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Ochre communities of practice in Stone Age Eswatini

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Our species and other hominins have used earth mineral pigments since at least ~500,000 years ago, if not earlier. Its preservation and ubiquity within archaeological records across sub-Saharan Africa are well documented, but regional-scale networks of mineral selection, mining, transport, and use is an underdeveloped field. Here, we present a framework for interpreting regional variations within an overarching ochre-behavioral community of practice. Deep-time records of ochre provisioning span the final Middle Stone Age and Late Stone Age in modern day Eswatini, revealing longstanding cultural continuities in the intergenerational transmission of shared knowledge on landscapes, geology, and the desired physicochemical properties of mineral pigments. These communities of practice did not develop in isolation, and were part of a wider system of relations that were influenced and mediated by social interactions, such as technological learning, seasonal traveling, material culture exchange, and symbolic expression. We use compositional analyses to determine localized ochre procurement strategies and long-distance transport across a network of fifteen archaeological sites and mineral resources. Newly refined chronologies from Lion Cavern at Ngwenya using optically stimulated luminescence dating also reaffirm its antiquity as the oldest known evidence for intensive ochre mining worldwide (~48,000 years ago).

Identifying evidence for long distance mobility and social interactions is at the forefront of archaeological research in the Middle Stone Age and Late Stone Age (MSA/LSA) of sub-Saharan Africa[1–5]. The analysis of earth mineral pigments, or ochres, has played a role in interpretations of cognitive behaviors[6,7], is linked to rock art and symbolic expressions[8], and is associated with tool hafting adhesives[9,10], bedding[11], and several examples of compositional analysis and provenance[12–17]. But, it has rarely been conceptualized in a framework that advances identifying ochre communities of practice[18–21]. Lave and Wenger first introduced the concepts of communities of practice and situated learning to describe how shared knowledge and traditions emerge within, and are reinforced and communicated across, social

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groups[19,22]. Learning a body of knowledge or skill is an inherently social practice that can occur at different nodes within a cultural framework, whether formal, passive, embedded, peripheral, or restricted. These concepts have been applied to understand the social dimensions of lithic technologies[23], ceramic production[24,25], and early metallurgical practices[21]. Here, we apply the concept to the *chaîne opératoire* of ochre behaviors, from mineral selection through to its end use or deposition in at archaeological sites. The social practices that motivated people to seek out and use ochre were group activities such as seasonal traversal through different ecological environments, teaching and learning on where to locate geologic formations that bear raw materials with desirable characteristics, and the techniques necessary for mining and processing of ochres with different material properties. The earth mineral-based mixtures that were produced by people signify material and cognitive complexity, often including ingredients that reflect traditional knowledge of mineral and biological resources used in the deep past, such as iron and manganese oxides, and also composite mixtures that included wetting agents and binding media, such as milk, fat, blood, and plant resins[26,27]. Those components were selected for their properties (color, durability, adhesion, resistance to weathering), but also for sociocultural reasons, such as spiritual potency and the geographic origins of minerals. The ochre *chaîne opératoire* was an inherently social activity that existed as part of a broader system of relations that had significance to people living in the MSA and LSA. Here, we show evidence for long term, regional-scale behavioral stability in ochre provisioning and transport, but also, distinctive site-specific variations in procurement-based technological choices. The diversity and complexity of these behaviors illustrates precisely how an overarching, shared cultural identity surrounding ochres can unify various localized and contextually-contingent communities of practice[21].

Ochre is widely recognized as a red, yellow, or violet pigmentaceous earth mineral, often conflated with manganese oxide (black ochre), and its importance throughout human history is indisputable[28,29]. It survives deep time records[7], was extracted by intensive mining practices[30], transported great distances[3], used in symbolic and mortuary expression[31,32], underwent processing to enhance its properties and performance in complex paint mixtures[33]. It continues to retain widespread cultural importance in many descendant communities today[34–39]. Yet, the differences between ochres are not always obvious, and pigments that appear the same in color and texture often have distinct physicochemical properties[40]. Scientific analyses are needed to reconstruct the *chaîne opératoire* of ochre behaviors, such as shared knowledge on its provisioning, the selection or enhancement of ochre with particular physical qualities, and traditional techniques used to turn powdered ochres into composite paints. Here, we present a framework for understanding ochre communities of practice in the final MSA and LSA of Eswatini using a multi-proxy approach. We used a combination of analytical techniques to determine the provenance and physicochemical properties of ochre assemblages from ten sites, and raw materials collected from nine geological source localities (Fig. 1, Supplementary Table 1, Supplementary Notes 1.0 and 2.0). Following the local traditional nomenclature and geology of Eswatini, we use the term ochre to describe all earth-derived mineral pigments, with specific reference to red or yellow ochre (iron oxide and iron-rich clay), black ochre paint (including magnetite, manganese oxides, or charcoal) and white ochre paint (kaolin).

Eswatini is home to a number of deeply stratified MSA/LSA rock shelter and open-air sites with sizeable assemblages of ochre and lithic artifacts and over fifty rock art sites. Recently, the significance of those assemblages, most excavated in the 1980's by Price Williams[41], was

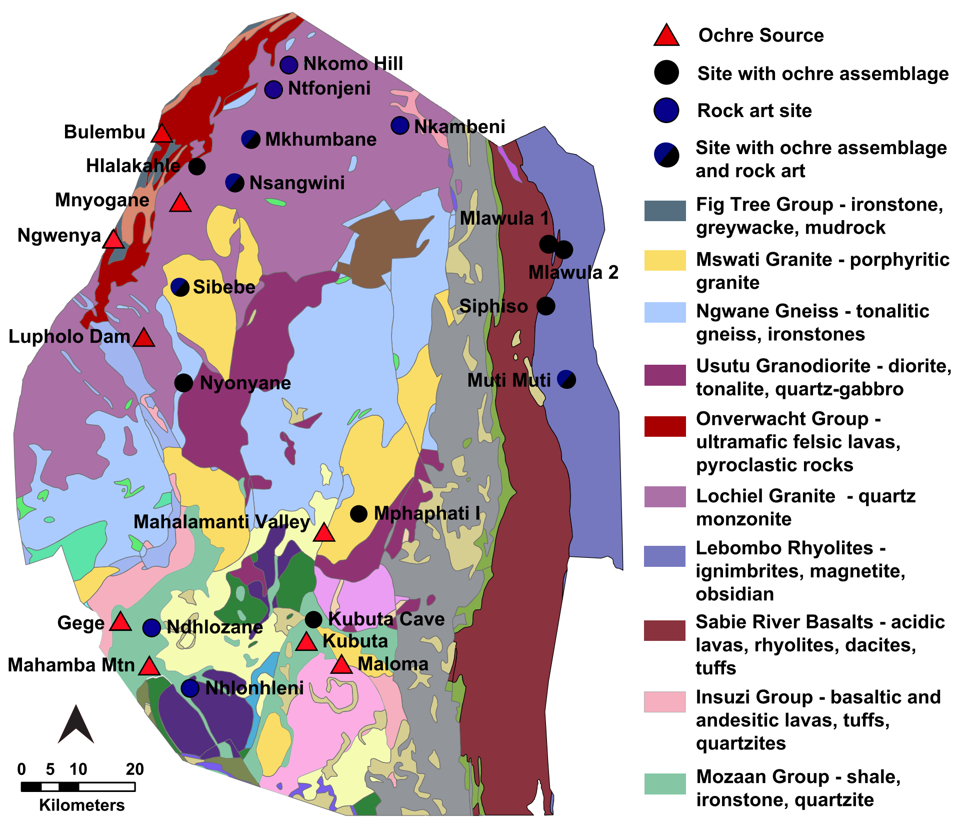

Fig. 1 | Geologic map of Eswatini. Key archaeological sites and earth mineral pigment source locations are shown. Sites Nkomo Hill, Ntfonjeni, Nkambeni, Ndhlozane, and Nhlonhleni are shown to illustrate the locations of rock art relative to raw material sources.

highlighted for contributing to new insights on debates surrounding the MSA/LSA transition[42]. Eswatini also hosts Lion Cavern, a high-elevation (highveld) site situated atop the Ngwenya Massif ironstone and hematite deposit, which is presently the oldest direct evidence for ochre mining during the MSA[43,44] (Fig. 1). It first received archaeological attention in the 1960's by Dart et al., among others[45]. The current study focuses on ochre artifacts and raw materials from fifteen sites and sources that span the final MSA and LSA, through to the Iron Age (Fig. 2, Supplementary Table 1, Supplementary Data 1, Supplementary Notes 1.0 and 2.0). For decades, archaeologists working across the surrounding region have collectively assumed that any ochre present in artifact assemblages in the area[46], including nearby Border Cave in South Africa, likely came from Ngwenya[47–50]. In the absence of geochemical data this would be a reasonable assumption as Ngwenya ochre is high in quality and desirable properties for creating pigment (rich red hue, fine-grained with minimal impurities, contained localized brilliant specularite), is a highly visible landmark, and was present in massive abundance prior to modern mining activities[47,48,50,51]. Excavation records documented at least four prehistoric mining sites along the Ngwenya Massif: Lion Cavern, Banda Cavern, Castle Cavern, and Castle Quarry, the latter two of which were subsequently destroyed during industrial mining[44]. In this study we used the optically stimulated luminescence technique to produce new dates foe Lion Cavern that reaffirm its status as the oldest known intensive ochre mining to date at ~48 kya (Supplementary Table 1, Supplementary Data 1). For the ochre provenance component of this study we systematically surveyed outcrops along ~22 km of the Ngwenya Massif, including the prehistoric quarry sites at Lion Cavern and Banda Cavern, and localities at the northern (Bulembu) and southern (SW Spur) extents of the formation.

In addition to the ore deposits across Ngwenya Massif, we surveyed six sources of iron (FeOx) and manganese oxide (MnOx) elsewhere in Eswatini, including the Maloma ore deposit, Kubuta manganese ore field, the Gege goethite-magnetite deposit, the secondary deposits of banded ironstone at Mahamba Mountain in the south, the Mnyogane and Lupholo Dam lateritic-saprolitic clays in the western middleveld, and one locality around Siphiso, located along the Lebombo Mountain range in the east. A total of 173 samples were analyzed from the source locations (see Supplementary Notes 1.0).

