Supplementary Materials for

Paleoindian ochre mines in the submerged caves of the Yucatán Peninsula, Quintana Roo, Mexico

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SI-Text 1: Reported Contexts of Paleoindian Ochre Use in North America

This brief review of Paleoindian and Early Archaic ochre finds is not exhaustive, but attests to the ubiquity and contexts of ochre use throughout the early human occupation of North America. Whether ochre use constitutes evidence for ritual or symbolic behavior remains an ongoing anthropological debate (18), however current consensus suggests the two are not mutually exclusive (19). Ritual behavior is typically characterized by the presence of material objects used in connection with other realms of existence or afterlife, and includes symbolic repetition, showing great investment in wealth, the presence of exotic materials, ritual breakage (42), or pristine objects. These instances are commonly identified in burials and “ritual” caches, where ochre is frequently used to cover all or part of human remains, or objects such as lithic bifaces, tools, or beads made of bone or shell (43). For instance, at Anzick (Montana), an infant burial dated to ~12,600 BP included a collection of artifacts, some of pristine quality and all caked in red ochre (10, 44), which was interpreted as an assemblage “meant to provision the child in the afterlife” (45). At Gordon Creek, ochre was used to cover human remains (46), and animal bones (47). At Horn Shelter (Texas), a burial dated to 11,100 BP of a juvenile female and an adult male contained an elaborate assortment of ritual objects interpreted as the toolkit of a shaman or healer, which included an ochre slab, shell beads, hawk talons, and badger claws (48, 49). The Browns Valley site in western Minnesota had ochre deposited on human remains and grave goods (50). The Sheaman site (Wyoming) had two bison bones thickly coated in ochre and scattered on the living floors (51). The Cooper Site had an ochre-painted bison skull, interpreted as evidence for a ritual act (52). At Upward Sun River Site (Alaska), a juvenile burial was recovered with ochre fragments being the only identified associated artifacts (11). The Powars II
site (Wyoming) is situated on an ochre deposit that was recently reinterpreted as a ritual hunting
site (15, 16).

The predominance of ochre in Paleoindian caches also highlights its significance. Caches
most often contain stone tools with high artifact type diversity, high toolstone raw material
diversity, exotic materials, and in most cases, red ochre and human remains (45). The Fenn
(Utah/Wyoming/Idaho), Simon (Idaho) and East Wenatchee (Washington) caches each contained
assemblages of large ovoid bifaces, point preforms, and finished points covered in red ochre (12,
14, 53). The Sloan site (Arkansas) contained red and yellow ochre in association with a stone
tool assemblage (54-56). The Thurman Station site (New York), a late Paleoindian cache dated
to 10,800 – 10,000 cal BP, contained multiple ochre stained artifacts and bifaces (57). On the
Channel Islands (California), Erlandson et al. (58) identified one deeply-striated hematite nodule
bearing usewear consistent with pigment processing.

Other instances suggest utilitarian use of ochre, for instance on diagnostic tools as part of
grease or mastic, making hunting weapons more durable and effective (59); or simply cited as
“domestic” contexts. Sites including Hanson (60), Agate Basin (61), and Cattle Guard (62) had
ochre stained habitation floors or ochre grinding stones indicative of pigment processing (43).
The Lindenmeier site contained an assemblage of five faceted hematite abraders, ochre stained
chipped and groundstone tools, a drilled hematite bead, and red staining indicative of a pigment
processing or hide tanning area (13, 29). Ochre paintstones were found near the spring at
Blackwater Draw site (Texas) (63). Yerkes and Koldehoff (64) report an ochre stained pit floor
at the Jens site that represents a hide tanning and pigment processing area. Mayer et al. (65)
report an assemblage of sandstone abraders covered in ochre alongside ochre nodules and floor
staining at the Barger Gulch site (Colorado). The Clary Ranch site (Nebraska) yielded over
10,000 ochre nodules and ochre stained scrapers (66), making it one of the largest deposits of ochre reported from a site of this time period.

**SI-Text 2: Qualitative and Geochemical Characteristics of Ochres**

Ochre collection and preparation practices are studied widely, especially where ochre finds are associated with human evolutionary development (9, 30, 31, 67-70). Provenance studies have determined evidence for selective and long-term acquisition of specific ochre resources, in some cases despite the availability of multiple viable sources within a localized landscape (8, 71-73), and in others, where annual long-distance pilgrimages to socially-significant ochre sources are centuries-old community practices (74). These studies show that ochre sources were targeted for a myriad of reasons, including pigment quality, territorial boundaries, or as embedded within other subsistence-related activities of mobile hunter-gatherers. Physicochemical studies have demonstrated further evidence for ochre treatment behaviors, including grinding, levigation, thermally-induced hue enhancement, and preparation with organic binding agents (68, 75-77). Understanding each step in the process of ochre source collection and treatment is a key piece of evidence for reconstructing decision-making in pigment selection and preparation in the past.

