/s001260050106>
- Feathers JK, Evans M, Stratford DJ, de la Peña P (2020) Exploring complexity in luminescence dating of quartz and feldspars at the Middle Stone Age site of Mwulu's Cave (Limpopo, South Africa). *Quaternary Geochronology* 59:101092. <https://doi.org/10.1016/j.quageo.2020.101092>
- Havenga D, Akoba R, Menzi L et al (2022) From Himba indigenous knowledge to engineered Fe₂O₃ UV-blocking green nanocosmetics. *Sci Rep* 12(1):1–10. <https://doi.org/10.1038/s41598-021-04663-0>
- Henshilwood CS, Marean CW (2003) The origin of modern human behavior: critique of the models and their test implications. *Curr Anthropol* 44(5):627–651. <https://doi.org/10.1086/377665>
- Henshilwood CS, d'Errico F, Watts I (2009) Engraved ochres from the Middle Stone Age levels at Blombos Cave, South Africa. *J Hum Evol* 57(1):27–47. <https://doi.org/10.1016/j.jhevol.2009.01.005>
- Henshilwood CS, d'Errico F, van Niekerk KL et al (2011) A 100,000-year-old ochre-processing workshop at Blombos Cave, South Africa. *Science* 334:219–222. <https://doi.org/10.1126/science.1211535>
- Henshilwood CS, d'Errico F, Niekerk KL et al (2018) An abstract drawing from the 73,000-year-old levels at Blombos Cave, South Africa. *Nature* 562:115–118. <https://doi.org/10.1038/s41586-018-0514-3>
- Henshilwood CS, Sealy JC, Yates R, Cruz-Urbe K, Goldberg P, Grine FE, Klein RG, Poggenpoel C, van Niekerk K, Watts I (2001) Blombos cave southern cape south africa: preliminary report on the 1992–1999 excavations of the middle stone age levels. *J*

- Archaeol Sci 28(4):421–448. <https://doi.org/10.1006/jasc.2000.0638>
- Hodgskiss T (2010) Identifying grinding, scoring and rubbing use- wear on experimental ochre pieces. *J Archaeol Sci* 37(12):3344–3353. <https://doi.org/10.1016/j.jas.2010.08.003>
- Hodgskiss T (2013) Ochre use in the Middle Stone Age at Sibudu, South Africa: grinding, rubbing, scoring and engraving. *Journal of African Archaeology* 11:75–95. <https://doi.org/10.3213/2191-5784-10232>
- Hodgskiss T, Wadley L (2017) How people used ochre at Rose Cottage Cave, South Africa: sixty thousand years of evidence from the Middle Stone Age. *PLoS One* 12(4):e0176317. <https://doi.org/10.1371/journal.pone.0176317>
- Hodgskiss T (2012) An investigation into the properties of the ochre from Sibudu, KwaZulu-Natal, South Africa. *Southern African Humanities* 24:99–120. <https://hdl.handle.net/10520/EJC127432>. Accessed 4 May 2023
- Hodgskiss T (2014) Ochre use at Sibudu Cave and its link to complex cognition in the Middle Stone Age. *Azania: Archaeol Res Africa* 49(4) 544–545. <https://doi.org/10.1080/0067270X.2014.939451>
- How, M. W. (1962). *The mountain bushmen of Basutoland*. Pretoria: JL van Schaik
- Kiehn AV, Brook GA, Glascock MD et al (2007) Fingerprinting specular hematite from mines in Botswana, Southern Africa. In: Glascock, M.D., Speakman, R.J., Popelka-Filcoff, R.S. (Eds.), *Archaeological Chemistry: Analytical Techniques and Archaeological Interpretation*, ACS Symposium Series No. 968. American Chemical Society, Washington DC, 460–479
- Laue GB (2001) *The Rock Art of Kurumakatiti Game Reserve*. Report in the Rock Art Research Institute, University of the Witwatersrand
- Lombard M (2007) The gripping nature of ochre: the association of ochre with Howiesons Poort adhesives and Later Stone Age mastics from South Africa. *J Hum Evol* 53(4):406–419. <https://doi.org/10.1016/j.jhevol.2007.05.004>