The ochre artifact assemblages included those from two deeply stratified occupational sites, Sibebe (MSA/LSA) and Siphiso (LSA), and smaller or shorter-term occupation sites and rock shelters including Nyonyane, Nsangwini, Mlawula (1 & 2), Muti Muti, Ndhlozane, Hlalakahle, and Mphatiphati I (Supplementary Table 1). Supplementary Table 2 list the sites, numbers of ochre artifacts, and source materials included in the study. Four of the sites, Sibebe, Muti Muti, Nsangwini, and Ndhlozane, have extensive panels of monochrome red or polychrome (red, violet, black, brown, yellow, white, orange) rock art motifs. All artifact assemblages, with the exception of surface finds from Muti Muti and Ndhlozane, were drawn from previously excavated collections archived at the Eswatini National Museum (Lobamba). The ochre assemblages were organized into one of seven litho-typological groups based on characteristics such as color, relative grain size, texture, hardness, morphology, and presence of non-ferrous inclusions (see Supplementary Notes 2.0 and Supplementary Table 2). Our selection of artifacts for characterization was based on the litho-typological groups, with effort to incorporate an even distribution across site temporal phases wherever possible, and to minimize impact to the assemblages by avoiding the destruction of whole artifacts or those exhibiting usewear, as defined by Hodgskiss and others, as grinding, rubbing or scoring[52,53]. A total of 361 of a possible 931 artifacts were selected for compositional analysis. The artifacts were granted export permits by the Eswatini National Trust Commission and transported to the Archaeometry Laboratory at the University of Missouri Research Reactor for formal analysis by one or more methods of portable X-ray fluorescence (pXRF), neutron activation analysis (NAA) and laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) for trace element analysis, and X-ray diffraction (XRD) or Raman spectroscopy for mineralogical identification. The methods and experimental conditions are detailed in Supplementary Notes 3.0 and 4.0. All original data and summary tables are provided in Supplementary Data files 3-12.

Results

Differentiating ochre sources: the results of the ochre source characterization revealed several key patterns. Supplementary Fig. 6 shows representative specimens from each source. The sources across Eswatini have compositional signatures that satisfy the provenance postulate, a fundamental concept for all sourcing studies, that the elemental and mineralogical variability between sources or compositional groups must exceed the variability within sources or compositional groups[54]. Supplementary Fig. 18 is a scatterplot showing the results of a principal component analysis (PCA) of the trace element analysis of iron oxide source materials by NAA. Supplementary Data 3 and 4 detail the results of all elemental analyses and statistical procedures. The largest deposit, the Ngwenya Massif, showed some intra-source variation. The Ngwenya-HG (high grade ore), which included massive blue ironstones at Lion Cavern, consolidated deep red hematite at Banda Cavern, and nodules of specularite at the southernmost extent of the deposit, all had chemically consistent compositions despite their variations in mineral morphology. Those localities were situated at the highest elevation points across the top of the Ngwenya Massif, following the main strata of the Fig Tree hydrothermal ironstone formation. At an approximately 25 m elevation drop, exposed only by modern mining activity, was a layer of ferruginous shale that was chemically distinct from the Fig Tree ironstones. We also identified localized nodules of manganese oxide at Banda cavern, where the mineralization is a psilomelane type; high MnOx (30-60%) with high K and Ba (6–10%). Another variation observed along the Ngwenya Massif was Bulembu, an outcrop that resurfaces ~22 km north of the Lion Cavern area. There, the ochre is higher in silicon (up to 23%), and there are localized enrichments of manganese oxide (braunite, $Mn_7O_{12}Si$) with elevated quartz (up to 10%).

Other sources shown in Fig. 3 include Maloma and Mnyogane. The Maloma deposit consists of black ironstones (20–40% Fe), with minor silicon, aluminum, manganese, and potassium. XRD data confirmed the presence of magnetite, quartz, pyrolusite, and manganite. The Mnyogane samples consist of the iron-rich lateritic and saprolitic clay sources located in the western middleveld. These materials are friable, high in mica and kaolinite, and the deposits are still collected today by local communities for medicinal and ceremonial use (Supplementary Notes 1.4 and 1.5).

Sourcing Ochre Artifacts: nine distinct compositional groups were identified within the artifact assemblage dataset (Fe- groups G1/G1b to G5), with 37 of the 337 artifacts remaining unassigned to a group. Figure 4 shows examples of artifacts from each group. Supplementary Data 12 summarizes the groups, organized by archaeological site and time period. The artifact groups were determined using a combination of iterative, pattern-recognition techniques including PCA, cluster analysis, and element-pair bivariate plots, and cross-evaluated using Mahalanobis distance equations. This bottom-up multivariate approach was applied to the NAA (FeOx) and the LA-ICP-MS (MnOx) datasets independently for reasons described in Supplementary Notes 3.0. Supplementary Data 2 list the group assignments for each sample by method and an assigned group. Following this, the FeOx artifact groups were tested against the sources by combining them in PCA and cluster analysis, and using Mahalanobis distance to calculate membership probability scores (Supplementary Data 4 and 7).

Figure 5a shows a scatterplot illustrating the PCA of NAA results for the FeOx artifact groups. Note that the groups that separate along

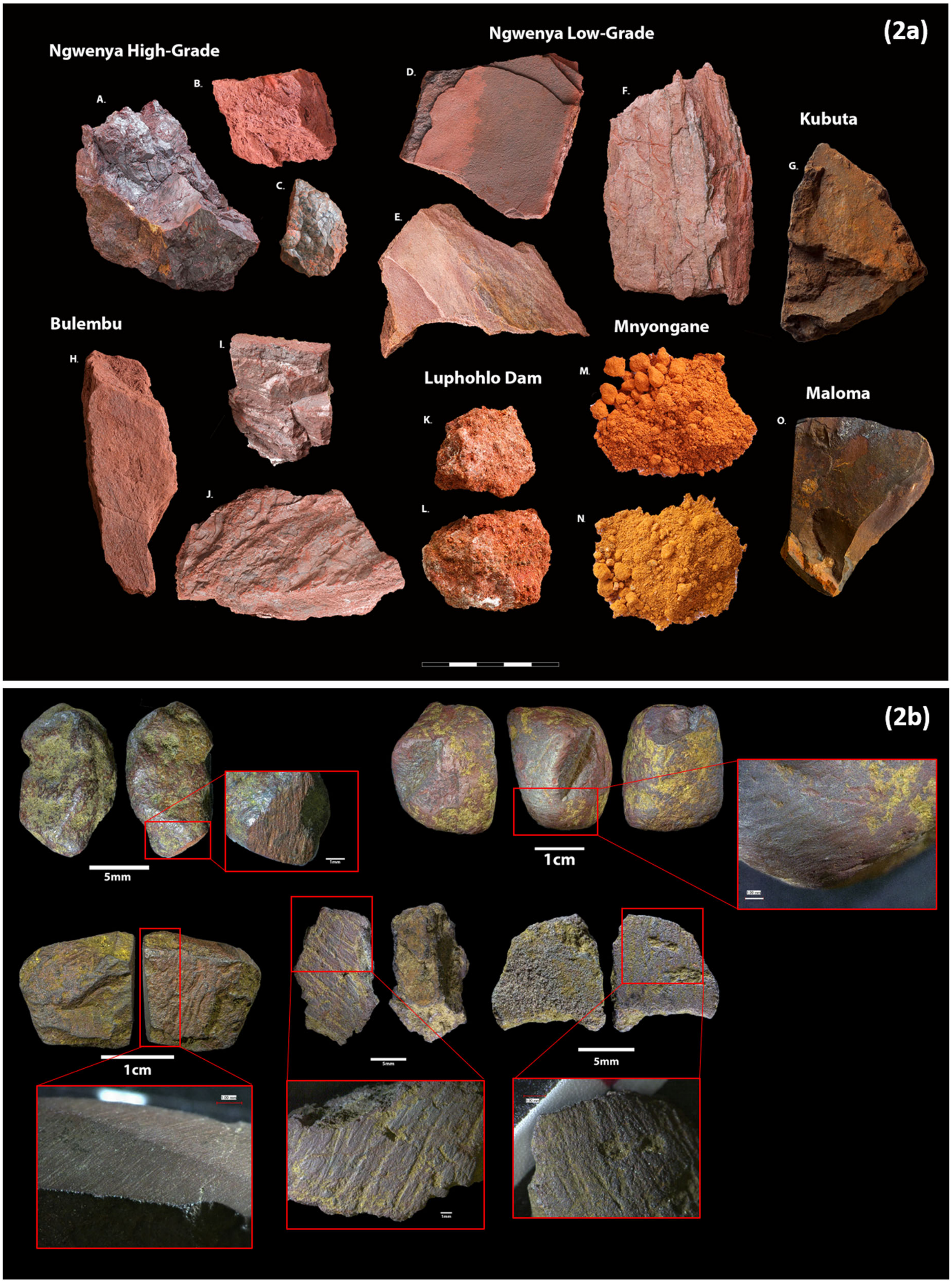

Fig. 2 | Examples of ochres from sources and archaeological sites. a Selected hand specimens of samples from sources included in this study. Ngwenya high grade (A–C), Ngwenya low grade (D–F), Kubuta (G), Bulembu (H–J), Lupholo Dam (K, L), Mnyongane (M, N), Maloma (O). Scale = 1 cm. **b** Examples of modified ochre artifacts from Sibebe, showing evidence for modification, including grooves created during grinding or scoring.