One aim of this study was to determine the qualitative and geochemical characteristics of ochre from Sagitario (La Mina), Camilo Mina, and Monkey Dust. Those characteristics can provide insight into whether the ochre was conducive to the production of high-quality pigment, and potentially why individuals valued these ochres so highly. Considering the difficulty and risk involved with collecting ochre from the cave systems, we ask: was the ochre extracted from Sagitario (La Mina), Camilo Mina, and Monkey Dust conducive to the production of high-
quality red pigment? Are there chemical and mineralogical differences between the ochre from each of the three sites, and what can this inform about source selection? These characteristics are determined empirically, using a combination of morphological, molecular, and geochemical analyses. We used optical microscopy and scanning electron microscopy (coupled with an energy dispersive spectrometer, SEM-EDS) to determine pigment morphology, including grain size, texture, and mineral inclusions. X-ray Diffraction (XRD) was used to identify major mineral phases in the ochre pigment matrix. Trace element geochemistry was determined by neutron activation analysis (NAA). Selected samples were analyzed using a Cox ITRAX Core Scanner, which simultaneously obtained data on radiography, μ-XRF, and optical imaging.

**Ochre Samples**

Ochre samples were collected from six stations in Sagitario (La Mina) (shown in Fig. 1c), plus two samples from the above ground surface (LMR-S1, LMR-S2). One sample collected from an ochre pile in Monkey Dust (Fig. 5a) and two samples from ochre pits in Camilo Mina (Fig. 5d, 5e) were also examined (SI-Data 1). Samples identified here as LMR are from La Mina, MD are from Monkey Dust, and CM is from Camilo Mina. For the Sagitario (La Mina) samples, a sub-set each of the levigated and non-levigated fractions was examined separately. The levigated samples were heated to 450°C in a muffle furnace (held for 5 h). The purpose of sub-sampling was to determine the extent to which levigation and thermal exposure could alter the physical characteristics of the ochre, an important feature in our evaluation of its use as a red pigment.

*La Mina, Camilo Mina, and Monkey Dust Ochre Characteristics*
The color and texture of ochre pigments are impacted by factors including grain size (78, 79), mineral adjuncts and impurities, and heat treatment (80-82). Grinding Fe-oxides to smaller particle sizes can intensify hue to deeper and richer red and violet tones. Mixtures of mineral adjuncts and impurities can contribute to color composition and texture, including white, matte calcite or chalk (CaCO₃), white kaolinite (Al₂Si₂O₅[OH]₄) or dark manganese oxides (pyrolusite, ‘umber’, black or deep purple-red). Heat-treatment also is an effective means to enhance color, and studies show that thermal exposure was often performed to accelerate oxidation and intensify hue in ochres (82, 83).

Most Sagitario (La Mina), Monkey Dust, and Camilo Mina ochre specimens are brown to red-brown or yellow-red, and muted or matte in luster. Samples from Sagitario (La Mina) contain small white calcite particles visible to the naked eye. Sample LMR-P20, had an earthy, soil-like texture and luster, its matrix interspersed with deep red, rust-like particles. Levigated samples were notably finer-grained and typically more intense in hue after thermal treatment. For most samples, the low temperature heat treatment altered the color properties, intensifying the brown and yellowish-brown untreated samples to more vivid red or deeper red hues. Samples LMR-P05 and LMR-P20 showed least hue intensification, and instead transformed to orange-brown. SI-Fig. 1a illustrates examples of heat-induced color transformation for samples LMR-P01A and LMR-P19A. Ochre samples collected from Camilo Mina and Monkey Dust are notably different in color and texture than that of Sagitario (La Mina). Camilo Mina ochre is fine-grained, has an earthy, matte texture and luster, with a light yellow-red hue. Monkey Dust ochre is comparatively coarser grained with a brown to brown-red hue.

Mineralogical Characterization by XRD
Levigated samples from Sagitario (La Mina) were analyzed by powder XRD at McMaster University X-Ray Diffraction facility in Summer 2018. Samples from Camilo Mina and Monkey Dust, which were collected in Spring 2019, were analyzed using by powder XRD at University of Missouri (Department of Chemistry). SI-Data 1 provides a description of experimental conditions and a spreadsheet of all XRD data, organized by 2θ and peak intensity values and summarizes the major mineral phases.