- Lombard M, Wadley L, Deacon J et al (2012) South African and Lesotho stone Age sequence updated (I). *South Afr Archaeol Bull* 67(195):123–144
- Lombard M (2006) Direct evidence for the use of ochre in the hafting technology of Middle Stone Age tools from Sibudu Cave. *South Afr Humanit* 18(1):57–67. <https://hdl.handle.net/10520/EJC84771>. Accessed 4 May 2023
- de Lumley H, Audubert F, Khatib S et al (2016) Les crayons d'ocre du site acheuléen de Terra Amata. In: de Lumley HF (ed) *Terra Amata: Nice, Alpes-Maritimes, France, Comportement et mode de vie des chasseurs acheuléens de Terra Amata*, vol 5. CNRS. Paris, p 536
- de Lumley-Woodyear H (1969) *Le Paléolithique inférieur et moyen du Midi méditerranéen dans son cadre géologique*. In: Tome I (ed) *Ligurie-Provence*. Gallia Préhistoire supp. 5. CNRS, Paris
- Mason R (1962) *Prehistory of the Transvaal*. Witwatersrand University Press, Johannesburg
- Mauran G, Lebon M, Lapauze O et al (2020) Archaeological ochres of the rock art site of Leopard Cave (Erongo, Namibia): Looking for Later Stone Age sociocultural behaviors. *Afr Archaeol Rev* 37(4):527–550. <https://doi.org/10.1007/s10437-020-09394-7>
- Mauran G, Caron B, Détroit F et al (2021) Data pretreatment and multivariate analyses for ochre sourcing: Application to Leopard Cave (Erongo, Namibia). *J Archaeol Sci: Rep* 35:102757. <https://doi.org/10.1016/j.jasrep.2020.102757>
- McBrearty S, Brooks AS (2000) The revolution that wasn't: a new interpretation of the origin of modern human behavior. *J Hum Evol* 39(5):453–563. <https://doi.org/10.1006/jhev.2000.0435>
- McGrath JR, MacDonald BL, Stalla D (2022) Middle Stone Age mineral pigment procurement at Pinnacle Point 5–6 North, Western Cape province, South Africa. *Archaeometry* 64(1):193–217. <https://doi.org/10.1111/arc.12694>
- Oberthür T, Melcher F, Fusswinkel T, van den Kerkhof AM, Sosa GM (2018) The hydrothermal Waterberg platinum deposit, Mookgophong (Naboomspruit), South Africa. Part 1: geochemistry and ore mineralogy. *Mineral Mag* 82(3):725–749. <https://doi.org/10.1180/minmag.2017.081.073>
- Perlès C (1987) *Les industries lithiques taillées de Franchthi (Argolide, Grèce) I, Présentation Générale et Industries Paléolithiques*. Excavations at the Franchthi cave, Fascicle 3. Excavations at Franchthi Cave–Greece-. Indiana University Press, Bloomington, Indianapolis
- Popelka-Filcoff RS, Zipkin AM (2022) The archaeometry of ochre sensu lato: a review. *J Archaeol Sci* 137:105530. <https://doi.org/10.1016/j.jas.2021.105530>
- Porraz G, Val A, Dayet L et al (2015) Bushman Rock Shelter (Limpopo, South Africa): a perspective from the edge of the Highveld. *South Afr Archaeol Bull* 70(202):166–179
- Porraz G, Val A, Tribolo C et al (2018) The MIS5 Pietersburg at '28' Bushman Rock Shelter, Limpopo Province, South Africa. *PLoS One* 13(10):e0202853. <https://doi.org/10.1371/journal.pone.0202853>
- Pradeau JV, Binder D, Vérati C et al (2016) Procurement strategies of Neolithic colouring materials: Territoriality and networks from 6th to 5th millennia BCE in North-Western Mediterranean. *J Archaeol Sci* 71:10–23. <https://doi.org/10.1016/j.jas.2016.05.007>