PC1, indicated by a dashed line, coincidentally reflect the relative geographic regions where those artifact groups are dominant. For instance, Fe-G1, Fe-G4, and Fe-G5 are the dominant types at sites located in the western highveld and south-western middleveld, whereas FeOx groups Fe-G2a to Fe-G2d, and Fe-G3 are situated in the eastern Lebombo Mountain range of Eswatini, which runs north-south and broadly defines the geopolitical borders with Mozambique and South Africa. To further refine the groups, they were divided by sub-

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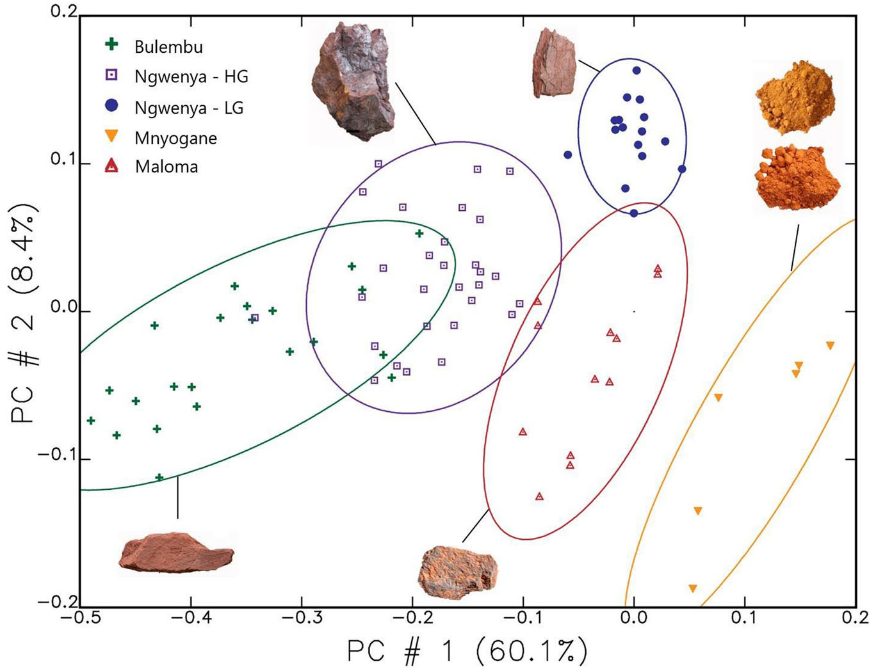

Fig. 3 | Results from the statistical analysis of NAA data on ochre sources. Scatterplot of principal component 1 (PC1 60.1%) vs principal component 2 (PC2 8.4%). The distribution of source materials from Ngwenya (high grade, HG or low grade LG), Bulembu, Maloma, and Mnyogane (including Luphohlo Dam), are shown. Ellipses are drawn at 90% confidence. Source data are available in Supplementary Data 3.

region and compared to available source materials. Figure 5b shows the artifact groups from the western region, Fe-G1/G1b, Fe-G4, and Fe-G5, projected against the confidence ellipses representing the FeOx sources. Figure 6 shows the distribution of the eastern-dominant compositional groups. No ochre mining sites have been located in the Lebombo Mountains, but we have inferred the location of Fe-G2a based on the analysis of finds of the same raw material naturally occurring around the vicinity of Siphiso.

Fe-Group 1/Ngwenya-HG**:** this group has 82 artifacts of high-purity FeOx. Fe content ranges from 50 to 66% Fe, with Al up to ~3% and Si up to ~3%, with depleted trace elements. XRD results showed hematite, minor magnetite, quartz, and Al_2O_3. Statistical comparison to source materials showed a very strong probability (>80%) that Fe-G1 artifacts were collected from one of the outcrops at Ngwenya: Lion Cavern, Banda Cavern, or SW Spur (or conceivably, Castle Quarry or Castle Cavern). Most of those artifacts were recovered from Sibebe ($n = 66$). This Ngwenya ochre is also the dominant type at Nyonyane, Hlalakahle, and Nsangwini, as well as in smaller quantities at the Lebombo montane sites of Siphiso ($n = 5$) and Mlawula ($n = 2$).

Fe-Group 1b/Bulembu: this group of 25 artifacts is similar in composition to the Ngwenya-HG source, but is higher in Si (~12%) and Mn (~0.64%), and relatively depleted in Ba, K, and Na. Group membership probabilities of these artifacts show moderate to strong scores for the Bulembu outcrop of the Ngwenya Massif, located ~22 km north of the mining sites around Lion Cavern. This type was most often found at Sibebe ($n = 22$), but also occurs in low numbers at Nsangwini ($n = 1$), and Nyonyane ($n = 1$), and at the eastern site of Siphiso ($n = 1$).

Fe-Group 2a/Siphiso area: this group consists of 20 artifacts, seven of which are from the Siphiso excavations, and five of which were collected in the vicinity of the Siphiso. This indicates that Fe-G2a is a signature that is present around Siphiso, however, it is not the main type within its artifact assemblage. This type is also dominant among the surface finds at Muti Muti ($n = 7$), which occurs along the same strata of the Lebombo Mountain range rhyolite band as Siphiso (~12 km south), further reinforcing that Fe-G2a is likely a locally available raw material. It also occurs in one instance at Ndhlozane shelter in the western middleveld, suggesting transport >80 km. The elemental data for Fe-G2a are consistent with iron-rich volcanogenic-type deposits that are characteristic of the Lebombo Mountain range (30–50% Fe, elevated Zr, Hf, and Al). The XRD results showed peak patterns for iron silicate, periclase (MgO), Al_2O_3, quartz, hematite, and minor FeK_3O_2.

Fe-Group 2b/Eastern Region: this group has 24 artifacts, 22 of which are from various strata throughout Siphiso's occupational sequence. Two samples of this type were identified in the surface finds at Muti Muti. This group has a composition similar to Fe-G2a, but is differentiated by its higher concentrations of K, Si, Ba, Ca, Eu, Mn, and Sr, and depleted Cr and V. Artifacts in this group had markers for goethite, K_2O, and distinctive Al-K-Mg alkali compounds (Supplementary Notes 4.0). We have not located this source but infer that it is local to the lowveld or Lebombo Mountains as it only occurs at Siphiso and Muti Muti. Its high Ca and K contents suggest a possible association with oxidized deposits from the Sabie River basalt formation located a short distance west of Siphiso and Muti Muti (<5 km).

Fe-Group 2c/Eastern Region: this group of 25 samples is entirely from the Siphiso assemblage. It varies in Fe concentration ~30–40%, with elevated Mg and K (up to 2.9%). XRD results showed patterns for muscovite-like mica ($Al_3KO_{12}Si_3$), pyrophyllite [$Al_2Si_4O_{10}(OH)_2$], almandine ($Al_2Fe_3O_{12}Si_3$), K_2O, periclase (MgO), quartz, and hematite. We do not know this source location, but based on principles of the criterion of abundance, where the dominant compositional group present at a site is mostly likely a locally-available material, would infer that it is in the vicinity of the eastern lowveld or Lebombos.

Fe-Group 2d/Eastern Region: this is the largest group represented in the Lebombo Mountains, consisting of 66 artifacts; 62 of which are from Siphiso, one from Mlawula, and the remainder from western sites of Nyonyane ($n = 1$) and Sibebe ($n = 2$). It is the preferred material by inhabitants occupying Siphiso consistently through the entire LSA sequence. It is a distinct type of high-purity magnetite with trace element concentrations (K, Zr, Sr, Rb) suggesting a volcanogenic origin in the Lebombo Mountains.

Fe-Group 3/Eastern Region: this group has 18 samples of a titaniferous magnetite, up to ~31% Fe and 19.9% Ti. It is mainly present at eastern sites Mlawula ($n = 7$), Siphiso ($n = 9$), and Muti Muti ($n = 1$), but

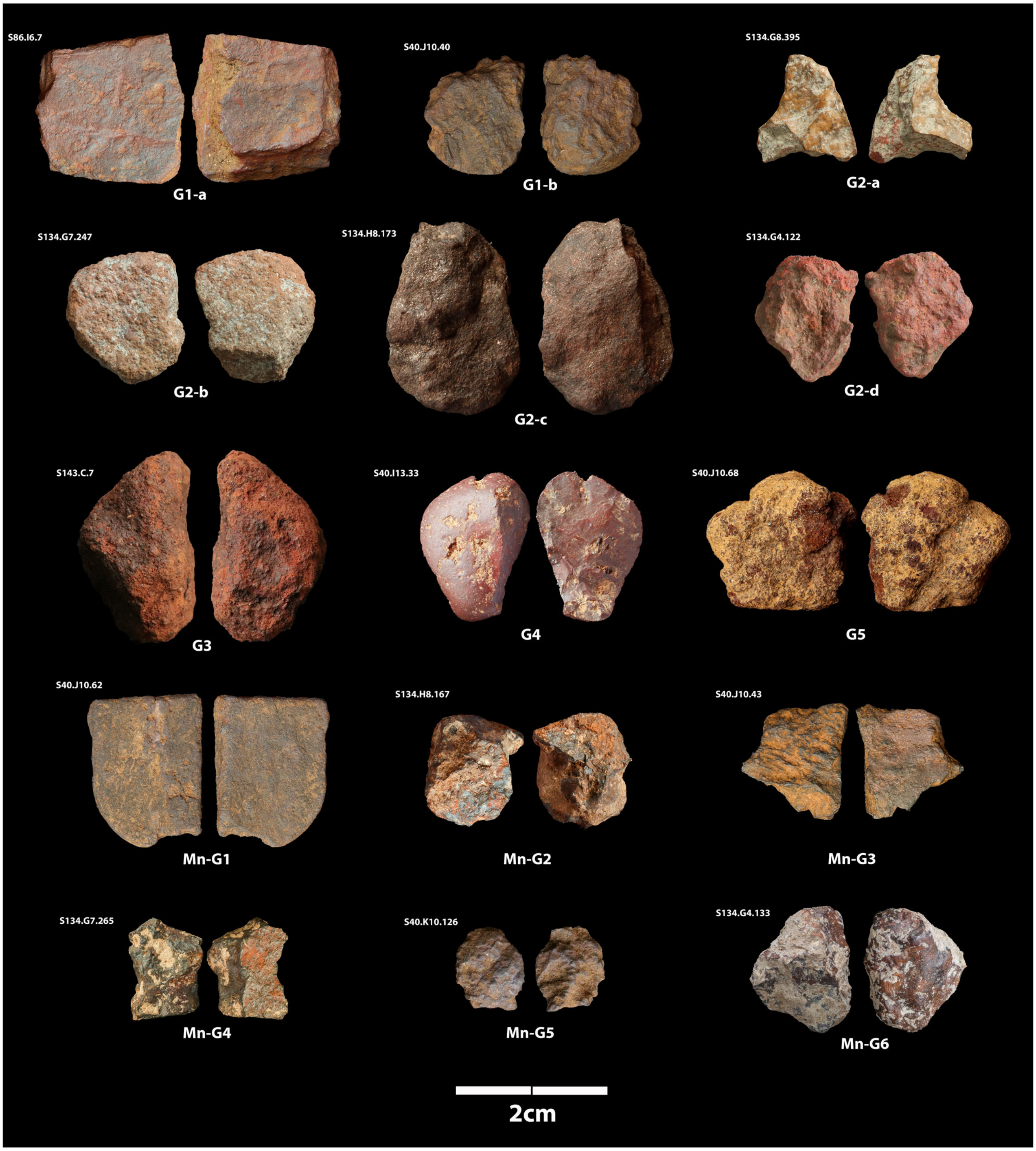

Fig. 4 | Exemplar artifact examples representing each compositional group. Examples of artifacts from each iron oxide (G1-G5) and manganese oxide group (Mn G1-G6).

also in one instance at Sibebe. XRD results showed the presence of anatase (TiO_2), magnetite, and minor hematite, albite ($NaAlSi_3O_8$), and quartz. The sole source of titaniferous magnetite in the region is located within ~20-25 km of Mlawula, Siphiso, and Muti Muti, near the Mbuluzi, River, which discharges into the Indian Ocean as the Rio Maputo in Mozambique[55].