All samples from Sagitario (La Mina) have similar mineralogical profiles with the exception of LMR-P20. Samples LMR-P01 through LMR-P19 are dominated by one or both of Fe-oxide phases goethite (hydrated iron oxide), or intermediate phase iron oxide-hydroxide lepidocrocite. Most samples contain quartz (SiO$_2$), and one or more of calcite (CaCO$_3$), lime (CaO), or periclase (MgO). Sample LMR-P09 contains alabandite, a manganese sulfide mineral often associated with calcite, suggesting a localized Mn-enriched area. Other samples also enriched in Mn include LMR-P03, LMR-P04, LMR-P10, and LMR-P20, (although less than 1.0%). Sample LMR-P20, which was recovered from a dry airdome location, contains a combination of bohemite and kaolinite, both rock-forming minerals found in bauxite, consistent with a karstic carbonate bauxite-rock origin. Bauxite is formed by lateritic weathering of limestone in humic tropical soils. Iron phosphate (FePO$_4$) is found in sample LMR-P20 and LMR-S, in contrast to hydrated forms of Fe-oxide found in other inundated samples. The Sagitario (La Mina) surface sample (LM-S) is lacking in hematite or goethite, but is dominated by quartz, iron phosphate (FePO$_4$), bohemite, and almandine (Al$_2$Fe$_3$O$_{12}$Si$_3$), a common rock-forming silicate. The ochre sample from Monkey Dust lacks any stochiometric Fe-oxide phases, but is primarily composed of almandine, quartz, and lime (CaO). The sample from Camilo Mina is dominated by goethite and lime.
**Scanning Electron Microscopy**

Scanning electron microscopy was used to examine selected samples (SAG LMR-P04, LMR-P09, LMR-P20, and MD and CM) to capture electron micrographs of mineral grains. All specimens from Sagitario (La Mina) showed similar characteristics of a heterogeneous matrix composed of larger, coarser particles of calcite, periclase, or Al-oxides, interspersed in a finer-grained matrix of smaller goethite and/or lepidocrocite particles. SI-Fig. 1b shows sample LMR-P04. The ochre matrix consists of large (~10-20 µm), angular particles of calcite and periclase, interspersed in a dominant matrix of clumps of fine-grained (< 1 µm), flakey goethite and lepidocrocite particles. Sample LMR-P09 (SI-Fig. 1b) was the only specimen in which alabandite was identified. SI-Fig. 1b shows remnants of the octahedral crystal form covered in small particles of Fe-oxide. SI-Fig. 1c shows sample LMR-P20 as a heterogeneous mixture of Fe-oxides, (iron phosphate), interspersed with Al-enriched particles attributed to either bohemite or kaolinite. Note the stacked kaolinite platelets in (A, B) and, blocky bohemite crystals in (C, D). SI-Fig. 1c shows ochre collected from the surface at Sagitario (LMR-S), which is relatively depleted in iron oxide and enriched in large, angular crystals of quartz, bohemite, and almandine. SI-Fig.1d shows pit ochre collected from Camilo Mina. The matrix is fine grained and dominated by flaky goethite particles. SI-Fig. 1d shows the ochre morphology from Monkey Dust, lacking in fine-grained iron oxides and dominated by large, coarse-grained silicates (quartz, almandine) and periclase.

**Core Scanning - µ-XRF, Radiography, and Optical Microscopy**

Thirteen samples were selected for characterization using a Cox ITRAX core scanner at McMaster University. Nine of the samples were from cave contexts in Sagitario (La Mina) and
Monkey Dust, and four samples were surficial/detrital ochre (i.e. terra rossa soils) from the area around La Mina. The samples were loaded into a sequential sample reservoir (SSR) containing cuvettes of ~ 1cm (84). The analyses included optical imaging (RBG), radiography (Mo-radiation, 30 kV and 25μA), and μ-XRF (Mo-radiation, 30 kV, 18 μA for 10 seconds per scan step) at 1 mm resolution, with results averaged over the length of the individual cuvettes (n=12 measurements). SI-Fig. 1d shows the results of radiographic images of the samples (RAD), optical imaging (RBG), XRF signal intensity (counts) for each sample for elements Fe and As, and for ratios of coherent and incoherent scattering (INC/COH). The latter of these is indicative of the relative organic content and porosity of the samples.

The radiographic (RAD) images illustrate the contrast between ochre found in the mine pits at the bottom of the cave with surficial terra rossa or detrital ochre sourced directly from the surface (i.e. LMR-P20, MD). The cave ochres are relatively higher in density of higher atomic mass elements (i.e. iron), hence the darkened radiographs. This is also supported by the μ-XRF results showing high counts for Fe and As, and a coherent/incoherent ratio indicative of low organic content and sediment porosity. The RBG micrographs show an overall brighter color intensity of red, orange-red, and yellow-orange ochre. In contrast, the surficial terra rossa ochres are lower in density and porosity (indicated both by radiography and INC/COH ratio), and iron and arsenic content. The RBG micrographs show that most surficial ochre samples also are darker and more brown in color (due to high organic content), and higher in visible mineral inclusions.