- Rifkin R (2011) Assessing the efficacy of red ochre as a prehistoric hide tanning ingredient. *J Afr Archaeol* 9(2):131–158. <https://doi.org/10.3213/2191-5784-10199>
- Rifkin RF, Dayet L, Queffelec A et al (2015) Evaluating the photoprotective effects of ochre on human skin by in vivo SPF assessment: implications for human evolution, adaptation and dispersal. *PLoS One* 10(9):e0136090. <https://doi.org/10.1371/journal.pone.0136090>
- Rifkin R (2015) Ethnographic and experimental perspectives on the efficacy of red ochre as a mosquito repellent. *South Afr Archaeol* 70:64–75. <https://www.jstor.org/stable/24643609>. Accessed 4 May 2023
- Robbins LH, Murphy ML (2011) An overview of the Later and Middle Stone Age at Tsodilo Hills. *Botsw Notes Rec* 43:130–139
- Robbins LH (2016) "Sebilo": 19th century hairdos and ancient specularite mining in Southern Africa. *Int J Afr Hist Stud* 49(1):103–131. <https://www.jstor.org/stable/44715444>. Accessed 4 May 2023
- Robbins LH, Murphy ML, Campbell AC, Brook GA (1998) Intensive mining of specular hematite in the Kalahari ca. AD 800–1000. *Curr Anthropol* 39(1):144–150
- Roebroeks W, Sier MJ, Nielsen K et al (2012) Use of red ochre by Early Neanderthals. *Proc Am Philos Soc* 109(6):1889–1894. <https://doi.org/10.1073/pnas.1112261109>

- Rosso DE, Pitarch Marti A, d'Errico F (2016) Middle Stone Age ochre processing and behavioral complexity in the horn of Africa: evidence from PorcEpic Cave, Dire Dawa, Ethiopia. *PLoS One* 11(11):e0164793. <https://doi.org/10.1371/journal.pone.0164793>
- Salomon H (2009) Les matières colorantes au début du Paléolithique supérieur: sources, transformations et fonctions. Ph.D. dissertation, Université Bordeaux I
- Schmidt P, Stynder D, Conard NJ, Parkington JE (2020) When was silcrete heat treatment invented in South Africa? *Palgrave Commun* 6(1):1–10. <https://doi.org/10.1057/s41599-020-0454-z>
- Schoonraad M, Beaumont P (1968) The North Brabant Shelter, north western Transvaal. *S Afr J Sci* 64(8):319
- Soriano S, Villa P, Wadley L (2009) Ochre for the toolmaker: shaping the Still Bay points at Sibudu (KwaZulu-Natal, South Africa). *J Afr Archaeol* 7:41–54. <https://doi.org/10.3213/1612-1651-10121>
- Stewart BA, Mitchell PJ (2018) Late Quaternary palaeoclimates and human-environment dynamics of the Maloti-Drakensberg region, southern Africa. *Quatern Sci Rev* 196:1–20. <https://doi.org/10.1016/j.quascirev.2018.07.014>
- Thackeray AI, Thackeray JF, Beaumont PB (1983) Excavations at the Blinkklipkop specularite mine near Postmasburg, Northern Cape. *South Afr Archaeol Bull* 17–25. <https://doi.org/10.2307/3888211>
- Tobias PV (1949) The excavation of Mwulu's Cave, Potgietersrust district. *South Afr Archaeol Bull* 4(13):2–13. <https://doi.org/10.2307/3887072>
- Tomasso A (2018) Territoire de mobilité et territoire social: structuration du territoire d'approvisionnement en matières premières lithiques dans l'Épigravettien récent au nord-ouest de l'Italie. *Bulletin de la Société préhistorique française* 115(4):677–700. <https://www.jstor.org/stable/26645149>. Accessed 4 May 2023
- Val A, de la Peña P, Duval M et al (2021) The place beyond the trees: renewed excavations of the Middle Stone Age deposits at Olieboomspoor in the Waterberg Mountains of the South African Savanna Biome. *Archaeol Anthropol Sci* 13(7):1–32. <https://doi.org/10.1007/s12520-021-01302-7>
