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RESEARCH

Ochre use at Olieboomspoort, South Africa: insights into specular hematite use and collection during the Middle Stone Age

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Abstract

Recent excavations at Olieboomspoort (OBP) in the Waterberg Mountains of South Africa confirmed previous research at the site that highlighted an abundance of ochre in the Middle Stone Age (MSA) deposits. Here, we report on the results of an analysis of the ochre from the MSA deposits excavated in 2018–2019. Fossilised equid teeth from these deposits were recently dated to approximately 150 ka, an early date for such a sizeable ochre assemblage in southern Africa. Calcium carbonate concretions were removed from ochre pieces using hydrochloric acid. Macro- and microscopic analyses were undertaken to identify raw material types and to investigate utilisation strategies. There are 438 pieces in the assemblage and only 14 of them show definite use-traces. The predominant raw material is a micaceous, hard specular hematite, which is rare at MSA sites elsewhere in southern Africa. A preliminary investigation into the geological nature of the ochreous materials in the archaeological sample and those available in the area was performed using semi-quantitative portable X-ray fluorescence (pXRF), XRF, and inductively coupled plasma mass spectrometry (ICP-MS). Together with site formation processes, we suggest possible, primarily local sources of the ochre found in the deposits. The data do not support previous suggestions that OBP was used as an ochre caching site that may have formed part of an exchange network during the MSA. Instead, the local abundance of nodules of specular hematite within the Waterberg sandstone, the limited number of used pieces in the assemblage, and the stratigraphic context indicate a more natural, less anthropogenic explanation for the abundance of ochre at the site.

Keywords Waterberg · Sourcing · Middle Pleistocene · MIS 5 · MIS 6 · Ochre cleaning

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Introduction

Ochre *sensu lato* is described as a variety of ferruginous materials that produce a colourful streak or powder (Dayet 2021; Popelka-Filcoff and Zipkin 2022). Apart from the more obvious uses of ochre as a pigment for artistic or symbolic purposes (Rudner 1982; Thackeray et al. 1983; Watts 2002; Taçon 2004; Henshilwood et al. 2011; Villa et al. 2015), "ochre" and iron-oxide-rich clayey minerals have a wide variety of functional applications—ethnographically and archaeologically. These include use as a sunscreen (Rifkin et al. 2015; Havenga et al. 2022), topical insect repellent (Rifkin 2015), an ingredient in adhesives for tool hafting (Wadley 2005; Lombard 2007; Wadley et al. 2009), hide preservative (Audouin and Plisson 1982; Rudner 1982; Rifkin 2011), and topical medicine (Buthelezi-Dube et al. 2022). Ochre pieces found at archaeological sites are often interpreted as pigments used for symbolic and artistic purposes, but in the last two decades in-depth analyses of ochre assemblages around the world have increased in frequency and shown that humans had more varied interactions with these colourful minerals in the deep past. Uses of ochre have been interpreted as indicators of complex cognition as well as evidence for symbolically mediated behaviours among early *Homo sapiens* (Henshilwood and Lombard 2013; Wadley 2010, 2013). Southern African Middle Stone Age (MSA) sites have been the focus of archaeological research into the emergence of innovative behaviours and abilities by *H. sapiens*. There is, however, a distorted geographical focus within southern Africa, with relatively less focus on MSA ochre assemblages from inland sites compared to sites along the coast (Dayet Bouillot et al. 2017; Culey 2022).

Ochre use in southern Africa dates back to the Middle Pleistocene. Specular hematite and hematite pieces, some utilised, were found in ~500,000 year old (ka) layers at Kathu Pan (Watts et al. 2016); specular hematite pieces are found in ~300 ka layers at Canteen Kopje (Chazan et al. 2013; Watts et al. 2016); and a single piece of crayon-shaped, ground hematite was found in ~200 ka layers at Nooitgedacht (Beaumont and Morris 1990; Barham 2002). Ochre is commonly found at MSA sites (approx. 300–30 ka) (Watts 2002; Wadley 2015) in southern Africa, particularly in Late Pleistocene archaeological assemblages, after the onset of Marine Isotope Stage (MIS) 5 at roughly 130 ka. After 100 ka, evidence of humans making deliberate markings, or engravings, on ochre, as well as evidence of processing ochre into a secondary form—powder—is reasonably common (e.g., Watts 2002; Dayet et al. 2013, 2016; Hodgskiss and Wadley 2017; Dapschauskas et al. 2022). Notably, large amounts of anthropogenically modified (utilised) ochre pieces have been found at Blombos Cave. This includes the oldest known geometric engraved design found on an ochre piece dating to ~77 ka (Henshilwood et al. 2009), as well as ochre processing kits dated to 100 ka (Henshilwood et al. 2011). These consist of ground ochre pieces, ochre-stained grindstones, and abalone shells containing a compound mixture of ochre powder, crushed bone, and charcoal.

The ochre assemblages at most Middle Pleistocene and early Late Pleistocene sites in southern Africa generally contain a small percentage of pieces that have been used or modified. Some of these sites include Pinnacle Point (Marean et al. 2010; Watts 2010), Border Cave (Watts 1998; Backwell et al. 2018), Rose Cottage Cave (Hodgskiss and Wadley 2017), Bushman Rock Shelter (Watts 1998, 2002; Porraz et al. 2018), Cave of Hearths (Mason 1988; Watts 1998), Wonderwerk Cave (Chazan and Horwitz 2009), Wonderkrater (Backwell et al. 2014), and Mwulu's Cave (Watts 1998; de la Peña et al. 2019). Most evidence of utilisation on ochre pieces from this period comes in the form of grinding or abrasion striations on the surface of the pieces, with rare instances of engravings, pecking, or crushing activities. The purpose of the powders produced by grinding is largely speculative as many possible uses are archaeologically invisible. Ochre powder residues are found on various artefacts such as grindstones, bone tools, and lithics, as well as various types of shell beads with most evidence coming from assemblages younger than 100 ka (Henshilwood et al. 2001, 2004; Dayet et al. 2013; Wojcieszak and Wadley 2018). Ochre-stained grindstones are found at Diepkloof Rock Shelter, Klasies River, and Mwulu's Cave in layers older than 100 ka (Watts 1998; Dayet et al. 2013; Culey 2019; Feathers et al. 2020). Online Resource 1 provides an inventory of MIS 6 and 5 ochre assemblages from South African sites and we refer to Hodgskiss (2020), Watts (1998, 2002), and Dapschauskas et al. (2022) for overviews of ochre finds at (southern) African MSA sites.

It is worth noting that harder, more obviously micaceous specular hematite generally appears more frequently within earlier MSA assemblages than softer, less specular ochre varieties such as ferricretes, shales, and mudstones (see Online Resource 1). This may suggest an earlier preference for this harder, specular raw material, which was later gradually abandoned for softer ochre varieties that required less effort to process. This overall change in raw material type may be indicative of ochre application changes through time, perhaps dependent upon behavioural or social changes. However, given that most of the sites at which this specular hematite occurs at this early date are within the interior (mostly in the Limpopo), this may simply be due to different geological contexts.

The MSA ochre finds at Olieboomspoort (OBP) are distinctive compared to other MSA ochre assemblages. Firstly, there is an exceptionally high volume of specular hematite, which is relatively rare at archaeological sites elsewhere in southern Africa, and, secondly, few of the pieces have been utilised. Specular hematite does occur at other sites in the Waterberg region and in the broader area, such as Red Balloon Rock Shelter (Wadley et al. 2021; Mauran 2023) and Mwulu's Cave (de la Peña et al. 2019), but not in the quantities found at OBP. The uniqueness of this assemblage has spurred questions about the origin of this specular hematite: was it brought to the site intentionally by humans or is it an accumulation resulting from natural processes (or some degree of both)? Van der Ryst (2007) and Watts (1998, 2002) suggested—based partly on the anomalously high quantities of unutilised specular hematite at the site—that OBP may have been a "special purpose" or "factory" site. They proposed that pieces might have been collected from a nearby source, brought back to the site, cached, and processed as needed, with some of the pieces being transported as part of a (hypothetical) regional trade network.

Olieboomspoort

OBP is a red sandstone rock shelter situated within the Mogalakwena Formation in the Waterberg Mountain Range of Limpopo Province, South Africa (23° 52′ 42″ S; 27° 38′ 17″ E). The shelter is 70 m in length, and the overhang extends, today, to a maximum of 7 m deep, although much of the shelter is shallower (van der Ryst 2007; Val et al. 2021) (Fig. 1). The shelter lies a few metres away from the perennial and shallow Riet Spruit River, which is a tributary of the Mokolo River that runs across the Waterberg plateau. The Mogalakwena geological formation within the Waterberg group is comprised of coarse-grained sandstones and quartz conglomerates, interbedded with micaceous shales, likely with fluvial depositional influences (Brandl 1996; Corcoran et al. 2013; Masia 2022). The Mogalakwena is a likely source of the various iron-rich minerals common in both the MSA and Later Stone Age (LSA) levels of OBP (van der Ryst 2007; Eriksson et al. 2008; Masia 2022). It includes iron oxides or hematite in the form of locally abundant hematite nodules, crystal matrices, and sheets of specularite (van der Ryst 2007).

The archaeological deposits at OBP contain evidence of human visits to the rock shelter perhaps as far back as the Earlier Stone Age (ESA) and more regularly in pulses during the MSA and again during the last two millennia (Mason 1962; van der Ryst 2007; Val et al. 2021). The ESA presence at OBP is ephemeral and followed by repeated use of the shelter during the MSA. An extensive chronological hiatus separates the Middle Pleistocene archaeological deposits from the late Holocene phase, which preserve mixed LSA and Iron Age cultural remains (van der Ryst 2007; Val et al. 2021). Evidence of recent occupations also occurs in the form of densely painted, but poorly preserved, rock art on the shelter walls—rock art styles associated with San hunter-gatherers, geometric Khoekhoe herder finger paintings, as well as geometric and animal depictions made by Bantu-speaking groups, likely northern Sotho-speakers (van der Ryst 2007).