Fe-Group 4/Unknown locality: this group consists of 17 artifacts, fifteen of which are from Sibebe and two from Siphiso. The samples are high in iron (~ 20-50%) and Al (up to 12%), and XRD results suggested magnetite, hematite, quartz, and minor Al-O-Fe compounds which occur in iron-rich argillaceous or ultramafic deposits (Supplementary Note 4). It is unclear where this type originates, but it is a poor statistical match to any of the western highveld sources.

Fe-Group 5/Mahalamanti Valley: this group consists of 14 samples: Sibebe ($n = 4$), Siphiso ($n = 5$), Nyonyane ($n = 1$), Hlalakahle ($n = 1$), and Muti Muti ($n = 1$). The two remaining samples provide key context for its source locality; they are ochre nodules recovered from an iron working kiln at the Iron Age site Mphaphati I, located ~4 km from the main iron ore source in Mahalamanti Valley[56]. Raw iron ore, which included nodules of red hematite, was recovered in great quantity from Ohinawa's 1990's excavations[56]. Those ores were the local material collected and transported to Mphaphati I for smelting,

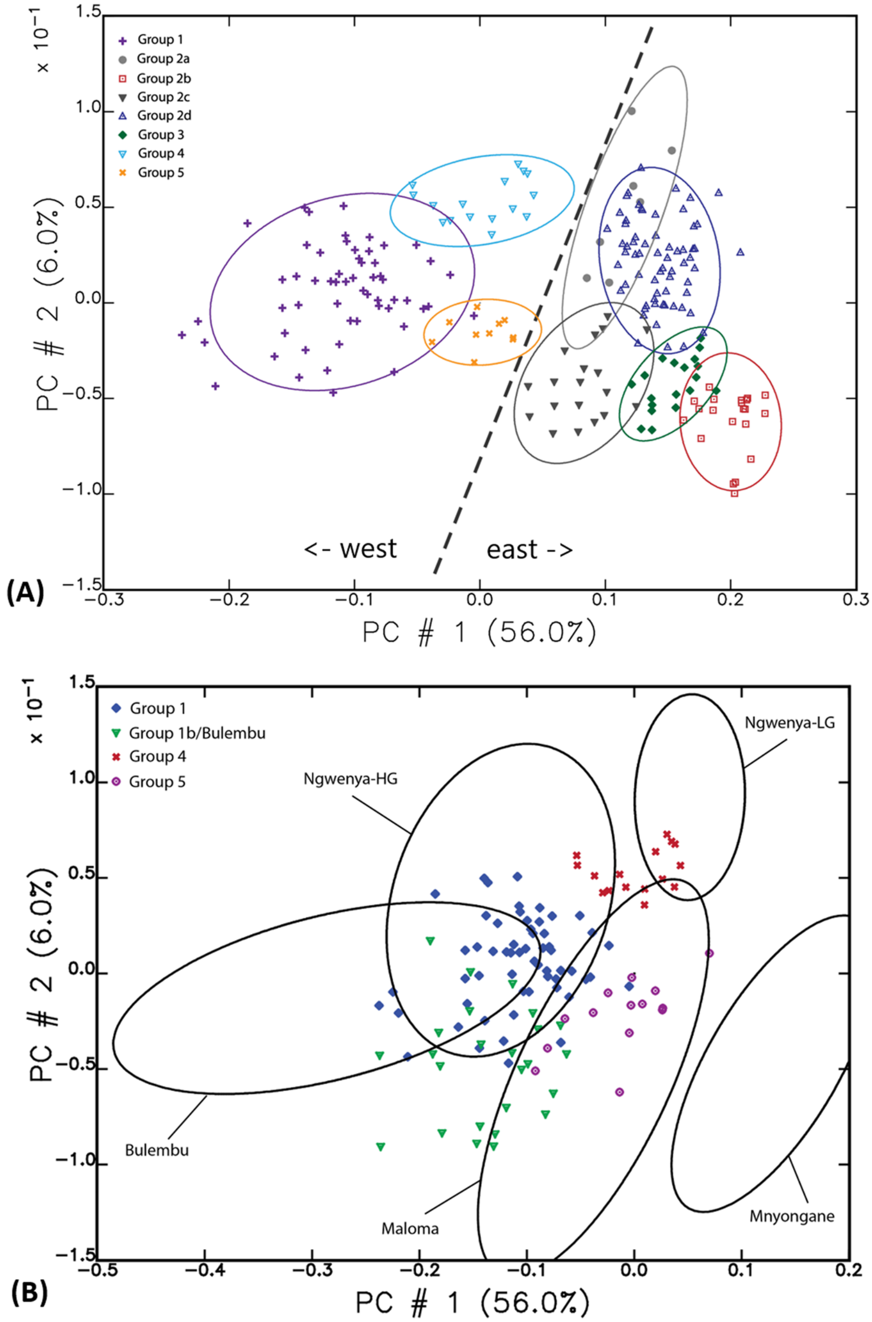

Fig. 5 | Results from the statistical analysis of NAA data on ochre artifacts. **(A)** Scatterplot showing results of principal component 1 (PC1 56.0%) vs principal component 2 (PC2 6.0%) for the NAA, showing all ochre artifact groups. Ellipses are drawn at 90% confidence. Note that there is an artificial divide, indicated by a dashed line, between compositional groups that are dominant at sites located in the western highveld and middleveld (G1, G4, G5) and eastern Lebombo regions (G2a-d, G3) of Eswatini. **(B)** Results from the statistical analysis of NAA data, comparing ochre sources and artifacts. Scatterplot of PC1 (56.0%) vs PC2 (6.0%) for the NAA, showing the distribution of artifact datapoints (icons) projected against known source materials (ellipses). Ellipses are drawn at 90% confidence. Source data are available in Supplementary Data 3.

derived from a discrete ironstone outcrop ~900 ft. long. To date we have not had the opportunity to locate this source. However, given its presence in association with the ore-rich kiln deposits at Mphaphati I, we can suggest Mahalamanti Valley as the likely locality for the source represented in Fe-G5. Interestingly, the ochre artifacts from Fe-G5 have a widespread geographic distribution, occurring in equal quantities at sites in the western highveld (Sibebe, Hlalakahle, Nyonyane), and the eastern Lebombo Mountain (Siphiso, Muti Muti). That its geographic origin occurs in the southern-central lowveld, relatively equidistant to the western and eastern regions, provides a key link for understanding long-distance connections across the geographic divide of Eswatini.

Manganese Oxides – Sources and Artifacts: in this study a subset of 56 manganese oxide source and artifact materials were identified. They were analyzed independently of the FeOx assemblage using LA-ICP-MS. The rationale for and results of this approach are described in Supplementary Notes 5.2. Statistical analysis of the data showed the presence of six MnOx groups, three of which are linked to sources we identified in our field survey (Supplementary Table 4). Figure 7a and b shows a hierarchical cluster analysis and PCA scatterplot of the results.

MnOx Group 1/Bulembu: this group consists of 10 samples, six of which are source materials from Bulembu and four of which are artifacts from Sibebe. Mineralogical analysis showed the presence of one

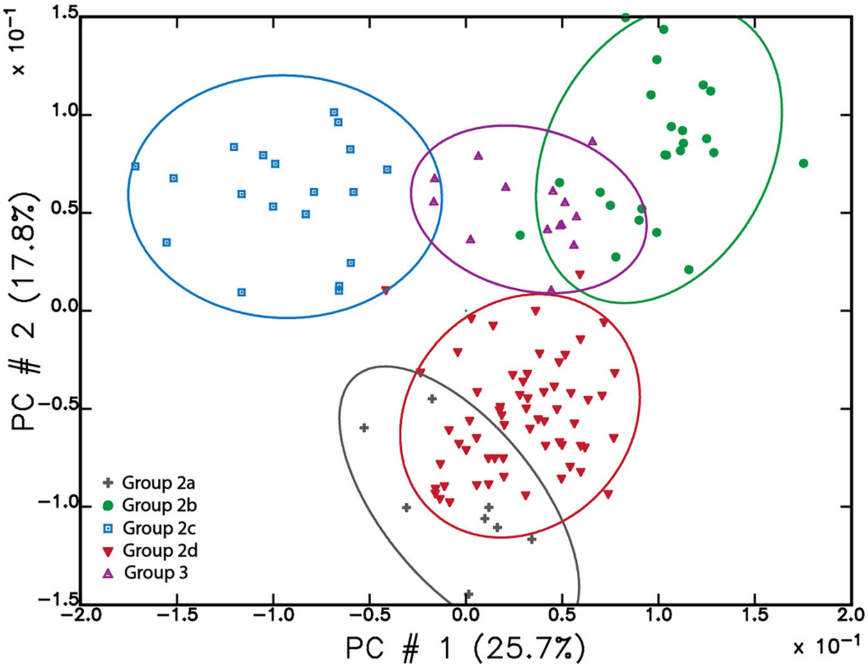

Fig. 6 | Results from NAA data on ochre artifacts from eastern Eswatini. Scatterplot of principal component 1 (PC1 25.7.0%) vs principal component 2 (PC2 17.8%) of the NAA data on ochre artifacts primarily from eastern Eswatini (G2a-G3). The plot shows the distribution of artifact datapoints organized into compositional groups, with ellipses are drawn at 90% confidence. Source data are available in Supplementary Data 3.

or more of pyrolusite, (MnO_2), hematite, alabandite (MnS), and minor quartz and siderite ($FeCO_3$).