- van der Ryst MM (2007) Seeking shelter: Later Stone Age hunters, gatherers and fishers of Olieboomspoor in the Western Waterberg, South of the Limpopo. PhD thesis. University of the Witwatersrand, Johannesburg
- Velliky EC, MacDonald BL, Porr M, Conard NJ (2021) First large-scale provenance study of pigments reveals new complex behavioural patterns during the Upper Palaeolithic of south-western Germany. *Archaeometry* 63(1):173–193. <https://publons.com/10.1111/arc.12611>
- Vettese D, Blasco R, Cáceres I et al (2020) Towards an understanding of hominin marrow extraction strategies: a proposal for a percussion mark terminology. *Archaeol Anthropol Sci* 12(2):1–19. <https://doi.org/10.1007/s12520-019-00972-8>
- Wadley L (2005) Ochre crayons or waste products? Replications compared with MSA 'crayons' from Sibudu Cave. *South Africa before Farming* 2005(3):1–12. <https://doi.org/10.3828/bfarm.2005.3.1>
- Wadley L (2005) Putting ochre to the test: replication studies of adhesives that may have been used for hafting tools in the Middle Stone Age. *J Hum Evol* 49:587–601. <https://doi.org/10.1016/j.jhevol.2005.06.007>
- Wadley L, Hodgskiss T, Grant M (2009) Implications for complex cognition from the hafting of tools with compound adhesives in the Middle Stone Age, South Africa. *Proc Natl Acad Sci USA* 106(24):9590–9594. <https://doi.org/10.1073/pnas.090095710>
- Wadley L, Murungi M, Witelson D et al (2016) Steenbokfontein 9KR: a Middle Stone Age spring site in Limpopo, South Africa *S Afr Archaeol Bull* 71(204):130–145
- Wadley L, Mauran G, Sievers C et al (2021) Red Balloon Rock Shelter: Iron Age and Middle Stone Age occupations on the Waterberg Plateau in Limpopo, South Africa. *Southern African Humanities* 34(1):19–58. https://hdl.handle.net/10520/ejc-nmsa_sah-v34-n1-a2. Accessed 4 May 2023
- Wadley L (2015) Those marvelous millennia the middle stone age of Southern Africa. *Azania Archaeol Res Afr* 50(2):155–226. <https://doi.org/10.1080/0067270X.2015.1039236>
- Watts I (1998) The origin of symbolic culture: the Middle Stone Age of southern Africa and Khoisan ethnography. Unpubl. Ph.D. thesis: University of London
- Watts I (1999) The origin of symbolic culture. In: Dunbar R, Knight C, Power C (eds) *The evolution of culture*. Edinburgh University Press, Edinburgh, pp 113–146
- Watts I (2002) Ochre in the Middle Stone Age of southern Africa: ritualized display or hide preservative? *South Afr Archaeol Bull* 57:1–14. <https://doi.org/10.2307/3889102>
- Watts I (2009) Red ochre, body painting, and language: interpreting the Blombos ochre. In: Botha R, Knight C (eds) *The cradle of language*. Oxford University Press, Oxford, pp 62–92
- Watts I (2010) The pigments from Pinnacle Point Cave 13B, Western Cape, South Africa. *J Hum Evol* 59:392–411. <https://doi.org/10.1016/j.jhevol.2010.07.006>
- Watts I, Chazan M, Wilkins J (2016) Early evidence for brilliant ritualized display: Specularite use in the Northern Cape (South Africa) between ~ 500 and ~ 300 ka. *Curr Anthropol* 57(3):287–310. <https://doi.org/10.1086/686484>
- Yokoyama Y, Falguères C, Quaegebeur JP (1986) E.S.R. Dating of quartz from Quaternary sediments: first attempt. *Nuclear Tracks* 10(4–6):921–928. [https://doi.org/10.1016/0735-245X\(85\)90109-7](https://doi.org/10.1016/0735-245X(85)90109-7)