OBP was first excavated by Prof. Revil Mason in 1954 (Mason 1962; Watts 1998) and by Dr Maria van der Ryst in 1997 (van der Ryst 2007). The most recent excavations in 2018 and 2019 were led by a team from the University of the Witwatersrand (Val et al. 2021). Excavations at OBP have yielded a rich, predominantly quartzite and quartz, lithic assemblage including *Levallois* flakes, cores, and retouched pieces. Well-preserved faunal remains signal a dominance of species relying on open grassland and riverine environments, as well as the rocky surroundings. Pollen spectra

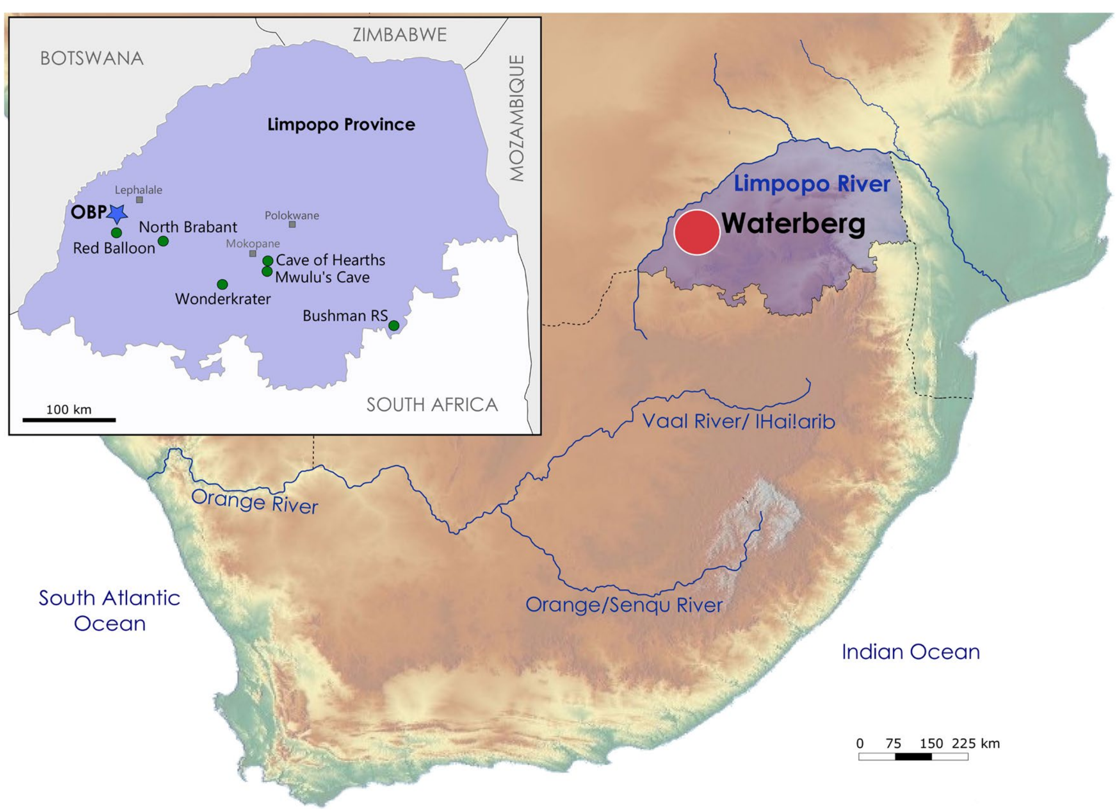


Fig. 1 Location of the Waterberg Mountains (red dot) and Limpopo Province (in light blue) in South Africa, and the location of OBP and other sites mentioned in the text within Limpopo Province (adapted from Wadley et al. 2022)

and phytolith data reflect vegetation typical of savanna and woodland environments within the Summer Rainfall Zone. We refer the reader to Val et al. (2021, 2023) for detailed information about the site and recent excavations. The 2018 and 2019 excavations identified an unclear and deflated stratigraphy (Val et al. 2021). Three distinct sedimentary units inside the MSA deposits were differentiated based on Munsell chart colours and textures. These include grey sediment (GS), dark reddish grey sediment (DRG), and yellow-reddish sand (YRS), which form part of a larger sedimentary unit corresponding to the MSA. Uranium-series combined with electro-spin resonance (ESR) dating on two fossilised equid teeth from the GS unit provided an age estimate of approximately 150 ka for the MSA deposits at the site (Val et al. 2021). This tentatively places these three MSA units within the MIS 6—190–130 ka.

Previous ochre research at OBP

The OBP ochre assemblage from Mason's excavation, re-analysed by Ian Watts, is one of the heaviest ochre assemblages excavated at an MSA site in southern Africa—comprising only 304 pieces but weighting 12 kg (Watts 1998). To put this into context, Sibudu, for example, had an analysed assemblage of 5449 pieces weighing 15.4 kg (Hodgskiss 2012). The Mason OBP ochre sample comprises 8.2% of the total ochre and lithic assemblage from the MSA deposits, but by weight accounts for nearly half of the assemblage (Watts 1998). Specularite (or specular hematite) dominates the ochre assemblage in both the MSA and LSA levels (Watts 1998, 2002; van der Ryst 2007). Roughly 13% of the ochre pieces excavated by Mason exhibit use-traces, in the form of striations, faceting, and polish (Watts 1998, 2002), most of which indicate use in the form of grinding or abrasion against a rock or lower grindstone. Several ochre-stained grindstones were reported from the MSA levels that confirm this activity (Mason 1962; van der Ryst 2007). Mason (1962) suggested that the ochre powder was possibly used for cosmetic and art purposes.

Watts (1998) identified the majority of the ochre pieces (98.7%) as "specular hematite", whereas van der Ryst uses the term "specularite" to refer to the same raw material, with the terms being used interchangeably. In this study, we have chosen to use the term "specular hematite" (see the description of the raw material in the methodology). This material is hard (generally Mohs 5) and streak colours are mostly hues of red, ranging from dark red to metallic grey-red (Watts 1998). Some of the ground pieces have a polished/reflective surface, increasing the natural shiny nature of the raw material, and Watts (1998) questioned whether this shine might have been a goal of the abrasive actions. Both utilised and unutilised "ochre", hematite, and specular hematite pieces are noted in archaeological assemblages from sites in the Waterberg (e.g., Red Balloon Shelter) (Wadley et al. 2021). The high quantity of unutilised specular hematite at OBP is unusual, and speculations that the rock shelter may have functioned as a factory site for ochre (Watts 1998, 2002; van der Ryst 2007) were underlying factors that initiated this research.

Water has likely affected the deposits at OBP, which are a significant palimpsest of concentrated material, including specular hematite nodules. Some of these nodules could be derived from disintegration processes of the surrounding sandstone. Waterberg-based geologist Dr Richard Wadley observed that the lower sediments of the Waterberg supergroup contain an unusually high concentration of iron oxides, mostly in the form of hematite (Jansen 1982; Callaghan 1987; R. Wadley, pers. comm. 2022). Due to various tectonic events in the deep past (i.e. 1600–200 ma), intrusive diabase dykes and sills resulted in contact metamorphism of the sediments (Callaghan 1987; R. Wadley, pers. comm. 2022). One of the consequences of this metamorphic process of the iron-rich sandstones seems to have been the mobilisation of the iron and its reconstitution in nodules within the sandstone, i.e. secondary, or diagenic mineralisation of the sediments (R. Wadley, pers. comm. 2022). There is evidence of these hematite nodules found in the sediments, especially in proximity to the current diabase contacts. These are nearly always associated with the Mogalakwena formation. The precise process whereby the disseminated iron within the sediments was mobilised and re-concentrated in nodules within the sediments has not yet been adequately described, but that it was secondary, or diagenetic in nature, is generally accepted (R. Wadley, pers. comm. 2022). When these sediments erode, masses of iron-rich quartzitic gravels are found; some of these concretions comprise crystalline specular hematite.

Further investigation into ochre use patterns, ochre types, and proximity of OBP to specular hematite and other "ochre" sources is necessary to obtain a clearer picture about whether OBP functioned as a factory site or was part of a regional network during the MSA. In this study, we investigate how much of the ochre found at the site is the result of deliberate collection and use, and how much is the result of natural accumulation processes.

Materials and methods

The assemblage reported on here was excavated from OBP in 2018 and 2019. The MSA assemblages from the Mason and Val excavations are curated by the Archaeology Division of the School of Geography, Archaeology, and Environmental Studies at the University of the Witwatersrand, Johannesburg, South Africa. The material analysed from the 2018 to 2019 excavation includes both the plotted specimens and the >10 mm pieces retrieved during sorting of sieved material.

Like most bone remains and lithic artefacts, many of the ochre pieces from OBP are covered in calcium carbonate concretions, some almost entirely. This can obscure raw material type classification and prevent use-trace identification. Given the lack of literature presently available on cleaning methods for archaeological ochre pieces, particularly those involving chemicals, it was necessary to perform experimental cleaning with modern ochre samples and different acid solutions in order to develop an effective way to remove these concretions without damaging the surfaces of the archaeological pieces. Diluted hydrochloric acid was chosen as it has been used to successfully remove calcium carbonate concretions on lithics.

Six modern ochre samples were chosen for testing—including hard hematite, soft shales, and snuffbox shale (hard ironstone "casings" with soft pockets of soft iron-rich pigment, see Hodgskiss 2012)—as well as two pieces from the OBP assemblage that did not exhibit any visible signs of use. Four samples were placed in 10% hydrochloric acid and tap water solution, and four in 20% solution. All the samples were left in the solutions for approximately 40 min while being monitored. Both solutions were equally effective in removing the concretions, with the 20% solution acting faster. The samples were removed from the solution and rinsed with tap water. Surfaces were examined macroscopically and these tests indicated that this form of hydrochloric acid treatment did not cause macroscopic damage to the surfaces of the ochre varieties (Fig. 2).