*Elemental Characterization by NAA*
All ochre samples were prepared at MURR Archaeometry Laboratory using standard procedures reported elsewhere (85-87). Approximately 1 g of each ochre sample was ground into a fine power by agate mortar and pestle. All preparation instruments were carefully cleaned between specimens. Two aliquots were prepared from each powdered sample. One aliquot of ~75 mg was weighed into a high-density polyethylene vial used for short irradiations, and ~100 mg was weighed and sealed into high-purity quartz vials used for long irradiations. Individual sample weights were recorded to the nearest 0.01 mg. Standards were prepared from National Institute of Standards and Technology certified reference materials, SRM-1633b (coal fly ash) and SRM-688 (basalt rock), and quality control samples of SRM-278 (obsidian rock) and New Ohio Red Clay (in-house quality control).

Irradiation and Gamma-Ray Spectroscopy

Neutron activation analysis of archaeological materials at MURR consists of two irradiations and a total of three gamma counts (88, 89). Two thermal neutron irradiations were conducted to collect data on elements that produce short-, medium-, and long-lived radioisotopes. Ochre samples and standard reference materials in polyvials were irradiated via pneumatic tube system for 10 s at a flux of $8 \times 10^{13} \text{ n cm}^{-2} \text{s}^{-1}$. Samples were each allowed to decay for 25 min, at which point gamma ray energies for elements that produce short-lived isotopes (Al, Ba, Ca, Dy, K, Mg, Mn, Na, Ti, and V) were measured by a hyper-pure germanium detector (HPGe) for 12 min. The quartz-encapsulated samples were subjected to a 24-hour irradiation at a neutron flux of $6 \times 10^{13} \text{ n cm}^{-2} \text{s}^{-1}$. After a 7-10 day decay, the radioactive samples were measured for 2,000 sec to obtain data on medium-lived isotopes (As, La, Lu, Nd, Sm, U, and Yb), and again after 2-3 weeks for 8,200 sec to measure for long-lived isotopes (Ce,
Co, Cr, Cs, Eu, Fe, Hf, Ni, Rb, Sb, Sc, Sr, Ta, Tb, Th, Zn, and Zr). The spectral data were calculated to elemental concentrations using in-house software and calibrated to NIST reference materials by comparator method.

**Geochemical Results**

The NAA procedure produces elemental concentration values for 35 elements in most analyzed samples. Concentration values for all samples (including limits of detection) are provided in SI-Data 1. Some elements were significantly depleted below typical concentrations normally observed in Fe-oxides, including Cs, Rb, Ta, Tb, Th, Sr, Zr, Ba, K, and Ti. SI-Data 1 summarizes the averages and standard deviations for each duplicate group of samples.

When comparing averages between sampling locations at Sagitario (La Mina), LMR-P05, LMR-P20 and LMR-S (surface sample) are unique in major element chemistry. In LMR-P05, Fe is substituted by calcite in the bulk chemistry, whereas in LMR-P20 the Fe is substituted by Al, Ti, and K, attributed primarily to clay mineral content. Mg ranges from 0.25% - 0.8%, with the exception of LMR-P20, which has elevated Mg. Calcium concentrations range from 0.8%-2.5% for most samples, with the exception of LMR-P05 at ~19.01%. Aluminum ranges from 1.5%-2.4% for all samples, yet is elevated to 17.9% in LMR-P20. Potassium is only present above the limit of detection in sample LMR-P20. Sample LMR-S is depleted in iron, but enriched in aluminum, which is consistent with mineral phase identification by XRD. LMR-S is also highly enriched in rare earth elements, comparable to that of LMR-P20.

All samples from Sagitario (La Mina), with the exception of LMR-P20 and LMR-S, exhibit some degree of trace element depletion or dilution, which is attributable to element mobilization due to redox conditions. Manganese is highly variable regardless of depositional environment (inundated or non-inundated), with concentrations in P09 approaching 1.0%. This
variation may be a function of variable element substitution within the Fe-oxide matrix throughout the deposits, or localized ‘hotspots’ of elevated Mn. Elements Na and Ti are low or below limit of detection, with the exception of LMR-P20. Conversely, vanadium is elevated in all but LMR-P20, and also is comparatively depleted in LMR-P05, suggesting that it may be directly associated with Fe-oxide content as a transition metal substitution for Fe. Elements As, Ba, Ni, Co, As, and Sb are highly variable throughout, which also may be explained by element mobility. Chromium is fairly uniform throughout, and hafnium is the only consistently detectible high field strength element (HFSE). Elements that are poorly distributed or depleted below the limit of detection for most samples include Cs, Ta, Rb, Sr, Sc, Sr, and Zr. Rare earth elements (La through Lu, plus Th) are depleted in all samples, yet are enriched in sample LMR-P20 and LMR-S, a common result of element mobility due to leaching, pH, ferrolysis, or weathering (85, 90–92). However, U concentrations are inverse to this trend with enrichment in all samples and depletion in LMR-P20.