MnOx Group 2/Eastern region: these two samples are from Siphiso and consist of high purity pyrolusite (>70%) with 3-4% BaO and 0.8-1.0% K_2O, suggesting a hollandite-type mineralization. Both pyrolusite and K_2O are dominant in XRD results.

MnOx Group 3/Western region: this group of three samples from Sibebe are 50–62% Mn with 10-20% Fe and 0.8-2.1% Ba. XRD results showed peak patterns for barium iron oxide (Ba_8 Fe_8O_{21}), iron-manganese-sulfide (Fe_9 S_{10} and $Fe_{0.95}$ $Mn_{0.05}$), alabandite, and hausmannite ($Fe_{0.297}Mn_{2.703}O_4$).

MnOx Group 4/Banda Cavern: this group has 14 samples, two of which are from the source at Banda Cavern, three from Sibebe, and nine from Siphiso. The samples have Mn content ranging from 10-70%, with Fe content up to 33%. This group has notably elevated trace and rare earth elements: up to 1% BaO, a few hundred ppm each of La and Nd, and Ce values ranging from 0.08 to 2.1%. XRD data indicated the dominant minerals are pyrolusite, braunite, $BaMnO_3$, bixbyite ($FeMn_2O_3$), jacobsite ($Fe_{1.2}Mn_{1.6}O_4$), and magnetite.

MnOx Group 5/Kubuta MnOx Ore: this group contains 14 samples, one of which is from Sibebe and the remaining are source samples from the Kubuta manganese ore field. Mn concentrations are up to 28.8%, with iron ranging from 30.1-61.2%, suggesting a composition similar to umber pigment. Raman and XRD results showed magnetite, hematite, bixbyite, pyrolusite, with minor quartz.

MnOx Group 6/Eastern region: this group has ten samples from Siphiso. The source location remains unknown. Mn concentrations reach as high as 64.8%, with Fe concentrations at trace level (<250 ppm). Potassium (up to 7.8%), quartz (up to 29%), and Al (up to 16.8%) impurities are high. XRD results show patterns for pyrolusite, manganite (MnOOH), and quartz.

MnOx unassigned/outliers: one artifact from Sibebe could not be confidently linked to any source or compositional group. Two source samples from Kubuta were outliers, showing notable variance from the remaining samples due to high Mg, Na, and elevated trace elements.

Diachronic Changes at Sibebe and Siphiso: our sampling strategy seized the opportunity to study ochres from two deeply stratified sites, Sibebe and Siphiso. Figure 8 shows the distribution of the compositional groups across five temporal phases at Sibebe. Strata I and II, representing the final LSA (including pottery), are dominated by ochre from nearby outcrops at Ngwenya (Fe-G1a) and Bulembu (Fe-G1b). The FeOx artifacts that match the dominant group at Siphiso (Fe-G2d) are present, but in small proportion of the total assemblage ($<0.5\%$). All MnOx artifacts from this period are in compositional groups that were either matched to one western highveld source (MnOx-G1, Bulembu), or to a group that is exclusively found at Sibebe (MnOx-G3), yet to be sourced.

The Stratum III deposits, covering the LSA components without pottery, similarly show a dominant proportion of Ngwenya (Fe-G1a) and Bulembu (Fe-G1b) ochres, but also a moderate proportion from Fe-G4, a source type that also appears at Siphiso. This stratum also has three artifacts that are unassigned to a group, but showed a weak to moderate probability match to the Maloma source in south-central lowveld. The manganese oxide artifact assemblage showed matches for MnOx-G1 (Bulembu source) and MnOx-G3 (source unknown) in similar proportions to those observed in Strata I and II. Overall, the key differences between the temporal phases include the abandonment of Fe-G4 in favor of Fe-G2d in the later occupation, and the potential use of Maloma FeOx exclusively in Stratum III.

During the final MSA period at Sibebe, represented by Strata V and VI, the use of a proportionally higher number of different sources of both iron and manganese oxides is evident, although we note that there are limited samples to compare to. While Ngwenya and Bulembu continue to be the primary sources used, other ochre types such as Fe-G2d, Fe-G3 (titaniferous magnetite from the Lebombos), Fe-G4 (source unknown but dominant at Siphiso), and Fe-G5 (Mahalamanti Valley) are all present in minor proportions, indicating a higher frequency of long-distance transport from sources in the east and south to Sibebe in the west. The procurement of manganese oxides also shows distinct differences in raw material use, with the absence of MnOx-G1 (Bulembu) and MnOx-G3, and the only identified presence of MnOx-G5 (Kubuta), further evidence for long-distance transport from the south-central middleveld to the western highveld.

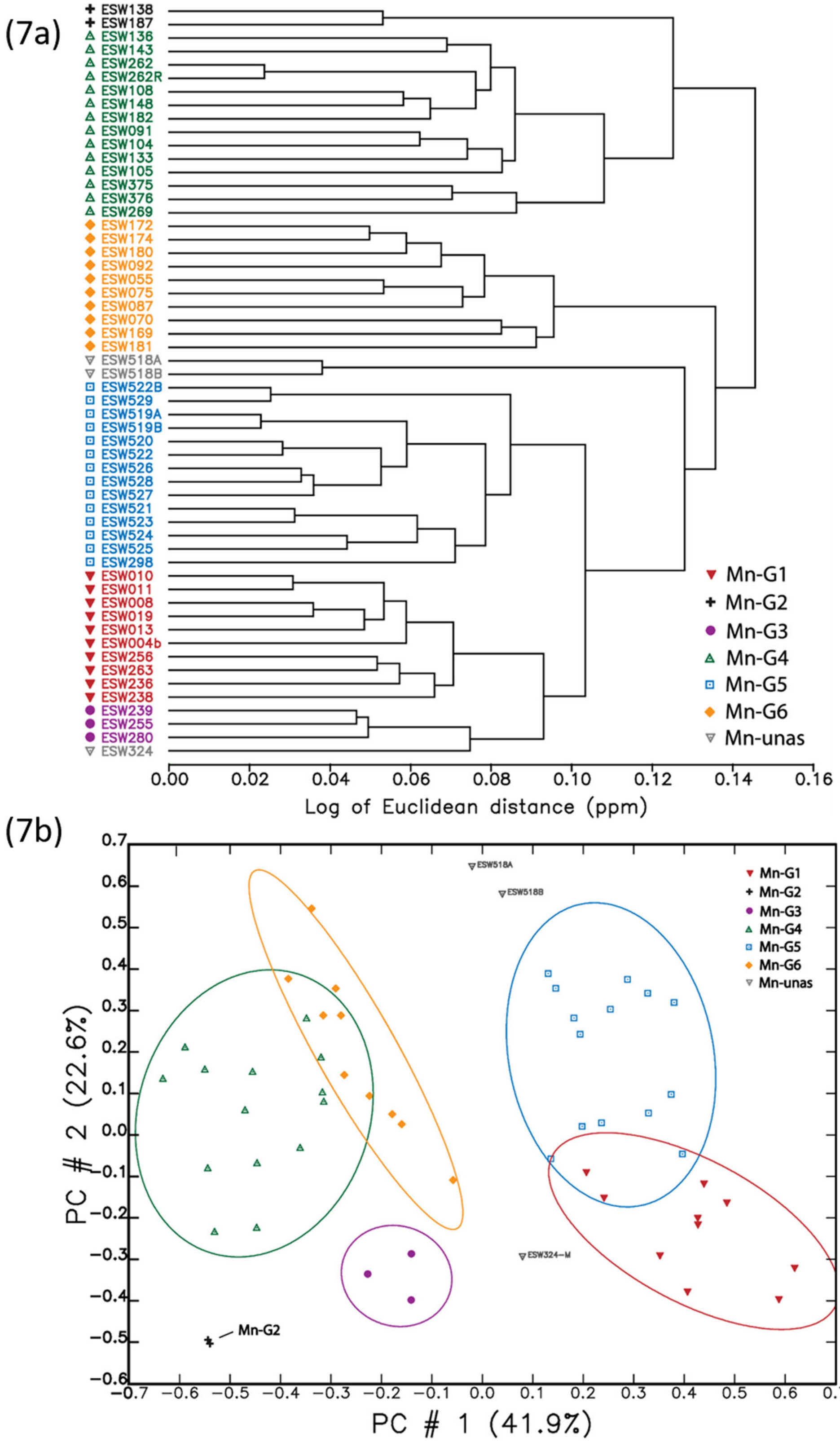

Fig. 7 | Results from the hierarchical cluster analysis of LA-ICP-MS data on manganese oxide artifacts. a (upper): Dendrogram showing the results of hierarchical cluster analysis of LA-ICP-MS data on 56 Mn-Ox artifacts and source samples. **b** (lower): Results from the multivariate statistical analysis of manganese oxide artifacts. Scatterplot of principal component 1 (PC1 41.9%) vs principal component 2 (PC2 22.6%), showing the distribution of Mn-Ox artifacts and source materials. Mn-G1 corresponds to the Ngwenya Bulembu source. Mn-G4 corresponds to the Ngwenya Banda Cavern source. Mn-G5 corresponds to the Kubuta source. Groups Mn-G2, Mn-G3, and Mn-G6 are exclusively artifacts with no known source origins at this time. Unassigned samples are individually labelled. Ellipses are drawn at 90% confidence. Source data are available in Supplementary Data 6.