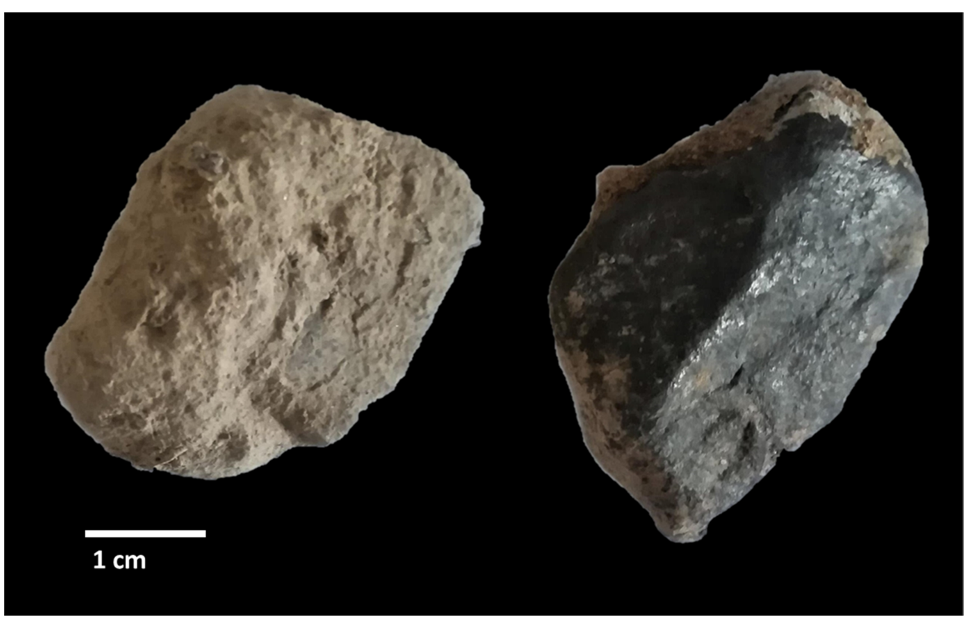
It was not necessary to acid clean all the archaeological ochre pieces. Rather, we selected pieces with significant concretions that inhibited accurate size and weight measurement, surface feature visibility, colour, and raw material identification. Additionally, pieces that had a surface shape that may indicate use (e.g. flat surfaces may suggest grinding or abrasion), or that had concretions obscuring a part of the piece that may potentially exhibit use-wear, were also chosen for cleaning. We did not clean soft or crumbly pieces that may get damaged during the cleaning process. Prior to cleaning, details and features of the pieces were photographed and recorded, as well as the percentage solution used, length of time in the solution, and any other relevant information.

Twenty percent solution was used for pieces with thick or extensive concretions. For pieces with fewer concretions, 10% solution was used. Spot treatment with the acid solution was performed on pieces where only a particular area of the piece needed to be exposed, and for pieces with signs of use. This was done to avoid submerging the areas with possible use, and minimise any possible damage on the surfaces. Pieces were submerged for 7 to 40 min, depending on how quickly the concretions dissolved. Cleaning was avoided on pieces that showed possible use-traces. This protocol can be built upon by further research, specifically concerning any microscopic and elemental effects of acid cleaning on ochre (specifically concerning whether hydrochloric acid could compromise iron oxide signatures), the effects of this cleaning on a broader range of ochre/residue types, and the efficacy of different acid types, such as acetic and citric acid.

Macroscopic analysis

A standard set of criteria was used to macroscopically describe each piece (Hodgskiss 2010, 2012; Rosso et al. 2016; Dayet Bouillot et al. 2017; Rosso et al. 2017; Velliky et al. 2018). These categories included raw material, colour, hardness, size, weight, approximate grain size, surface morphology, piece shape, and specularity (i.e.

Fig. 2 Hydrochloric acid treatment of specular hematite. OBP specular hematite piece with calcium carbonate concretions. Left: before acid treatment. Right: after acid treatment



the presence of specular particles giving pieces a glittery appearance). All pieces were examined under natural light, and each piece was individually numbered and stored in a labelled plastic bag.

The outer surfaces of most of the pieces in the OBP assemblage are dark (often black) and give little to no indication of the true piece/powder colour. Thus, streak tests were performed on the unutilised surface (or least likely surface to be utilised based on indications such as surface shape) of each piece against a matte white porcelain tile, altering a $<1\text{mm}^2$ area of the piece (Hodgskiss 2012; Velliky et al. 2018). Streak tests allow for more accurate determination of the colour of ochre pieces in terms of the powder they produce (Dayet Bouillot et al. 2017). Most of the tested areas are not macroscopically visible on the surface of the piece afterwards. The streaks were assigned a Munsell code and then categorised into general colour groups (e.g. dark red, dark red-purple) (Hodgskiss 2012; Velliky et al. 2018). Estimated grain size was determined by surface texture observations made through macroscopic and microscopic examination; "sandy" pieces have clearly visible grains, "silty" pieces have a gritty texture, but grains are small or only visible microscopically, and clayey pieces are fine-grained and do not have any gritty texture.

To clarify our terminology and define the raw material categories used in our analysis, we provide brief descriptions of each raw material below.

Specular hematite There are two main sub-types or varieties of "specular hematite" and "specularite" raw materials in the ochre assemblage from OBP (note: "specular hematite" as used throughout this paper encompasses both). The most common form of specular hematite in the study assemblage is medium to hard with a dark black, sometimes silvery grey, metallic outer colour (Watts 1998) (Fig. 3a). Streaks range between very dark purples and dark reds, which have a glittery appearance. This glittery quality is especially visible on the interior of pieces that have been broken (Fig. 3a). This specular hematite has a predominantly fine-grained surface texture. The second, less common, form of specular hematite found in the study assemblage has a flaky, micaceous structure that does not lend itself easily to powder production (Fig. 3b). Streak colours are similar to those produced by the first type.

Hematite While this raw material is in many ways similar to the predominant form of specular hematite, it does not have a glittery appearance. The hematite pieces have medium to hard Mohs scores, with dark grey/black surface colour (Fig. 3c). They predominantly have a clayey and clayey/silty texture. Streak colours range between red, dark red, dark purple, and dark red purple.

Mudstone These are soft, fine-grained pieces, which tend to be crumbly and mostly produce a dark red, vibrant powder when streak tests are performed (Fig. 3d).

Shale This raw material is medium to soft, fine-grained, and with a fissile or platy structure (Fig. 3e). Many of the pieces have a glittery appearance, although not all. Shales from OBP commonly produce a dark red streak, but some produce a dry, grainy powder.

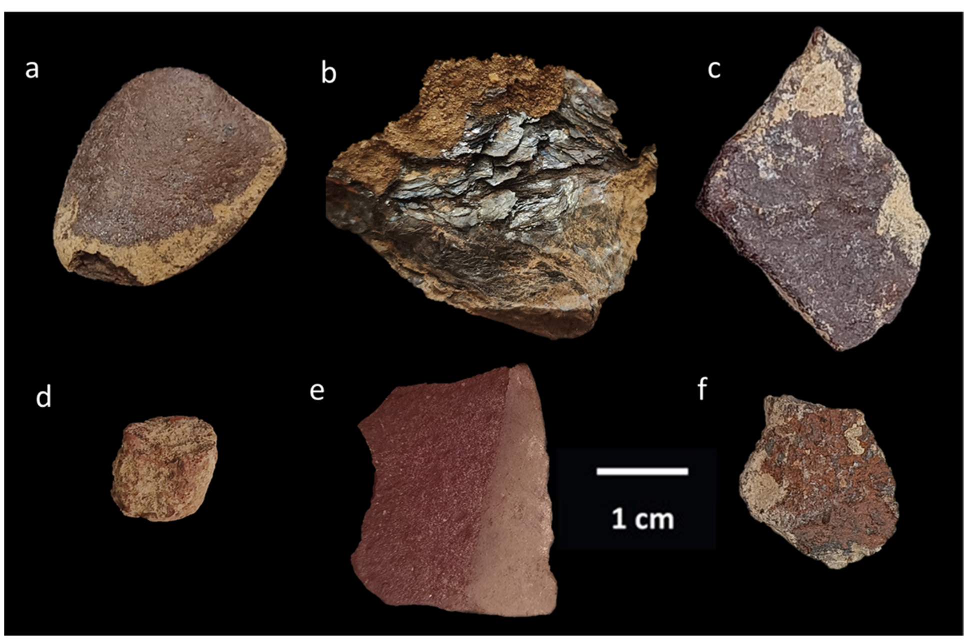


Fig. 3 Representative specimens of the ochre raw material categories found in the OBP ochre assemblage. **a** The most common form of specular hematite (OBP-1896-4; concretions form the light rim); **b** flaky, micaceous specular hematite (OBP-1744-1; note sandy concretions on the piece); **c** hematite (OBP-4708-2); **d** mudstone (OBP-2409-42); **e** shale (OBP-3290-4); **f** ferricrete conglomerate (OBP-321-1)

Ferricrete conglomerate This raw material is mostly sandy in texture with a medium harness, but is heterogonous and often has visible inclusions of quartz, which Watts (1998: 181) described as "material cemented by iron minerals" (Fig. 3f). They mostly produce a dark purple/red streak.

Microscopic analysis

Each piece was microscopically analysed using an Olympus SC50/SZ61 reflected light microscope fitted with an Olympus EP50 camera, using a variety of magnification levels from ×0.67 to ×4.5. Any surface modification on the pieces was noted—this includes abrasive and percussive traces, or any markings on the surface. Use-traces not clearly visible macroscopically, such as microstriations (microscopically visible parallel striations) or polish, were identified during this stage of examination. Pieces were placed into categories based on use-traces: definite signs of use ("utilised"), possible signs of use ("possibly utilised"), and "unutilised" pieces. Pieces with markings or use-traces that we could not determine whether they resulted from anthropogenic modification or natural processes were categorised as possibly utilised. Pieces with no use-traces were classified as unutilised.