Ochre from Camilo Mina has high iron-oxide content that is comparable to that of Sagitario (La Mina), which is consistent with XRD results suggesting a goethite-dominated matrix. Monkey Dust ochre is depleted in iron and high in aluminum, comparable to values reported from the Sagitario (La Mina) surface sample.

Discussion

Qualitative and geochemical analysis of the samples collected from Sagitario (La Mina), Camilo Mina, and Monkey Dust suggests some variation in the relative qualities of the ochre. Samples from the Sagitario (La Mina) mining area are iron-enriched, dominated by fine-grained goethite particles, and lacking in coarse mineral inclusions. This ochre is high purity, has a noticeably higher color saturation, and is arguably a ready-made paint. Ochre collected from a
mining pit at Camilo Mina is also of high quality, comparable to that of Sagitario (La Mina) in terms of iron purity, grain size and texture, and low proportion of coarse mineral adjuncts. Ochre from Monkey Dust is coarse grained, low in iron oxide content, and higher in iron-enriched alumina-silicate (almandine). The ochre from Monkey Dust is still pigmentaceous, yet may have required grain separation via sieving or levigation, or mixing with another higher purity ochre source to enhance or extend its color and texture properties.

Notably, the ochre from Sagitario (La Mina) and Camilo Mina are significantly enriched in arsenic (SI-Data 1), a well-known active ingredient in early pesticide production. If the ochres from these underground caves were collected at least in part for their insect repelling properties (32), the Sagitario (La Mina) and Camilo Mina ochre would be highly suitable for this purpose, whereas Monkey Dust would be less effective.

**SI-Text 3: Calcite Raft and Ochre Formation**

*Calcite Rafts*

Calcite rafts form at the air/water interface in caves and caverns of the Yucatán through CO₂ off-gassing of stagnant, carbonate-rich waters (Fig. 5f, SI-Fig. 2a) (7, 93). These calcite rafts then sink to the bottom when they get too large, or when the water surface is disturbed by groundwater flow, hanging roots in the cavern, falling bat guano, or dripwater from the ceiling. This water disturbance and sedimentation of calcite rafts then forms piles or cones or larger sheet-like accumulations on the cave bottom (7, 22) (SI-Fig. 2a). Recent studies have demonstrated that these accumulations can record Holocene paleohydrology and changing groundwater chemistry using elemental (Sr/Ca, Cl/Ca) and isotopic proxies (d¹⁸O, d¹³C,
which can also be radiocarbon dated (7) (SI-Table 1 therein). Here, we show how calcite raft sheets found on the cave bottom of La Mina can also provide high-resolution records documenting water level and flooding of the cave passages (Fig. 4b, SI-Fig. 2a).

Large sheet fragments of calcite rafts (some as large as 10 cm in diam.) are found covering the bottom of La Mina in many locations, but also under narrow overhangs of limestone and within mining pits. Individual fragments of calcite rafts were once interconnected in a relatively continuous sheet on the water surface and were deposited on the bottom when groundwater dropped likely during a dry period (~ 10-20 cm; (93). Because calcite rafts could only form when the air/water interface was within the narrow height range below the overhangs, they provide a very accurate water-level estimate (SI-Fig. 2a; SI-Table 1). Calcite rafts accumulating in larger cave passages do not provide the same height resolution because the air/water interface could have occurred over a wider depth range (7). Calcite raft sheets found covering the bottom of excavation pits likely formed as rising groundwater levels reached the bottom of the pit but did not overtop its rim (SI-Fig. 2a). Our estimate of water level in La Mina from the calcite rafts provides a terminal age (~ 7-8 ka cal yr BP) for the mining activity and the flooding of the cave (Fig. 1b, 1d; SI-Table 2).

Ochre Formation

The karst landscape of the Yucatán has a high pedodiversity including thin Rendzina soils as well as thick accumulations of terra rossa or red soils (94, 95). Locally, in the surrounding study area, Rendzinatas dominate, but there are localized accumulations of red-soils in karst sinkholes and depressions (96).
Primary geological deposits of ochre were likely soil-pipes (La Mina); (97) vugs (La Mina), and thin beds (1-2 cm; Camilo Mina) within the Neogene limestone. Secondary detrital ochre deposits occurred between layers of flowstone (Sagitario - La Mina) and infiltrates directly from the surface through cracks, fissures, and solution pipes in the overlying limestone (Sagitario, Monkey Dust; Fig 1b, SI-Fig. 2b, 2c; Fig. 5a, d, e, f, g, h, i). Primary ochre deposits are likely due to cave speleogenesis cross-cutting unconformities within the limestone, exposing soil pipes in La Mina and thin beds in laminated lithographic limestones with mudcracks in Camilo Mina. Secondary detrital ochre likely originated through erosion and deposition of these primary deposits and as direct inputs from surficial sources (94), which were then covered in flowstone during cycles of groundwater level rise and fall throughout the Quaternary.