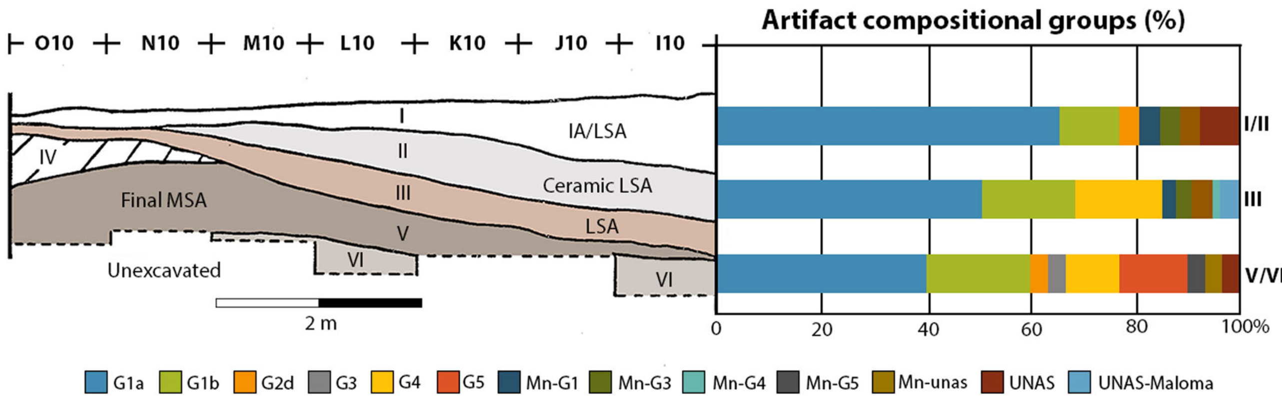

Fig. 8 | Sibebe excavation profile and ochre group distribution over time. Excavation profile and major temporal phases (left) and the relative proportions of iron and manganese artifacts in each phase (right) for Sibebe. Note that all artifacts included in the compositional analysis were selected from excavation units I10, J10, K10, L10, and M10. The reported calendrical dates for each phase are detailed in Table 1. Strata I and II correspond to mixed Iron Age and Ceramic LSA deposits. Stratum III corresponds to pre-ceramic LSA. Stratum V correlates with late final MSA. Stratum VI corresponds to early final MSA.

At Siphiso, a more refined chronological assemblage spanning the late Pleistocene and Holocene LSA has been determined with at least eleven distinct strata, eight of them containing ochres included in this study (Fig. 9, Supplementary Table 2). Throughout the entire sequence, the majority of the iron and manganese oxide artifact assemblages were collected from known or presumed local contexts (all of Fe-G2, Fe-G3, MnOx-G2, MnOx-G6). They show only minor variations in proportion over time, suggesting a longstanding continuity of preferences for multiple local ochre types. Some notable deviations from this pattern include the presence of Ngwenya and Bulembu iron oxides in Strata III, IV, and V (ceramic LSA and LSA), but not predating the Oakhurst litho-technocomplex phase at ~12.2-9.7 cal BP kya. Iron oxides from the unknown source Fe-G4 appear only in Strata VI and VIII, and those from Fe-G5 (Mahalamanti Valley) appear consistently, but in low quantity, in every stratum from IV to VII. The presence of Fe-G5 ochre in those strata are evident of long-distance transport from the Mahalamanti Valley in the central lowveld to Siphiso restricted to the Oakhurst and Wilton phases, with this practice abandoned by the ceramic LSA period.

Table. 1 | Summary of archaeological sites and source areas, including the numbers of artifacts analyzed per site and source area

Archaeological Site	No. artifacts analyzed	Total No. artifacts
Sibebe	129	325
Siphiso	176 (+5 surface)	549
Mlawula I & II	11	11
Nyonyane	14	20
Nsangwini	3	3
Hlalakahle	2 (surface finds)	2 (surface finds)
Muti Muti	13 (surface finds)	13 (surface finds)
Mphaphati I	2	2
Ndhlozane shelter	6 (surface finds)	6 (surface finds)
Total Artifacts	361	931
Source Samples	**No. of sub-samples**	**Notes**
Ngwenya (various locations)	52	Lion Cavern, Banda Cavern, Ngwenya LG outcrop, SW Spur
Bulembu	33	Roadcut outcrop
Ethnographic samples (markets)	6	Manzini and Mbabane market
Mnyogane	35	Roadcut outcrops
Luphohlo Dam	10	Roadcut outcrops
Kubuta	15	Source survey
Maloma	18	Source survey
Mahamba Mtn. Gorge	4	Secondary nodules transported downstream gorge
Gege	0	Source area ID'd but destroyed by modern mining activity
Total Source Samples	173	
Provenance Analysis Grand Total	534	

Ochre Transport to Other Sites: assemblages from the western sites, Nyonyane, Hlalakahle, and Nsangwini, allowed us to further examine networks of ochre transport or the shared used of sources. Nyonyane, situated ~15 km south of Sibebe, has ochre primarily from Ngwenya and Bulembu (~ 50% of the assemblage), but also has one of each from Fe-G2d, the dominant Siphiso source, and Fe-G5. Ochre artifacts from Nsangwini, although few in number ($n = 3$), all matched to Ngwenya or Bulembu. The ochre surface finds from Hlalakahle ($n = 2$) had one each from Ngwenya and Mahalamanti Valley (Fe-G5).

Three sites in the eastern Lebombo region near Siphiso also yielded ochre assemblages: Mlawula 1, Mlawula 2, and Muti Muti. The Mlawula sites are short-term encampments with Holocene LSA diagnostic material culture[51]. The assemblage at Mlawula 1 has Ngwenya ochre ($n = 1$) and one sample from eastern type Fe-G2c. Mlawula 2 exclusively contains ochre from eastern groups Fe-G2d and Fe-G3. Surface finds that were collected during a recent survey at Muti Muti ($n = 13$), directly underneath a large panel of rock art, all showed matches to Fe-G2a, Fe-G2b, Fe-G3, and one to Mahalamanti Valley Fe-G5.

Two sites in the southern middleveld were included in this study: Mphatiphati I, where Mahalamanti Valley ochre nodules were discovered in association with ore in an iron-working kiln, and Ndhlozane rock shelter, where six nodules of ochre were found underneath a wall of rock art motifs. Five of the six nodules from Ndhlozane could not be matched to any sources or compositional groups, but one matched to the Fe-G2a material sourced to the Siphiso rock shelter vicinity. This suggests the potential for another unidentified source in the vicinity of Ndhlozane in the southwest, as well as long-distance transport of Lebombos ochre to that same site.

Ochre Processing and Use: Fig. 10 illustrates a selection of ochre samples exhibiting usewear from the Sibebe assemblage. Remarkably, only nine of the 931 (<1%) artifacts examined this entire study showed clear evidence of usewear, six of which were from Sibebe, and four of

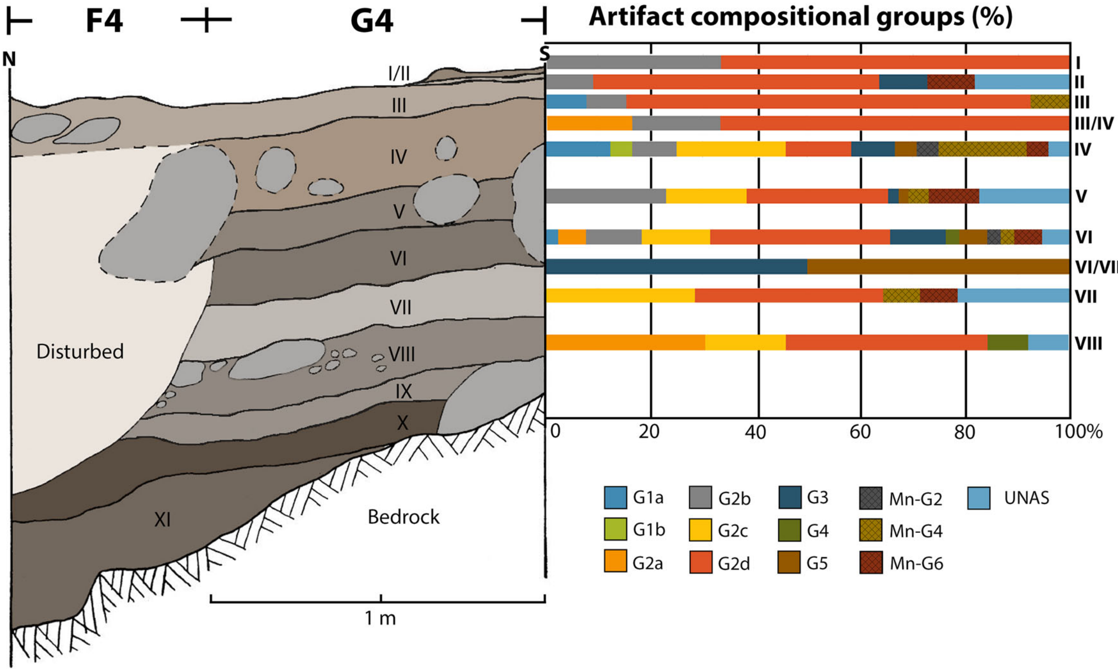

Fig. 9 | Siphiso excavation profile and ochre group distribution over time. Excavation profile and major temporal phases (left), and the relative proportions of iron and manganese artifacts in each phase (right) for Siphiso.