Pieces with clear signs of anthropogenic modification were categorised as utilised, and where possible assigned to use-wear activity categories (such as grinding/abrasion, pecking or rubbing). The identification of use-traces and inferences about the anthropogenic processes that may have caused them are based predominantly on previous experimental ochre use studies (e.g. Hodgskiss 2010; Rifkin 2012) and ochre use-trace analyses (e.g. Hodgskiss 2012; Velliky et al. 2018). Grinding or abrasion actions involve abrading an ochre piece back and forth against a second flat, harder raw material (grindstone), producing powder. This results in parallel striations, which often extend to the edges of the piece, and a flattened surface shape. Ochre rubbed against softer raw materials, such as hide or skin, acquires use-traces in the form of smoothing, microstriations, rounded edges, and polish. It should be noted that these studies do not include significant amounts of specular hematite. This highlights the need for locally procured reference materials and experimental analyses on this material.

Elemental analyses

A preliminary elemental investigation of pieces from the study assemblage and ochre source samples was undertaken to obtain qualitative and quantitative information about the nature of the archaeological assemblage and modern ochre sourced from around the site. Non-destructive portable X-ray fluorescence (pXRF) analyses were performed on the archaeological ochre. Given the wide variability in ochre raw material composition and that pXRF instrument calibrations rarely provide accurate quantitative characterizations (Dayet 2021; Popelka-Filcoff and Zipkin 2022), the results presented here only allow for qualitative and semi-quantitative elemental conclusions about the composition of the pieces. These analyses were performed with a Thermo Scientific Niton XL3t GOLDD+ spectrometer at the Earth Sciences Laboratory at the University of the Witwatersrand. Samples were analysed with an 8-mm diameter beam, with a 50kV excitation source and a current intensity of 1mA. No filters were used in the analyses. Thirty-four elements were quantified. Only elements that were reliably measured, and were above the limits of detection, were used in the analysis (Online Resource 2). Twenty-four archaeological pieces underwent pXRF analysis—23 from the current assemblage, and one from the Mason assemblage. Effort was made to include all raw material types but suitably sized pieces with a flat surface were not present for some types. The concretions were also tested to discern their qualitative geological composition. One reading of 240 s was taken on each piece.

Two geological surveys were carried out in the area in 2021 to determine preliminary sourcing possibilities. The first survey (undertaken by Jasmin Culey, Tammy Hodgskiss, and Aurore Val) focused on identifying possible sources of the specular hematite found in the study assemblage in the areas surrounding the site and aimed to develop an understanding of the different ochreous raw material types available in the OBP landscape. The second survey was undertaken by colleagues (Dineo Masia, Zubair Jinnah) and one of the authors (Paloma de la Peña) working on lithic sourcing in the area. Iron ore and iron mineral varieties were collected from several areas directly around the site, as well as from neighbouring farms and towns. Sampled sources include two local sources. The first (secondary) source identified during these two surveys comprises loose nodules found in large numbers within 1 km of the site and in the dripline of the shelter. The second source is an historic iron ore mine on a neighbouring farm (<10 km from the site). We sampled a non-local source, the commercial iron ore-mining region of Thabazimbi ~100 km from the site (Fig. 4). The nearby town of Lephalale, higher up the Mokolo River (Phalala), was also explored for ochre outcrops, but none were located. Sourcing of rocks and pebbles in the river was attempted, but due to vegetation growth, we were unable to find or determine if any ochre types occur in the riverbed. Pieces were informally experimented with by grinding and rubbing the pieces to test their colouring abilities, specularity, and use-trace formation (Fig. 5). These experiments allowed for a better understanding of the raw material characteristics and the formation of use-traces on the pieces during various activities.

The geological samples were prepared and analysed using equipment at the School of Geography, Archaeology and Environmental Studies at the University of the

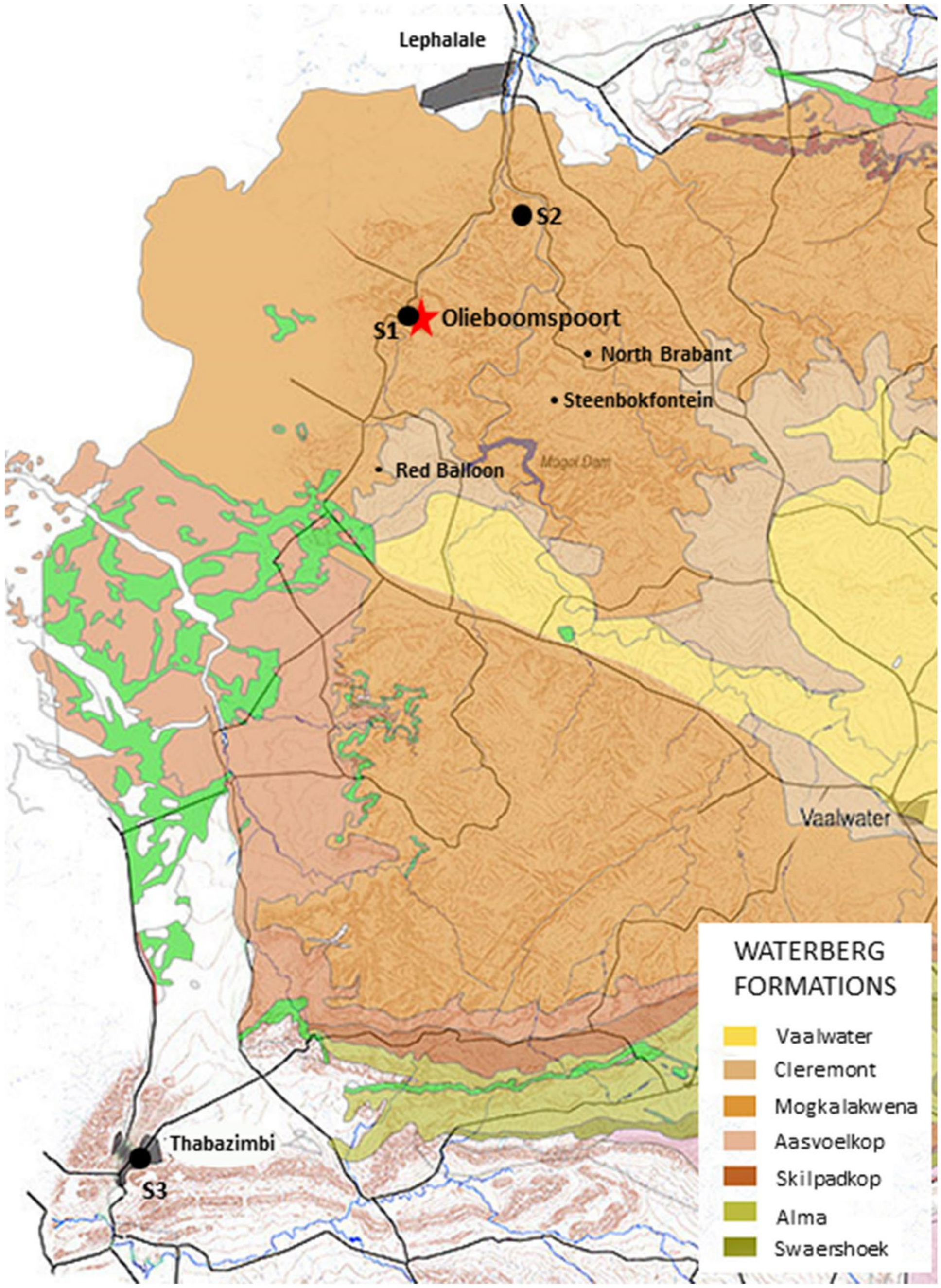


Fig. 4 Geological map of the Waterberg, showing the location of OBP, the geological ochre sample source areas, and approximate locations of some nearby MSA sites. S1: loose nodules within 1 km of the site and in the dripline of the shelter. S2: historic iron ore mine on a neighbouring farm (Masia 2022). S3: Thabazimbi mine. Map modified from Warwick Tarboton, Waterberg Bioquest

Witwatersrand. Large pieces were crushed with a jaw crusher, with all components cleaned with acetone and vacuumed to avoid contamination. Samples were milled in a TS-250 Mill. The samples were milled in steel milling pods, which were cleaned between samples with coarse silica sand and acetone. Invasive X-ray fluorescence (XRF) and inductively coupled plasma mass spectrometry (ICP-MS) tests were performed on the samples to establish the elemental composition of the ochre types (Popelka-Filcoff et al. 2007; Mauran et al. 2021). Major elements were determined using a Panalytical Axios XRF spectrometer, with samples analysed at 50kV and 50mA. Sample weight was 0.35 gm and flux weight 2.0 gm (see Online Resource 2 for additional information on analytical procedures). Oxides were determined using the Norrish Fusion technique (Norrish and Hutton 1969) using certified calibrations standards. ICP-MS samples were analysed on a Thermo Scientific iCAP RQ. Plasma temperature was 70,000°C, with Ar flow rates at 1L/min, and kinetic energy discrimination (KED) was used with He as the binding element. All measurements were done in triplicate. Forty-two elements were quantified. Further ICP-MS operating procedures are provided in Online Resource 2.