An alternate origin for the ochre in La Mina could be infilling of the cave with fine muddy sediment during the Stage 5e highstand of sea-level followed by oxidization and erosion of that sediment with the subsequent lowstand (SI-Fig. 2d). Van Hengstum et al. (98) found a thin (~1 cm) red mud deposit in the nearby Actun Ha Cave system containing marine foraminifera, which was also covered with a thin calcite crust. Because the red mud contained marine foraminifera which formed when the cave passage was flooded, and the calcite crust formed subaerially indicating a drop in sea-level, the mud deposit was associated with the Stage 5e highstand of sea-level. It is possible that the large pits of ochre (i.e. vs soil-pipes) found in La Mina could have formed under a similar scenario, but we do not believe this to be the case, because there was ochre in a vug on the cave wall (Fig. 1b, SI-Fig. 2b) which was high above the bottom (~1 m; Fig 1b; SI-Fig. 2). If the elevation of this vug reflects the original level of ochre in the cave, it would represent a very large amount of sediment to be weathered and eroded under the Stage 5e highstand scenario, which doesn’t seem plausible based on the small restricted cave
passages of La Mina. More detailed sampling and mapping are required to be definitive on the ochre origin in La Mina.

Estimating the amount of ochre mined in La Mina is complicated by the geologic origin of ochre deposits. Pit size and volume are not necessarily correlated with the amount of ochre retrieved during mining; not all of the pits contained viable ochre deposits, and some pits could have been failed prospection sites. In La Mina “soil-pipes” of ochre are often irregular in dimension and may have contained large fragments of limestone (70). For instance, the pit shown in Fig. 5f (main text) is approximately 4 x 1 x 1 m in size and the pit walls are highly irregular. This pit still retains a thin covering of ochre on the walls, and thus the original ochre volume, but the irregular dimensions of both the pit and the mine spoil presents difficulties for accurately estimating ochre volume, but especially in an underwater context where time is limited (Fig. 5f). Ochre beds between red-stained flowstone layers in La Mina could be more easily estimated; they were generally thin (~ 1-5 cm) with less complex geometries (Fig. 2d). However, these pits may have not yielded viable ochre deposits because the bed was too thin, or was too coarse in texture and these pits may have been failed prospection sites (Fig. 5g).

Ochre volume in Camilo Mina can be more easily estimated because it was mined from a sole source deposit with a more predictable geometry (1-2 cm thick lenticular beds that pinch out laterally). The mine pits all penetrate to a common depth (~ 23.5 m water depth) and the pit shape and surface area seems to follow the ochre bed as it thinned in the stratigraphy (Fig. 5d, e). A new pit was then started a short distance away (~ 0.5 to 2 m) and the process was repeated, so pit surface area is largely a reflection of the areal extent of the ochre bed, and its thickness is more consistent than the ochre deposits in La Mina.
Ochre and excavation pits were more difficult to discern in Monkey Dust because of the extensive flowstone covering the cave walls and bottom. However, a prominent pile of ochre from a dissolution pipe in the ceiling shows the detrital input of ochre from the surface. The ochre pile is closely associated with a stone marker cairn and a thick charcoal deposit (Figs. 5a, b, c).

**SI-Text 4: Carbonized Wood Identification**

*Introduction*

The formation of charcoal, by heating wood in an inert atmosphere, has been shown by several authors to retain the cellular structure of the original trees (99-107). Most of these studies focused on the amount of shrinkage of charcoal associated with heating, and a few of them used charcoal cellular structure as a taxonomic tool (108). Charcoal identification is widely practiced in archaeology and several commercial laboratories provide the service. The present study, using fragments of charcoal found in the flooded cave sites of Hoyo Negro (HN) and La Mina (LM) located on the Yucatán Peninsula, seeks to identify the taxonomic identity of their parent tree species. These samples were splits of samples that had been submitted for identification by the archaeological team for radiocarbon dating as well as bulk samples of charcoal from HN examined to capture a broader array of tree species than might have been represented by the small group of radiocarbon samples.