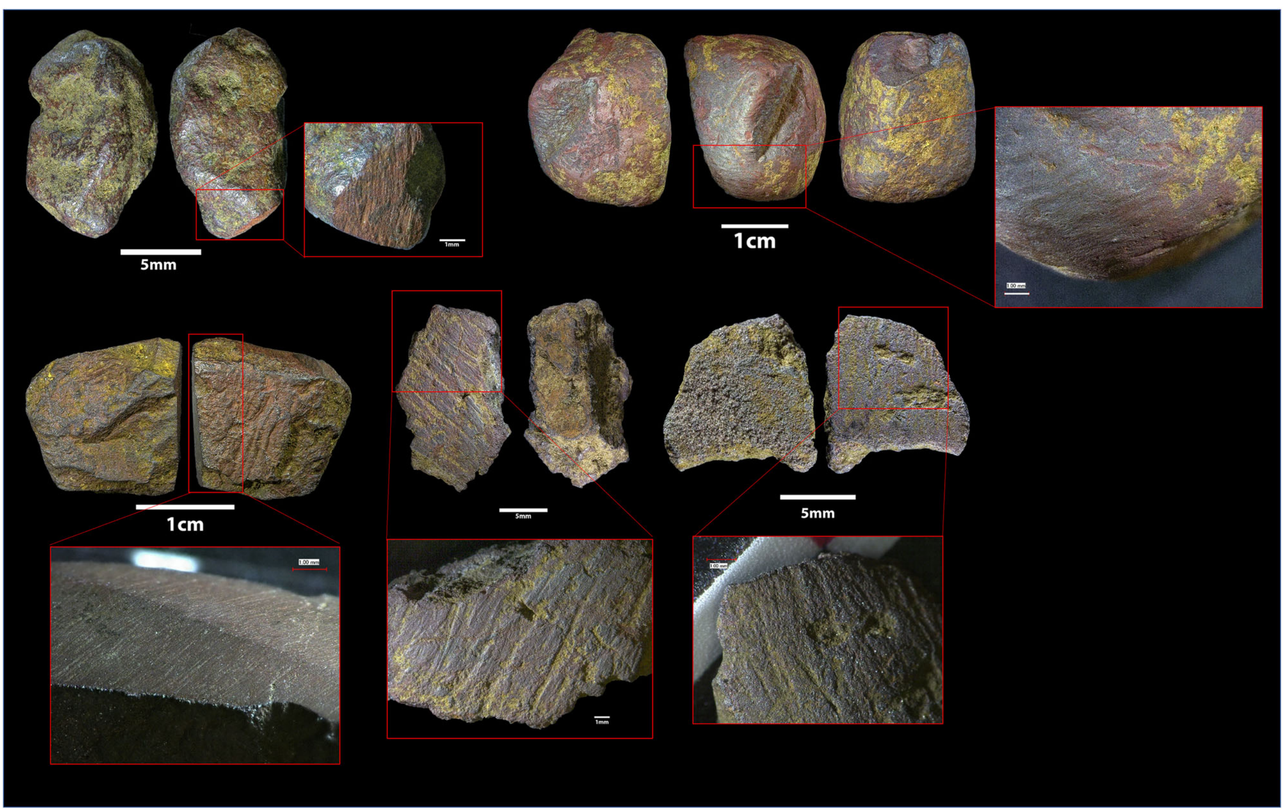

Fig. 10 | Modified ochre artifacts. Examples of modified ochre artifacts from Sibebe, showing usewear, striations, and evidence for grinding or scoring.

those limited to the uppermost stratum (I/II) at that site. Those six samples were either Type 2 (specularite) or Type 3 (fine-grained soft hematite) ochres. The remaining modified ochres were from Siphiso ($n = 2$, Type 2 specularite and Type 1c, weathered consolidated hematite) and Nyonyane ($n = 1$, consolidated hematite, stratum III). This low proportion of modified ochre artifacts in the Eswatini assemblages is in contrast to the few other comparable systematic studies on ochre processing. For instance, Hodgskiss' analysis of MSA ochre at Sibudu[52] identified 7.57% of the total assemblage exhibited evidence for usewear (more commonly during the pre-Still Bay period ~77 kya), and Hodgskiss and Wadley identified usewear (grinding, scoring, rubbing) in 11.2% of the assemblage at Rose Cottage Cave[53].

The results from the Eswatini assemblage suggests that the more common method for preparing ochres for use as paint to for objects, rock walls, or as body paint, may have been through grinding or pulverizing whole pieces using an anvil or mortar and pestle, rather than via repeated abrasion or scoring of the ochre pieces against other textured surfaces to produce powder.

Other indicators for the processing and end-use of ochre are evident in the abundance of rock art. There are currently 52 documented (mostly unpublished) rock art sites across Eswatini, several of them coincident with the assemblages studied here (Nsangwini, Sibebe, Muti Muti, Ndhlozane). The artifact assemblage recovered from Nsangwini also yielded 17 lithics (chert, hornfels) stained with dry-rubbed ochre or spatters of prepared ochre paint. Further study of those stone tools is currently underway.

Discussion

Ochre Communities of Practice – Regional-Scale Patterns versus Localized, Site-Specific Traditions: the emerging picture is one of a unifying culture of significance around ochre and its longstanding importance within social activities across Stone Age Eswatini, nuanced with unique and contextually-contingent variations in the ochre communities of practice. We confirmed that ochre was mined in large quantities from Lion Cavern, as early as at least 48 kya. At Sibebe, Ngwenya ochre was the predominant and continuously used type from the earliest known final MSA sequence through to the Iron Age. This preference was established from the outset of Sibebe's occupation, indicating that the community of practice surrounding Ngwenya ochre likely existed prior to the sites' use. This pattern is evident of the selective preference of Ngwenya ochre over other nearby sources (Mnyogane, Lupholo Dam), and would have necessitated the social contexts in which collecting ochre was part of regular group traversals between the middleveld and highveld of western Eswatini, and the passing down of this traditional knowledge over tens of thousands of years. From as early as our records show, this activity would have required the landscape knowledge on the extent, stratigraphy, and characteristics of the hydrothermal geologic formation where the Ngwenya iron ore was accessible at the earth's surface, whether it was accessed at the outcrops at Banda Cavern, Lion Cavern, Castle Quarry, or Bulembu. It required knowledge on the use of adequate tools and group cooperation for mining large volumes of highly consolidated iron ores. The people seeking it would likely have experimented with the mined ochres to test their properties for their intended use, among which would have included colorfastness, impurities, hue, dispersion in a binding medium (such as blood, fat, water, plant exudates), resistance to weathering, and adhesion to a surface such as skin or stone. Those characteristics would have been among the many material and symbolic motivations that made Ngwenya ochre desirable to the hunter-gatherers in western Eswatini who continuously chose it, and passed down that knowledge to their social networks, over thousands of years. Those preferences did not develop in isolation, and represent the evolution of a community of practice that connected different generations over time and space. Moreover, these preferences were not only restricted to peoples' occupation at or near Sibebe. Ngwenya ochre was transported to other sites in the western middleveld, including Nyonyane, Hlalakahle, and Nsangwini, the latter of which has an assemblage of ochre stained lithics and a rock art panel of over two dozen polychrome motifs[57,58]. Evidently, Ngwenya ochre was also transported over 100 km across different territories, from the western highveld through the central lowveld, and to the Lebombo region to Siphiso and Mlawula 1 during the LSA. Although this was done infrequently and sporadically, it reinforces a shared culture around the desirability and use of Ngwenya ochre.

In contrast to the patterns observed at sites in the western middleveld, the eastern sites showed a comparatively higher diversity of ochre types used, where as many as five unique signatures were either sourced to a geologic formation (Fe-G2a, at Siphiso), or at least local to the area around Siphiso, Mlawula 1 & 2, and Muti Muti. The higher diversity of likely local ochre sources used consistently over the LSA occupation at Siphiso is indicative of more varied procurement strategies and preferences, and perhaps different social contexts for its use. It suggests that ochre procurement may have been aligned with more frequent logistical mobility (sensu Binford[59]), and may have been embedded with other provisioning activities where the teaching and learning about networks of resources, such as lithic raw materials or seasonal food procurement, had more varied nodes of experience to share. In contrast to Ngwenya ochre, most of the Lebombos are source materials tend to be more weathered, friable, lighter in hue, and have a higher proportion of impurities, such as mica, quartz, feldspars, and clay minerals. Those ochres are much softer than the consolidated ores at Ngwenya, and consequently, their collection required different strategies and tools than the western and southern ore sources (e.g. digging sticks vs. hammerstones and lithic tools). The ochre collectors would have done some testing (grinding, scoring, mixing, applying to various surfaces, see Supplementary Notes 1.0) to understand their qualities for rock art or body paint, adhesives, and other potential uses. Based on our observations while processing the same materials in the lab, the impurities present in most of the eastern ochre sources would have reduced the tinting strength of the material, resulting in weaker color saturation. The main way to improve the opacity and adhesion properties would be to remove those impurities through dry sifting or levigation, a practice that continues today by vendors who sell prepared ochre balls at local markets (Supplementary Notes 1.5). The entire *chaîne opératoire* associated with ochre sources from the Lebombos would have involved a different body of knowledge needed to locate, extract, transform, and use those materials.

Long-Distance Transport versus Exchange of Ochre: at a regional scale it is clear that there is a shared, overarching culture of value ascribed to ochre procurement and use. But, how were these values expressed between communities and how did that change over time? Our data has shown long-term consistency in the use of locally-procured earth mineral pigments, practices of which were punctuated by sporadic events of long-distance transport of ochre across pathways that linked the western highveld, central lowveld, and eastern Lebombos of Eswatini. Patterns of primarily localized procurement are to be expected in the context of mobility strategies amongst hunter gatherers[60–62], and our results are consistent with observations by Barham that it was entirely possible for occupants in the eastern and western regions of Eswatini to thrive within their own biomes in terms of seasonal resource availability[51]. But the question remains: were the instances of long-distance transport of ochre evidence for cultural exchange or direct procurement, or both? Do the different modes of ochre provisioning and processing signal social boundaries revolving around the material, where the sources, settings, practical knowledge, and technical vocabularies are restricted? Research on other forms of material culture has proposed that long-distance reciprocal gift exchange, such as *hxaro*, a system of delayed reciprocity, was the main exchange system thought to underpin many social interactions in neighboring seasonally-mobile communities across southern Africa[1,51,60]. However, our data do not suggest that the exchange of ochre was an intensive and frequent part of *hxaro* cultural practices. The most frequent occurrence of eastern Lebombo ochre at Sibebe was in the late final MSA during a brief hiatus when Siphiso was not occupied (Supplementary Notes 2.2), therefore, exchange between groups was not evident. In contrast, the final 2000 years of occupation was the only period when both Siphiso and Sibebe were occupied simultaneously, with limited evidence for the shared use of sources during that time. Consequently, this points toward more direct, localized modes of ochre procurement.

The rarer instances of long-distance transport are more aligned with evidence that shows hunter-gatherer populations in Africa were structured into groups that were never fully isolated, but rather showed sustained partial connectivity, facilitated by the fluid structure of hunter-gatherer bands[60]. While ethnographic analogy cannot and does not explain the past, it is a useful tool for broadening our understanding of possible drivers of human behaviours and decision-making. For instance, the infrequent long-distance transport of ochre could be indicative of the itinerant movement of painters or *tangoma*, who were known ethnographically to collect and curate earth mineral pigments for painting and healing ceremonies, and for the *siyendle* hair style, where the head and hair was, and still occasionally is, smeared with ochre as a way to denote shamanic power[35]. It also may signify the relocation of women through intermarriage. In Nguni cultural practices extending to the present day, ochre is a significant component of the marriage ceremony *kuteka*, where the bride is covered with red ochre and animal fat on the morning of the wedding, typically by a female elder in the groom's family, to signify her new status in the community and to allow the husband's ancestors to locate her via the ochre. If the groom's family did not have good ochre in their territory, it would be brought in from afar as a symbolic exchange between families[34–38]. These examples further reinforce the notion that, although there was a regional-scale, unified understanding of ochre and its cultural significance across Eswatini, the localized activities associated with its *chaîne opératoire* were varied and contextually-contingent communities of practice.