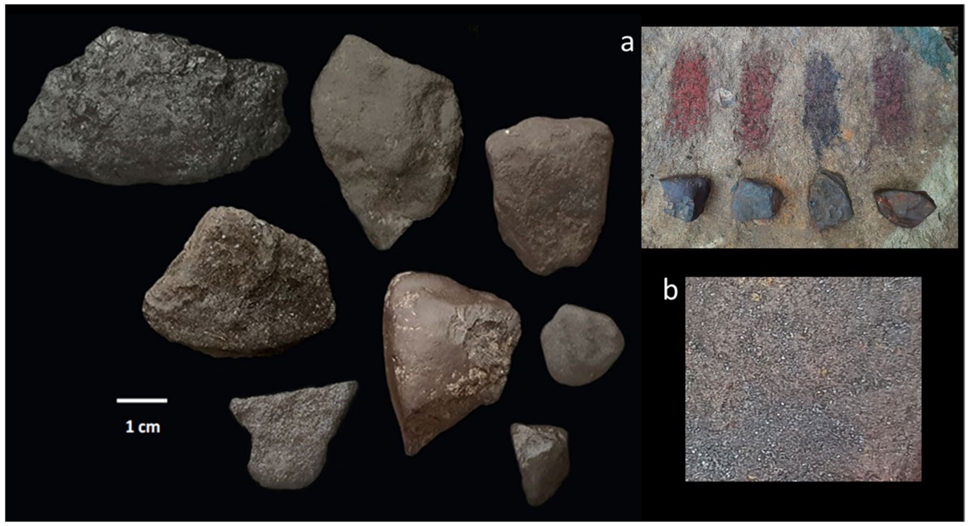


Fig. 5 Specular hematite nodules (non-archaeological) collected from the areas surrounding OBP. Inserts: **a** streak colours produced from these specular hematite nodules, **b** close up of the specular surface appearance

Given the heterogeneous nature of the samples, multivariate analyses were used to differentiate groups within the modern reference and archaeological ochre samples. Data processing and pre-treatment followed procedures used in previous ochre studies (Popelka-Filcoff et al 2007; Eiselt et al. 2011; MacDonald et al. 2011; Dayet et al. 2016; Mauran et al. 2021). Principle component analyses (PCA) that were based on hierarchical cluster analyses using the UPGMA algorithm in PAST (PAST®, Hammer et al. 2001) were used. From the datasets, only elements that were reliably measured, therefore above the limits of detection, were used in the analysis. All data for the PCA were log-transformed to remove bias towards major elements.

Results

Macro- and microscopic analyses

The 2018/2019 MSA ochre assemblage consists of 438 pieces of ochre (>10 mm) with a total weight of 4.6 kg from the sedimentary units YRS, GS, and DRG. Fourteen pieces show definite signs of utilisation (3%) with a total weight of 0.4 kg (364.7 g) (Fig. 6), 38 pieces have possible signs of utilisation (9%) with a total weight of 0.7 kg (706.4 g), and 386 pieces show no signs of utilisation (88%) with a weight of 3.5 kg (3532.1 g). Most pieces (70.81%, n=310) come from GS, 27% of pieces (n=124) are from YRS, and 1% (n=4) from DRG.

Most ochre pieces in the assemblage have streaks that are red-hued, with dark red, red, dark red-purple, and dark purple constituting 83% of the utilised and unutilised pieces (Table 1). Raw materials are mostly fine-grained, with 93% of the pieces with clayey to silty grain-size surface texture, and the majority of pieces are irregular or fragmented. Specular hematite varieties account for 79% of the ochre assemblage. The dark red and dark red-purple streaks also often contain visible glittery particles. This specular quality is especially noticeable on the exposed faces or edges on the interior of pieces, which have fragmented. Hematite, shale, mudstone, and ferricrete conglomerate are present in the assemblage in small quantities.

All 14 utilised pieces come from layers YRS and GS (Fig. 6; Table 1). Specular hematite is the predominant raw material in the utilised assemblage (n=10, 71%), and 93% of the utilised pieces (n=13) have dark red or dark red-purple streaks. A Fisher's exact test shows a significant correlation between dark red streak colour and utilisation (p=0.0139). Use-traces on the pieces are in the form of striations, microstriations, polish, and smoothing (Fig. 7)—often occurring together on one piece. Parallel striations are found on 86% of the utilised pieces, whereas microstriations and smoothing are found on 71% of pieces—often occurring together with each other, and macroscopically visible striations. Polish occurs on 36% of pieces, most of which also display microstriations within the polish (Fig. 7). These use-traces suggest the most common way these pieces were used was by grinding, abrading or rubbing pieces against a surface. Six pieces in the utilised assemblage have faceted edges from use and have flat and parallel, striated surfaces, consistent with grinding or abrasion against a hard surface (Fig. 8). Five of the pieces show rounded edges with unmodified surface shapes, together with microstriations, consistent with rubbing against a soft surface or a similar low intensity action.

Fig. 6 The utilised ochre assemblage from the 2018/2019 OBP excavations. **a** OBP-656-1 (specular hematite). **b** OBP-831-2 (mudstone), **c** OBP-582-1 (specular hematite), **d** OBP-4310-1 (hematite), **e** OBP-605-1 (specular hematite), **f** OBP-3290-4 (shale), **g** OBP-3820-1 (specular hematite), **h** OBP-1783-7 (specular hematite), **i** OBP-2238-1 (specular hematite), **j** OBP-4267-1 (specular hematite), **k** OBP-3646-8 (specular hematite), **l** OBP-2042-3 (hematite), **m** OBP-3290-3 (shale), **n** OBP-2408-2 (specular hematite)

Elemental analyses

Twenty-four of the archaeological specular hematite, hematite, ferricrete conglomerate, shale, and mudstone pieces underwent pXRF analysis (Fig. 9a; Table 2; Online Resource 3). The analysis on the concreted area on OBP-694-1 shows the highest proportion of calcium (Ca) among the analysed pieces, supporting the hypothesis that the concretions are calcium carbonate. High wt% of Fe2O3 were found in dark red hematite and specular hematite pieces. One piece that physically resembled hematite (OBP-1543-2) was ruled out as such due to elevated Si and low Fe proportions. The X-ray fluorescence (XRF) and inductively coupled plasma mass spectrometry (ICP-MS) results on the 10 (powdered) geological samples provided information on the major and trace elemental composition of the modern ochreous raw material (Table 2; Fig. 9b; Online Resources 4 and 5). A unique feature of the Mogalakwena formation is elevated Ti and Zn values (Corcoran et al. 2013); however, we find a negligible correlation between these two elements in both the geological and archaeological samples ($R = 0.0898$).

Based on log-transformed data of the overall content (excluding elements below the limits of detection), the geological and archaeological samples, when clustered and analysed in a PCA, show clear distinctions between the two samples in the composition of the pieces (Fig. 10a). This is possibly due to the nature of the semi-quantitative data received from the pXRF analysis (which is more useful for relative frequency comparisons), compared with the XRF data. Notably, the archaeological pieces have higher Al and Si levels than the modern pieces. The archaeological pieces also show greater variation in the iron concentrations with Fe levels varying between 11 and 90%, while the modern samples have Fe values between 80 and 97% (and with one outlier with 60% Fe). A third cluster of outliers are pieces that have Mg levels below the limits of detection.

Table 1 Comparison between the piece physical characterises (rows) in the unutilised and utilised 2018/2019 OBP ochre assemblages, per layer (column). Highest values for each category are italicised, indicating potential preferential selection. A dash represents 0%. *UNU* unutilised, *U* utilised (possibly utilised pieces not included)

Physical qualities		GS (*n*=282)		DRG (*n*=3)		YRS (*n*=115)		Total (*n*=400)	
		UNU	U	UNU	U	UNU	U	UNU	U
		n=274	*n*=8	*n*=3	*n*=0	*n*=109	*n*=6	*n*=386	*n*=14
Raw material	Specular hematite	*77.0%*	*87.5%*	33.3%	-	*81.7%*	*50%*	*78%*	*71.4%*
	Hematite	16.0%	12.5%	*66.6%*	-	11.0%	16.7%	15.0%	14.3%
	Mudstone	1.8%	-	-	-	0.9%	-	1.6%	-
	Shale	0.4%	-	-	-	5.5%	33.3%	1.8%	14.3%
	Ferricrete conglom.	4.7%	-	-	-	0.9%	-	3.6%	-
Raw material sub-total		100%	100%	100%	-	100%	100%	100%	100%
Colour	Dark red	*24.5%*	*75%*	*66.6%*	-	20.2%	*66.6%*	*23.6%*	*71.4%*
	Red	22.3%	-	33.3%	-	12.8%	16.7%	19.7%	7.1%
	Dark red-purple	20.4%	12.5%	-	-	17.4%	-	19.4%	7.1%
	Dark purple	13.9%	12.5%	-	-	*23.9%*	-	16.6%	7.1%
	Red-purple	3.3%	-	-	-	3.7%	-	3.4%	-
	Red-orange	4.7%	-	-	-	4.6%	-	4.7%	-
	Red-brown	3.3%	-	-	-	6.4%	-	4.1%	-
	Pink	1.1%	-	-	-	-	-	0.8%	-
	Black & very dark red/purple	5.8%	-	-	-	10.1%	-	7%	-
	Faint streak	0.7%	-	-	-	0.9%	16.7%	0.8%	7.1%
Colour sub-total		100%	100%	100%	-	100%	100%	100%	100%
Specular	Present	*78.5%*	*75%*	*33.3%*	-	*81.7%*	*83.3%*	*79%*	*78.6%*
Grain size	Clayey	29.6%	12.5%	33.3%	-	24.8%	33.3%	28.2%	21.4%
	Clayey-silty	31.0%	37.5%	-	-	*38.5%*	16.7%	*32.9%*	28.6%
	Silty	*31.8%*	*50%*	*66.6%*	-	30.3%	*50%*	31.6%	*50%*
	Silty-sandy	6.2%	-	-	-	2.8%	-	5.2%	-
	Sandy	1.5%	-	-	-	3.7%	-	2.1%	-
Grain size sub-total		100%	100%	100%	-	100%	100%	100%	100%
Piece shape	Nodule	15.7%	12.5%	*100%*	-	12.8%	33.3%	15.5%	21.4%
	Irregular/fragment	*84.3%*	*87.5%*	-	-	*87.2%*	*66.7%*	*84.5%*	*78.6%*
Piece shape sub-total		100%	100%	100%	-	100%	100%	100%	100%

Cluster analysis of the archaeological sample pXRF signatures groups the pieces into five clusters (Fig. 10b; Fig OR6.1). Looking at links between physical features and elemental data, there are a few observations, but we do note that raw material types or streak colours are not distinct. Cluster 1 (Fig. 10b) includes all specular hematite pieces with darker red streaks, with dark grey surfaces. Cluster 2 has more variety in the raw material composition and is comprised of specular hematite, ferricrete conglomerate, and shale pieces. Cluster 3 contains specular hematite pieces with various darker red/purple streak colours. Cluster 4 consists of mostly dark red streaked pieces, with a range of raw material types. A fifth cluster of three pieces has unique signatures from the others; two of these pieces are specular hematites that are utilised, and one is a specular shale, with possible utilisation; all three pieces have dark red streaks. Specular hematite nodules appear in all clusters suggesting a variable elemental composition of these pieces—perhaps indicating different sources.