*Materials and Methods*

Charcoal samples were trimmed so the cross-sectional surfaces (XS), radial sectional surfaces (RS), and tangential sectional surfaces (TS) were exposed on the same sample. The
trimmed samples were placed on aluminum stubs using either double-sided carbon tape or carbon glue and sputter-coated with a thin layer (30 nm) of gold palladium, before being examined and photographed with a Tescan LYRA 3 GMU scanning electron microscope. A Stereomaster dissecting microscope (7-45X) was used, both to facilitate trimming of XS, RS, and TS surfaces and to sort charcoal samples into “Torchwood” and “Non-torchwood” categories, once the significance of the expanded ray cavities had been determined using the SEM.

A combination of XS, RS, and TS observations were used to characterize taxonomically diagnostic cellular features of tree charcoal. Finally, based on SEM studies, these taxa can be categorized as either “Torchwood” or “Non-torchwood,” based on cell structure. Tentative identification of the taxa was done using standard comparative wood anatomical (cellular) features described by the IAWA Committee on Nomenclature (109) (1957) and IAWA (110). Metcalf and Chalk (111), and the online database InsideWood (112) were used to determine taxonomic relationships.

Results

In this study of the charcoal, the cellular structure of 10 tree taxa have been identified to date. All of the taxa are diffuse-porous hardwoods with simple perforation plates, similar in identity to extant forest species on the Yucatán Peninsula today. Cellular features of taxonomic value include vessel or pore arrangement, inter-vessel pitting size and type, vascular ray types, presence or absence of axial parenchyma, and fiber wall thickness.

The initial study of charcoal fragments focused on samples collected from the HN cave system collected in November 2018, of which a total of four genera were identified based on the diagnostic cellular structure of the fragments. These genera represent three plant families
commonly found today on the Florida Keys and the Yucatán Peninsula. These genera are Xanthoxylon and Amyris in the Rutaceae (Citrus family), Metopium in the Anacardiaceae (Brazil nut family), and Ficus, in the Moraceae (Mulberry family). Subsequent study of additional charcoal samples from earlier collection episodes at HN identified three additional genera from two plant families, Swientenia in the Meliaceae (Mahogany family) and Bursera and Protium in the Burseraceae (Torchwood or Frankincense family).

Of the seven genera identified to date, three (Amyris, Bursera and Protium) are resinous, and in extant members exhibit enlarged vascular ray cells called resinous idioblast. All seven of the types of charcoal found in HN display normal cellular structure (see SI-Fig. 4a). Samples of charcoal found in La Mina belong to the three resinous genera seen in HN, but exhibit an unusual type of cellular structure, in the form of dramatically expanded vascular ray structure, resulting in massive cavities (SI-Fig. 4b). Such cavities are very unusual and may have developed from explosive combustion of resin in rays composed of resinous idioblasts.

The samples studied to date fall into two broad categories: fragments of charcoal whose cross (XS) and tangential (TS) surfaces are marked with expanded vascular ray cavities described above (see SI-Fig. 4a), and those that do not exhibit the expanded vascular ray cavities (SI-Fig. 4b). Those samples with the massive expanded ray cavities were found exclusively in the specimens from La Mina, while the same resinous genera, when found in the HN cave system do not exhibit such cavities.

For reasons not yet understood, the resinous woods used for torches in Sagitario appear to have burned hotter than in the charcoal from natural forest fires. We hypothesize that the resinous taxa exhibiting expanded vascular ray cavities are from trees which were burned in a high oxygen environment, such as torches or illumination fires, while such resinous charcoal
from HN represents wood carbonized in a low oxygen environment, such as among masses of smoldering wood on a forest floor. Such resinous tree species are common on the Yucatán Peninsula today, and are often referred to as torchwoods or candlewoods. The resin from such trees, particularly Protium copal, is often used as incense in local churches. In this study, we refer to charcoal from resinous trees with expanded cavities as torchwoods and those from resinous and non-resinous trees which do not exhibit such cavities as non-torchwoods.

All of the charcoal samples found in La Mina exhibit expanded vascular ray cavities and were identified as resinous torchwoods, while in the Hoyo Negro cave system none of the resinous taxa exhibit such cavities. A possible explanation for this difference is that much of the resinous wood burned in La Mina was carried into the cave, to be used for light needed for the mining of ochre, while both the resinous and non-resinous samples found in the HN cave represents the natural mix of tree species occurring around the entrance of the cave. The fact that 100% of torchwood charcoal from La Mina exhibited expanded ray cavities suggests selective collection of resinous woods.

**SI-Text 5: Cave Mining in the Maya Lowlands**

For Mesoamerica peoples, caves were an integral part of the sacred landscape, and were widely associated with the concepts of emergence and fertility (35, 113). Among the Maya, notions of the cave as heart-of-earth were prevalent (114), as were associations with water and rain deities (36), especially across the northern Yucatán Peninsula. Evidence of Maya ritual practice in caves is diverse and abundant and, in addition to a range of material culture associated with offertory behaviors, mortuary practices (33), and rock art traditions (115-117)
have been well documented. Incontrovertible evidence for the ritual appropriation of caves by the ancient Maya can also be found in the construction of religious architecture in subterranean spaces (35, 118).