Significance for the MSA/LSA Transition: the MSA/LSA transition and the origins and spread of the LSA remains a longstanding debate[63–66]. In southern Africa, the prevailing consensus for many years was that ~30 kya BP represented the onset of the LSA, based mainly on lithic technocomplexes, but also an increase in the diversity of raw material use, personal ornamentation, mortuary traditions, and artistic expression[67]. One long-considered exception to this is Border Cave, in the Lebombo mountains overlooking Eswatini, with much older dates associated with a characteristic LSA lithic assemblage [~43 kya BP], prompting debate on whether it represents a singular origin of the LSA, or if multi-regional or multi-temporal hypotheses can be considered[68,69]. More recently, cultural and technological innovations typical of the LSA have been found in clear association with MSA-aged deposits at other sites including, but not limited to, Blombos Cave, Diepkloof, and Sibhudu[68,70–73]. Similarly, our research on lithics and ochre has shown evidence consistent with a more complex view on the timing and geographic distribution of this transition. For instance, the lithic technocomplex at the MSA/LSA site Sibebe, a mere ~100 km from Border Cave, has two distinct groups of MSA lithic assemblages dating between 43-27 kya BP[74], which are similar in their technological expressions to other sites with final MSA assemblages at Sibhudu or Umbeli Belli in KwaZulu-Natal[75]. The results from our study have shown that the selection and transport of ochre was subject to more subtle, regional-scale variations than the wide-spread temporal transitions implied by the term MSA/LSA transition. Recent studies have shown that LSA technologies may have gradually evolved out of those commonly called the MSA[76,77]. The emerging consensus is one where this cultural transition is neither cumulative nor persistent in any region, which has compelled researchers to reevaluate our collective understanding on what the MSA/LSA transition truly means, whether or not it requires redefinition, or if it existed at all[69,72,74,78–84].

Methods

Ochre characterization

The artifact assemblages were typologically examined at Eswatini National Museum and exported to the MURR Archaeometry Laboratory under permits granted by the Eswatini National Trust Commission (2019–2022). The sample selection, methodology, and experimental conditions are fully detailed in Supplementary Notes 3.0. The analytical methods included pXRF, NAA, LA-ICP-MS, Raman spectroscopy and XRD.

All samples were qualitatively screened using pXRF. The measurements were taken using a Bruker Tracer 5i energy-dispersive XRF spectrometer equipped with a Rh-based X-ray tube and thermoelectrically cooled silicon-drift detector (SDD). The instrument was operated at 50 kV and 35 μA (no filter) for 60 second assays on each sample.

Neutron Activation Analysis (NAA) was used for elemental characterization on 388 artifacts and sources. All specimens were washed in deionized water, dried, ground to a fine powder in an agate mortar, and pestle, and dried again for a minimum of 48 h in a low-temperature oven (<100 °C). Portions of each powder were weighed into polyethylene vials for short (25-75 mg) irradiation, and into high-purity quartz vials (100 mg) for long irradiation procedures. The short duration samples were irradiated alongside standard reference materials using a pneumatic tube system for 10 seconds at a flux of 8×10^{13} n cm^{-2} s^{-1}. The samples were allowed to decay for 25 min, at which time the gamma ray emissions were measured for 12 min using a hyper-pure germanium detector to detect elements that produce short-lived radioisotopes for aluminum (Al), barium (Ba), calcium (Ca), dysprosium (Dy), potassium (K), manganese (Mn), magnesium (Mg), sodium (Na), titanium (Ti), and vanadium (V). The quartz encapsulated samples were given a 24-hour irradiation at a neutron flux of 6×10^{13} n cm^{-2} s^{-1}. These samples were measured for 2,000 seconds after seven to ten days to measure medium-lived radioisotopes arsenic (As), lanthanum (La), lutetium (Lu), neodymium (Nd), samarium (Sm), uranium (U), and ytterbium (Yb), and then again after two to three weeks for 8,200 seconds to measure the long-lived radioisotopes cerium (Ce), cobalt (Co), chromium (Cr), cesium (Cs), europium (Eu), iron (Fe), hafnium (Hf), nickel (Ni), rubidium (Rb), antimony (Sb), scandium (Sc), strontium (Sr), tantalum (Ta), terbium (Tb), thorium (Th), zinc (Zn), and zirconium (Zr). The spectral data were calculated to elemental concentrations by comparator method using NIST standard reference materials and quality controls (NIST SRM-1633b, SRM-688, SRM-278, SRM-690).

LA-ICP-MS analysis was applied to 378 samples. Data were collected using a PerkinElmer SCIEX NexION 300 Quadrupole ICP-MS coupled with a Teledyne Instruments Inc. Analyte Excite HelEx 193 nm excimer laser ablation system. We selected between five and ten 40 μm x 80 μm ablation pass lines on each standard reference material, quality control, and unknown specimen. Unknowns specimens underwent a pre-ablation pass at 110 μm x 80 μm before data were collected. After each ablation the laser was paused for 25 seconds while the ICP-MS continued to collect signal intensity data. The laser moved at a rate of 5 μm/s firing laser bursts at a rate of 10/s. Laser power was set to 40.0% of the maximum output. The procedure bracketed ten unknown samples with a set of standards and quality control samples at the beginning and end of each to monitor instrument stability and drift throughout analytical runs (approximately every 30 mins). The ablated sample vapor was transported to the ICP-MS using He carrier gas and mixed with argon gas at the plasma torch, where the sample was ionized and passed through two detectors that measured the signal intensity in counts per second for 60 isotopes: ^{7}Li, ^{9}Be, ^{11}B, ^{23}Na, ^{24}Mg, ^{27}Al, ^{29}Si, ^{31}P, ^{34}S, ^{35}Cl, ^{39}K, ^{44}Ca, ^{45}Sc, ^{47}Ti, ^{51}V, ^{52}Cr, ^{55}Mn, ^{57}Fe, ^{59}Co, ^{60}N, ^{63}Cu, ^{66}Zn, ^{71}Ga, ^{75}As, ^{77}Se, ^{85}Rb, ^{88}Sr, ^{89}Y, ^{90}Zr, ^{93}Nb, ^{98}Mo, ^{107}Ag, ^{115}In, ^{118}Sn, ^{121}Sb, ^{133}Cs, ^{138}Ba, ^{139}La, ^{140}Ce, ^{141}Pr, ^{146}Nd, ^{147}Sm, ^{153}Eu, ^{157}Gd, ^{159}Tb, ^{163}Dy, ^{165}Ho, ^{166}Er, ^{169}Tm, ^{172}Yb, ^{175}Lu, ^{178}Hf, ^{181}Ta, ^{182}W, ^{197}Au, ^{205}Tl, ^{208}Pb, ^{209}Bi, ^{232}Th, and ^{238}U.

The results of trace element analyses were analyzed using multivariate statistics, detailed in Supplementary Notes 4.0 and 5.0.

Selected samples were analyzed using powder XRD to identify major mineral components. Small portions of prepared powders were analyzed using a Rigaku Ultima IV equipped with a Cu-Ka[1] (1.540562 Å) X-ray tube. The experimental conditions for each analysis involved scanning a 20° to 80° 2Θ spectral range at a step size of 0.06° at a rate of 4° per minute.

Raman Spectroscopy: selected samples were analyzed using a Bruker Bravo™ handheld Raman spectrometer. The instrument is equipped with two laser diodes (785 nm and 852 nm), operates at a maximum (combined) laser output of 100 mW and a spot size of 0.7 mm, and records signal intensity data at a spectral range of 300 – 3200 cm^{-1} at a resolution of 10-12 cm^{-1}.

Optically Stimulated Luminescence

OSL dating of Lion Cavern deposits was conducted by S.R. at the University of Cologne Luminescence Laboratory. Six samples were collected from outcrops of Lion Cavern 3 (NLC-1, NLC-2) and Lion Cavern 2 (NLC-3 to NLC-6). The samples were taken in opaque metal tubes to avoid the exposure of the sediment to sunlight. The samples were prepared under subdued red-light conditions in the Cologne Luminescence Laboratory (CLL). Hydrochloric acid (HCl, 10 %) and hydrogen peroxide (H_2O_2, 10 %) was used to remove carbonates and organic material. Sodium oxalate ($Na_2C_2O_4$; 0.01 N) was used to disperse the sediment particles. After chemical treatment the 4-11 μm fraction was isolated using Stokes law and subsequently etched in hexafluoridosilicic acid ($H_2(SiF_6)$, 34 %) for one week to extract fine grain quartz minerals. The extracted quartz was mounted on stainless steel cups by settling of 2 mg quartz in 200 μl distilled water. Luminescence measurements were performed on Risø TL/OSL DA20 readers equipped with a $^{90}Sr/^{90}Y$ beta source and blue LEDs operating at 90 % power. A single-aliquot regenerative dose (SAR) protocol was used for all luminescence measurements. All results are presented in Supplementary Table 6. The analytical procedure is provided in Supplementary Text 6.0.

The authors affirm that human research participants [or their parents/guardians] provided informed consent for publication of the images in Supplementary Figs. 8 and 9.

Reporting summary

Further information on research design is available in the Nature Portfolio Reporting Summary linked to this article.

Data availability

All data needed to evaluate the conclusions in the paper are present in the paper and/or the Supplementary materials. All source data used in figures can be found in Supplementary Data 2–12. All unconsumed archaeological specimens are in process of repatriation to the Eswatini National Museum (Ezulwini, Eswatini). Archived samples of all geological materials collected in the course of this project are located at the Eswatini National Museum. Inquiries regarding access to artifact or geological samples should be directed to co-author Bob Forrester (Eswatini National Trust Commission).

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Competing interests

The authors declare no competing interests.

Additional information

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