The modern geological samples group into loose clusters with most of the specular hematite and hematite pieces grouped in cluster A (Fig 10c; Fig OR6.2). The two pieces collected from the historic iron ore mine (M-OBP-7 and 8) cluster together (cluster A1) although they are physically distinct (shale versus specular hematite). Within the same loose cluster, four pieces of hematite and specular hematite collected near OBP in secondary contexts (M-OBP-1 to 4, cluster A2) are included, possibly linking the mine to the site, or suggesting similar geological origins. The four pieces are the same, in physical appearance at least, to many of the archaeological pieces in the OBP assemblage. The two pieces collected from Thabazimbi (M-OBP-9 and 10)

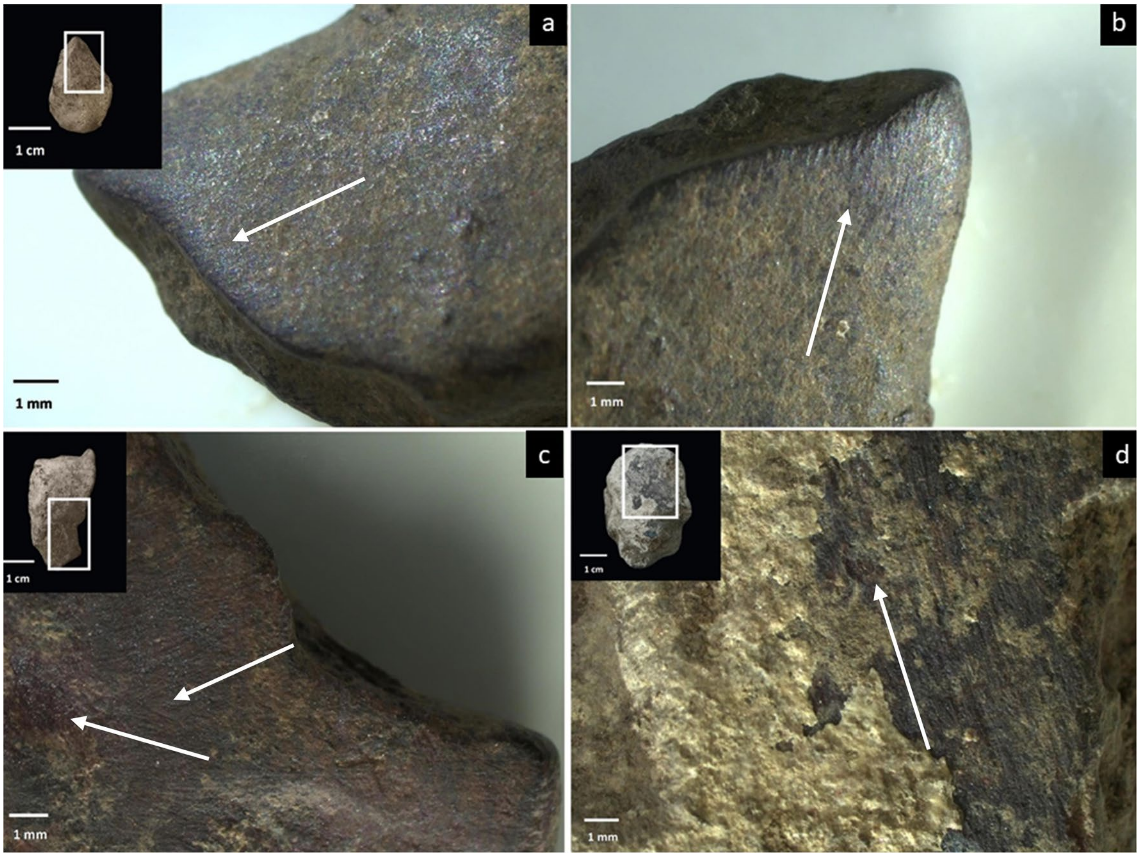


Fig. 7 Selection of utilised specular hematite pieces from the OBP 2018/2019 assemblage, with arrows indicating the direction of striations. **a** and **b** Specular hematite nodule (OBP-4267-1 from YRS) showing striations with polish and smoothing. **c** Hematite piece (OBP-4310-1 from YRS) with microstriations and polish. **d** Specular hematite nodule (OBP-831-2 from GS) with striations on flat surface

group together with a piece collected near OBP (M-OBP-5) in cluster B. All have high Fe values and lower V values and correlations in trace element frequencies, but raw material characteristics and streaks differ. M-OBP-6 a bright red shale fragment collected near the site; it has higher MnO and low Fe proportions and is a clear outlier from the other pieces.

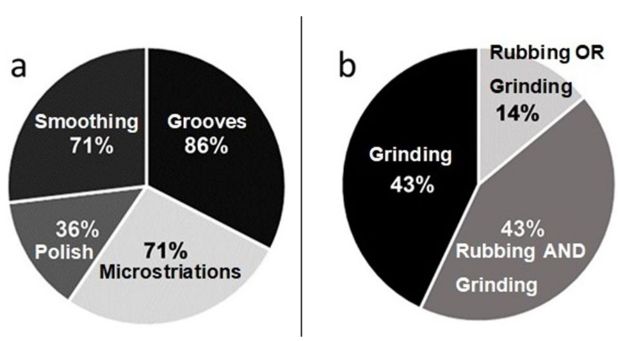


Fig. 8 Ochre utilisation at OBP. **a** Frequency of each use-trace in the OBP utilised ochre assemblage. **b** Percentage of pieces assigned to each activity category

Discussion

Natural wear vs anthropogenic use

It can be difficult to distinguish between certain forms of deliberate use, such as rubbing, and traces of natural wear, such as smoothing from river currents or post-depositional processes. Several of the pieces collected during the ochre sourcing trip in the OBP area, none of which was utilised, bear a close resemblance to the archaeological pieces in the study assemblage in terms of shape and surface features—nodular pieces that look smoothed and polished. Many pieces from the Mason ochre assemblage, analysed by Watts (1998), are also smoothed, have a nodular shape, and were described as "pebbles". Specular hematites form naturally in the sandstones in the Mogalakwena formation via diagenesis, although the exact process through which this occurs has not yet been fully understood or described (Callaghan 1987; R. Wadley pers. comm. 2022). At OBP, local weathering of the sandstone possibly resulted in some specular hematite nodules being deposited into the sediments (van der Ryst 2007; Val et al. 2021; R. Wadley pers.

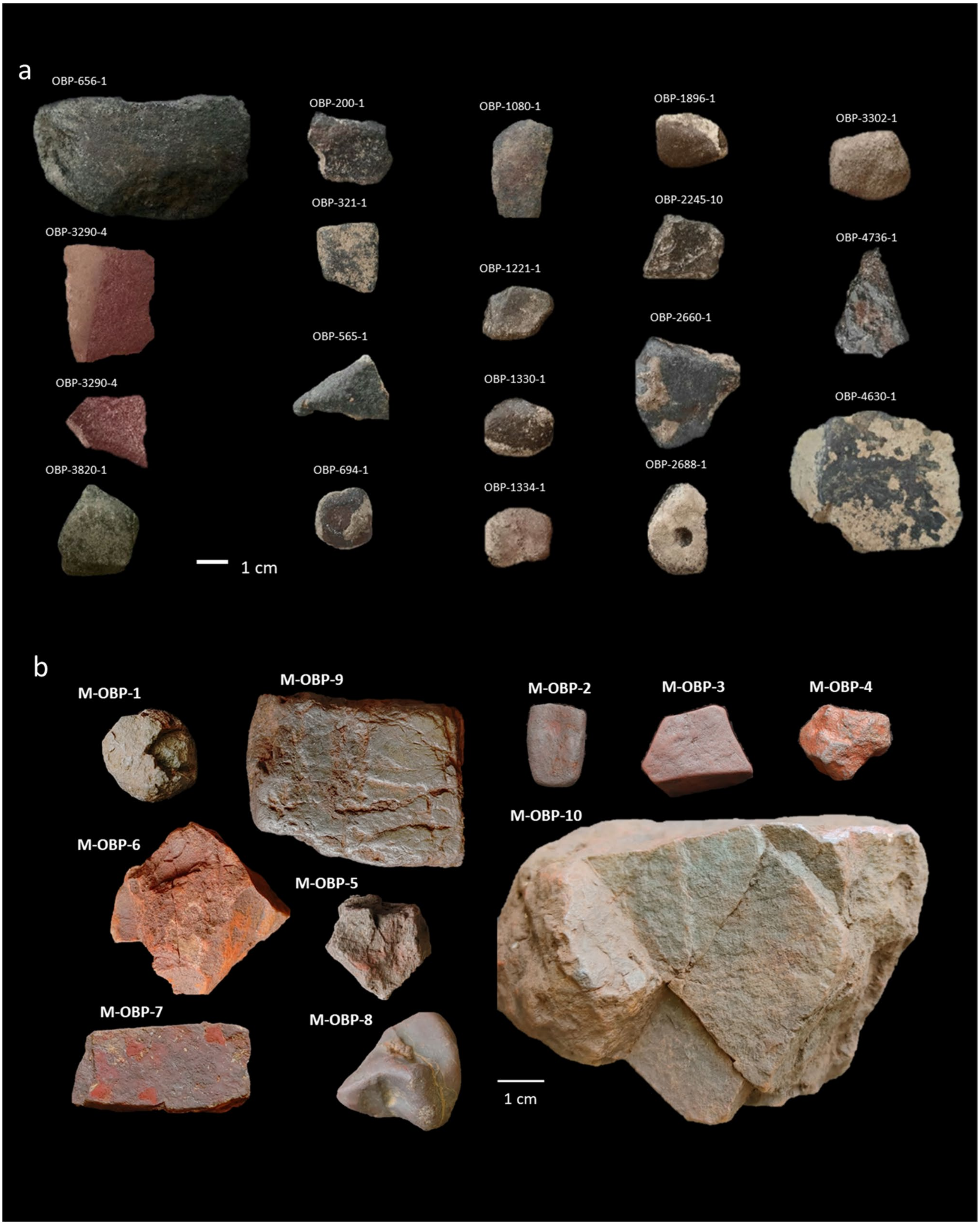


Fig. 9 Ochre pieces analysed to obtain elemental signatures. **a** Archaeological ochre pieces from OBP. **b** Modern geological samples analysed with XRF and ICP-MS to establish elemental composition. Piece numbers correspond to Table 2, Fig. 10, and OR6

Table 2 Physical attributes of ochre pieces and geological samples that underwent elemental analysis (Fig. 9). Groups based on cluster analysis in Fig OR6.1 and OR6.2 and illustrated in Fig. 10b and c. *UNU* unutilised, *U* utilised, *PossU* possibly utilised, "*OBP-*" pieces are archaeological, "*M-*" pieces are modern samples

Piece	Source	Location/layer	Surface marks	Acid prep	Raw material	Piece shape	Streak colour	Cluster
OBP-1080-1	OBP	GS	PossU	Y	Specular hematite; silty	Nodule	Dark red	1
OBP-1221-1	OBP	GS	PossU	Y	Specular hematite; silty	Nodule	Dark red	5
OBP-1330-1	OBP	GS	PossU	Y	Specular hematite; silty	Nodule	Red	1
OBP-1334-1	OBP	GS	PossU	Y	Specular hematite; clayey	Nodule	Dark red	3
OBP-1896-4	OBP	GS	PossU	Y	Specular hematite; silty	Nodule	Red	2
OBP-200-1	OBP	GS	PossU	Y	Specular hematite; clayey	Frag.	Black	3
OBP-2245-10	OBP	GS	PossU	Y	Specular hematite; silty	Frag.	Dark purple	4
OBP-2457-1					Ferricrete conglom; silty	Nodule	Dark red	4
OBP-2660-1	OBP	GS	PossU	Y	Specular hematite; clayey	Nodule	Dark red	1
OBP-2688-1	OBP	GS	PossU	N	Specular hematite; clayey	Nodule	Dark red	2
OBP-2790-1					Ferricrete conglom; silty	Frag.	Red-purple	2
OBP-3120-9	OBP	YRS3	UNU	Y	Mudstone, specular; sandy	Frag.	Dark red	4
OBP-321-1	OBP	GS	PossU	Y	Specular hematite; clayey	Nodule	Dark red	
OBP-3290-3	OBP	YRS4	UT	N	Shale, specular; silty	Frag.	Dark red	5
OBP-3290-4	OBP	YRS4	UT	N	Shale, specular; silty	Frag.	Dark red	5
OBP-3302-1	OBP	YRS5	PossU	Y	Specular hematite; clayey	Nodule	Dark red-purple	4
OBP-3820-1	OBP	YRS9	UT	N	Specular hematite; clayey	Nodule	Red	4
OBP-4630-1	OBP	YRS14	-	N	Hematite; clayey	Frag.	Dark red	4
OBP-4736-1	OBP	GS	PossU	N	Hematite; silty	Frag.	Purple	3
OBP-565-1	OBP	GS	PossU	Y	Specular hematite; silty-clayey	Frag.	Dark red-purple	1
OBP-656-1	OBP	GS	Utilised	N	Specular hematite; silty-clayey	Frag.	Dark red	1
OBP-694-1	OBP	GS	PossU	Y	Specular hematite; clayey	Frag.	Dark red	2
OBP13.15_68	OBP	GS	UNU	N	Hematite; clayey	Nodule	Dark red	2
M-OBP-1	Modern	Near OBP	-	N	Hematite; silty. Magnetic	Nodule	Dark red-purple	A.1
M-OBP-2	Modern	Near OBP	-	N	Specular hematite; clayey	Nodule	Dark red-purple	A.1
M-OBP-3	Modern	Near OBP	-	N	Specular hematite; clayey	Frag.	Red	A.1
M-OBP-4	Modern	Near OBP	-	N	Hematite; silty-clayey	Nodule	Red	A.1
M-OBP-5	Modern	Near OBP	-	N	Shale; silty	Frag.	Dark red	B
M-OBP-6	Modern	Near OBP	-	N	Shale; clayey	Frag.	Red	C
M-OBP-7	Modern	Historic mine	-	N	Shale; clayey	Frag	Dark red-purple	A.2
M-OBP-8	Modern	Historic mine	-	N	Specular hematite; silty-clayey	Nodule	Dark red-purple	A.2
M-OBP-9	Modern	Thabazimbi	-	N	Hematite; silty-clayey	Nodule	Dark red-purple	B
M-OBP-10	Modern	Thabazimbi	-	N	Shale/mudstone; silty	Frag.	Dark red	B

comm. 2023). Additionally, given the proximity of the site to the Riet Spruit (approx. 20 m away), it is probable that some of the pieces were transported by the stream when it swelled, although recent sourcing of the river bed itself was unsuccessful.

When rainfall in the Waterberg is particularly high, valleys and open floodplains become wetlands (Wadley et al. 2021). The Riet Spruit has been recorded to reach the talus of OBP (van der Ryst 2007). Flooding of the shelter and winnowing of the sediments and fine fraction could explain the concentrations of lithics and ochre found below the dripline of the site. The low number of utilised pieces compared to unutilised ochre at OBP (discussed further below) could confirm these processes. Furthermore, some of the lithics and faunal remains recovered from the GS and YRS layers are rounded, which suggests that they have been abraded by water (Val et al. 2021). Natural wear processes have to be taken into account when interpreting the polish and smoothing and nodular shape of the archaeological ochre pieces. The appearance of smoothing and polish are therefore problematic in discerning possible anthropogenic use on the specular hematite found at OBP.

Ochre use and collection at OBP

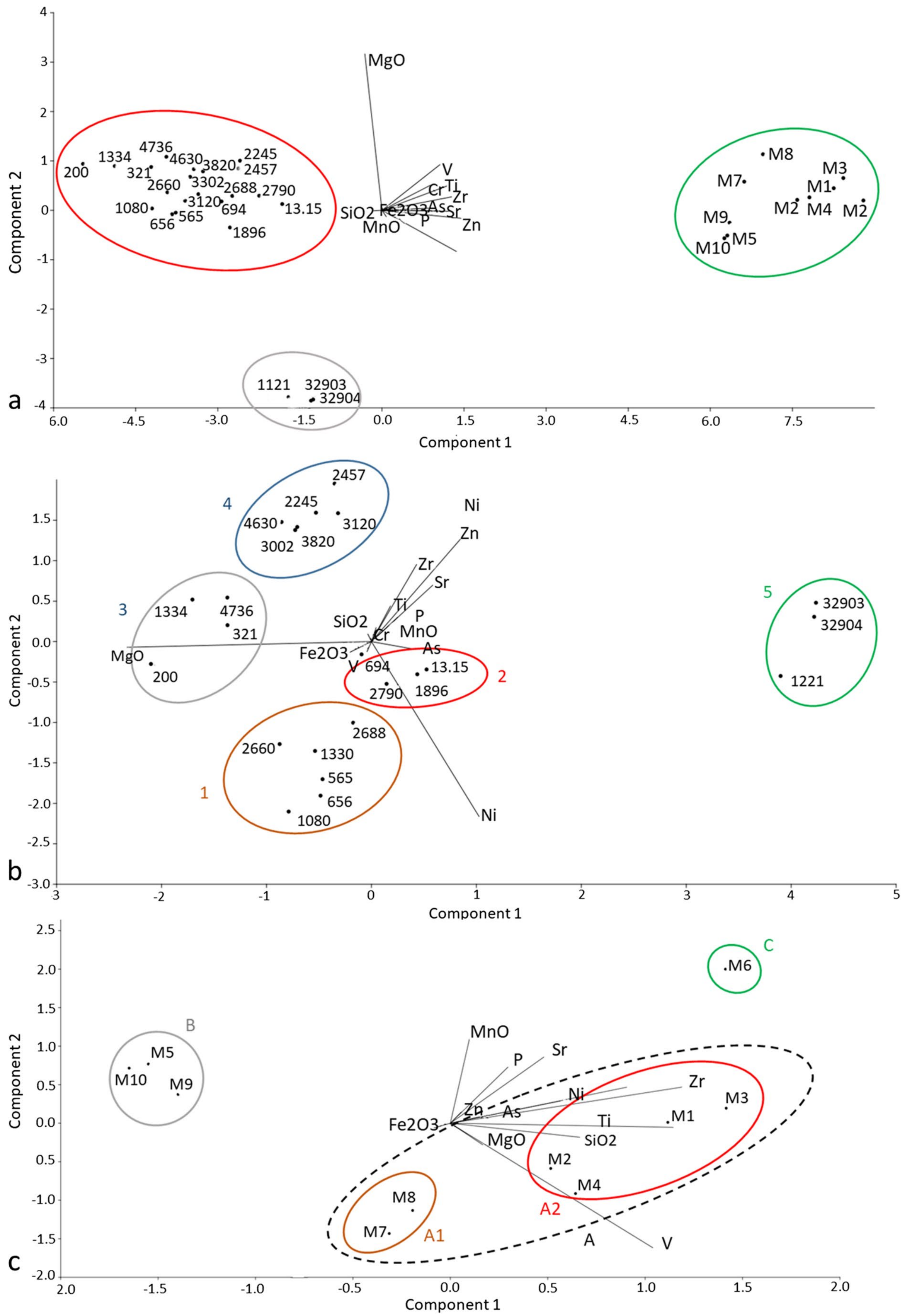
Within the ochre assemblage excavated from OBP during the 2018/2019 excavations, only 3% of pieces are utilised. This is a low percentage compared to MIS 6 and 5 ochre assemblages from other South African sites, where the utilised percentage usually ranges from 10 to 20% (Online Resource 1). Mwulu's Cave, an inland site dated to MIS 5, also in Limpopo Province, is the only site that has an assemblage with a smaller percentage of pieces (>2%) definitely utilised (de la Peña et al. 2019). A low percentage of utilised ochre is also noted for the MSA deposits at the neighbouring site of Red Balloon Shelter (Wadley et al. 2021; Mauran 2023). When considering possible explanations for this low utilised percentage at OBP compared to other sites elsewhere in South Africa, raw material types and colour should be taken into account. There is no significant relationship between ochre use at OBP and raw material (specular hematite), and specular hematite also dominates the unutilised assemblage. This suggests that specular hematite was likely not reserved solely for "special" purposes on account of its specular qualities as has been documented in ethnographic contexts, for example as a hairdressing cosmetic among the Tswana and San (Bleek and Lloyd 1911; Thackeray et al. 1983; Robbins 2016). In the utilised assemblage at OBP, it should be taken into account that the glittering quality of specular hematite draws the human eye in a way which other, less, or non-glittery ochre types do not. This specularity, as well as the dark red colour of the powder produced through use, both appear to have been important factors in determining which pieces were ultimately used.

Yet, this particular type of specular hematite, whether deliberately collected and brought to the site or naturally deposited at the site, was rarely used. It is possible that it was used in ways that do not modify the surface of the pieces, for example as an aesthetic/decorative or symbolic object, instead of being processed for its pigment, but this is pure conjecture. A possible explanation for this low use frequency was that OBP was an ochre procurement and caching site where specular hematite was deliberately collected and stored (Watts 1998, 2002). Comparison between the major and trace elements of the archaeological and (modern) sampled ochre, together with physical characteristics of the pieces, allows us to propose some cautious, preliminary conclusions on ochre provenance at OBP. We are cognisant of the restraints of the pXRF data, as well as using different analytical methods to analyse the archaeological versus modern samples, and hope this study will lay the ground work for further research into ochre provenance around OBP, and into specular hematite and hematite formations in the Waterberg. The two pieces collected from the historic mine on the neighbouring farm have similar elemental signatures to four specular hematite and hematite pieces collected in the drip-line of the site (Table 2; Fig. 10c). In physical appearance, the four pieces are also representative of many of the archaeological pieces in the OBP assemblage—possibly confirming links between the site and the historic mine source. The two pieces from Thabazimbi are elementally and physically distinct from these; Thabazimbi is therefore an unlikely source of the ochre at OBP.

Physically, the specular hematite types present at OBP are also found at neighbouring archaeological sites, Red Balloon Rock Shelter (Wadley et al. 2021) and possibly North Brabant (Schoonraad and Beaumont 1968). Not only was a very small amount of the specular hematite at OBP actually utilised, but the pieces that were utilised were not utilised intensively. Most pieces in the utilised assemblage have only one worked surface, and many of the unutilised pieces are fragmented or broken. It is possible that these pieces were only lightly utilised or broken to test their quality. This may further suggest that the raw material was available in such abundant quantities that the users could afford not to utilise pieces more extensively. The use-traces on the few utilised ochre pieces suggest grinding activities. Grinding or abrading of ochre, presumably to obtain powder, is one of the most predominant use-wear activities among ochre assemblages from both coastal and inland South African sites (Online Resource 1). The fact that the specular hematite found at OBP is hard compared to other ochre types also suggests that grinding would have been the most likely (i.e. the most successful) method of producing pigment powder. However, given that many pieces are fragmented, it is also possible that crushing and/or pounding may have been a likely method of processing, but the absence of use-traces indicating percussion on most of the pieces in the study assemblage means that this is difficult to corroborate.

Whether the specular hematite pieces were collected and "tested" before eventually being transported to other sites is unclear. Evidence of this would have to come from provenance studies of other similarly aged sites in the area. This then raises the question of *which* other sites this specular hematite would have been taken to and used at. Bushman Rock Shelter, Mwulu's Cave, and Red Balloon Rock Shelter are some of the few other sites in Limpopo with MIS 5 assemblages in which specular hematite occurs; in all of these assemblages, only a small number of pieces are utilised (Online Resource 1). It is of course possible that the ochre was processed and used up entirely, in ways which left no archaeologically visible evidence. The large quantities at OBP could certainly suggest a substantial demand and desire for this raw material. However, the presence of specular hematite at these other sites can be more parsimoniously explained by the fact that they are located within the same geological context as OBP.

Additionally, it is important to note that the rock art at the site is mostly orange, bright red, and yellow varieties


a
Component 1
Component 2
MgO
V
Cr
Ti
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Fe2O3
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200
1334
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4630
3820
2245
2457
321
2660
3302
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2790
1080
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694
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A
V

◂**Fig. 10** PCA of log-transformed major and minor elemental data from the OBP and geological ochres (elements included Fe, Si, Mn, Mg, P, Ti, V, Cr, Co, Ni, Cu, Zn, As, Rb, Sr, Zr, Sn, Sb). **a** PCA of major and trace elements in the archaeological and geological samples. "M" for modern; numbers correspond to piece numbers in Table 2. **b** PCA of the OBP archaeological sample pXRF signatures, with clusters indicated (Fig. OR6.1). **c** PCA of the XRF and ICP-MS majors and trace elements of the modern geological samples (Fig. OR6.2)

and seemingly different in colour from the dark reds and purples produced by the specular hematite that dominates the assemblage. This suggests that the people using the site during the Later Stone Age and Iron Age periods may have been collecting ochre from another source, which we have not been able to locate yet. This does not exclude the possibility that other colours may have been collected from a different area of the same sources, that taphonomic processes have affected the paint or that binders added to the pigments may have changed the colours. Further sourcing studies and research into comparisons between the historic iron ore mine and the rock art paint made by more recent hunter-gatherer and herder groups will be useful in understanding the fuller story of habitation and use of ochre at the site through time.

The nature of the specular hematite pieces in the archaeological assemblage, whether they were deposited near the site via the Riet Spruit, found as loose nodules in the sand or possibly collected from—as yet unknown—source areas, suggests that instances of collection were opportunistic. Users probably picked up nodules of specular hematite, which already occur naturally in the area, because these were available but perhaps also because they were attractive first on account of their specular qualities and then, after being gently utilised to reveal their colour, on account of their dark red streak colour, and utilised them. That specular hematite was available in abundance but was not more extensively utilised suggests that the ethnographically documented tradition of using specularite for "special" or cosmetic purposes does not extend this far back into the MSA at this site.

Conclusion

The recently excavated OBP ochre assemblage constitutes a unique MSA ochre assemblage in terms of raw material type and quantity, as well as usage patterns. There are few sites dated to over 100,000 years old in southern Africa that have ochre collections of this volume of specular hematite with such a low percentage of utilised pieces. The use of these dark-red pieces, mostly in the form of grinding or abrasion actions that would have produced a pigmented, micaceous powder, was not intensive and most pieces only have small areas of use. We have demonstrated that some of the specular hematite and other ochre varieties were brought to and used at the site. Site formation processes including local degradation of the surrounding sandstone matrix, flooding events of the nearby Riet Spruit, and the geology of the area almost certainly played a role in the volume of specular hematite found at OBP and the surface morphology of these pieces.

The preliminary geochemical and statistical analyses here—combined with physical analyses—have highlighted the local ochre types and location of a primary and some secondary ochre sources around OBP. Some of these ochre sources are geologically similar to some of the semi-quantitatively tested archaeological pieces. Physicochemical analyses suggest that the MSA users were most probably collecting ochre opportunistically around the site and bringing it back, occasionally to produce powder, or possibly collecting from outcrops, such as identified in the historic ochre mine ~10 km away from the site. Other ochre sources suggested in previous research, such as Thabazimbi, do not correspond geologically with the archaeological ochre analysed and it seems unlikely that this source—100 km away—was used by the MSA ochre users at OBP. The suggestion that OBP was used as an ochre caching and "factory" site that formed part of some sort of exchange network during the MSA could not be supported by the current data. Further in-depth analysis of the trace element properties of the archaeological ochre, combined with intra-site comparisons and additional ochre sourcing data, is necessary to draw conclusions on ochre provenance. This paper contributes to the body of knowledge on specular hematite collection and use during the Middle and early Late Pleistocene in southern Africa.

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Author contributions All authors contributed to the writing and editing of the manuscript. SW and TH conceived the research question. AV and PdlP allowed access to material and site, and facilitated sample sourcing. JC examined the archaeological assemblage, cleaned pieces, performed pXRF, and analysed the data, under the supervision of TH and SW. TH submitted samples for geochemical analysis and analysed data. JC and TH co-wrote the first draft. AV, PdlP, and SW contributed to the writing and editing of the manuscript, and have read and approved the final manuscript.

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Data availability The authors confirm that the relevant data supporting the findings of this study are available within this article and supplementary materials, and any further data is available upon request.

Declarations

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