In addition to the collection of water from caves – notably for ceremonial purposes (119) – the ancient Maya extracted geological materials, which may also have been imbued with symbolic significance (120). In the Yucatán, kancab (red earth) and sah kab (white earth) were mined from caves. Andrews (37) mentions red earth excavated from deep within Balankanche Cave, and Smith (38) reports kancab extraction from Cenote Ch’en Mul. Brady et al. (121) describe extensive red-earth mining from caves in the Cobanerita area in the Petén of Guatemala. In Cueva de las Pinturas (121), a flowstone cap was intentionally broken to reveal the red clay-like material, which was intensively mined (120).

The term sah kab can refer to both the powdery material – more commonly referred to as sascab – excavated from beneath limestone caprock and used in construction, and a material used as temper in pottery (122). Sascaberias (or sascab quarries) are prevalent across the Maya area. Often, natural karst depressions or collapse features were expanded via sascab mining, resulting in cave-like rooms, tunnels, or multi-chambered affairs (123). In Slater’s regional study in central Yucatán, nine sascaberias where documented and six caves revealed evidence of sascab mining (123). Most striking was the extent of sascab mining activity in Aktun Kuruxtun (123), where tool marks are plainly visible on the walls of the cave. Mining of dolomitic powder (similar in texture and appearance to sascab) was documented in Actun Toh in Quintana Roo (120, 124). Here, the limestone shelves that resulted from excavating the vein of poorly lithified dolomite were snapped off by the miners and stacked along the chamber’s entrance path (124).
Arnold (122, 125) and Arnold and Bohor (126) have proposed that clays rich in palygorskite (attapulgite), mined from caves in Yucatán, were used in the production of the pigment known as Maya blue. This variety of white clay is referred to as saklu’um, and Arnold and Bohor (126) suggest that the community of Sacalum takes its name from the clay mined from the town’s cenote. Brady and Rissolo (120) assert that the Sacalum clay source was more likely used for medicinal purposes, as also noted by Arnold and Bohor (126) and by Folan (127).

Speleothem breakage and removal is ubiquitous across the Maya area. Though speleothems as a possible source of calcite temper for pottery has been discussed, there is compelling evidence for a more symbolic function, including the inclusion of stalactites and stalagmites in ritual contexts at surface sites (34, 113, 128, 129). In Balam Na Cave 1, in the Petén of Guatemala, Brady et al. (113) determined that nearly 60% of the stalactites had been broken off. Regional studies by Rissolo (124) and Slater (123) document the extensive and intensive nature of speleothem mining in Yucatán and Quintana Roo, with some caves being nearly completely denuded of dripstone formations (124).
SI-Fig. 1a-e: Optical and Electron Micrographs of Ochre Samples

SI-Fig. 1a: Light microscope images of untreated and heated ochre from Sagitario (La Mina). (A) Unheated ochre sample LMR-P01A. (B) Heated ochre sample LMR-P01A. (C) Unheated ochre sample LMR-P19A. (D) Unheated ochre sample LMR-P19A. (B) Heated ochre sample LMR-P19A. Scale bars for all images = 1 mm.
SI-Fig. 1b: Backscattered electron micrographs of LMR-P04 and LMR-P09. (A, B, C, D) LMR-P04: Calcite and periclase particles, typically > 10 µm, are covered in flaky, fine-grained iron oxide particles. (E) LMR-P09: the ochre sample matrix is dominated by fine-grained (< 10 µm) goethite particles. (F) LMR-P09: alabandite (MnS) particle coated in fine-grained goethite.
SI-Fig. 1c. Electron micrographs of LM-P20 and LMR-S. (A, B) LMR-P20: stacks of platy kaolinite particles. (C, D) LMR-P20: large (> 50 µm) chunks of blocky bohemite crystals (lower). All large particles are covered in a fine layer of small (< 10 µm) iron phosphate particles. (E, F) LMR-S: surface sample is composed primarily of large (> 50 µm), angular, coarse-grained particles of quartz, bohemite, and almandine. (G) LMR-S: few iron-oxide enriched particles were present, shown here in light contrast (H) LMR-S: a zircon crystal interspersed in the sediment matrix.
SI-Fig. 1d: Secondary and backscattered electron micrographs of ochre from Camilo Mina and Monkey Dust. (A, B, C) Camilo Mina: the ochre matrix is dominated by a matrix of goethite particles ranging in size from 5-20 µm. (D) Camilo Mina: fine-grained, flaky goethite particles coat a small dispersion of CaO-enriched particles. (E, F, G) Monkey Dust: the ochre matrix is coarse-grained with large angular particles enriched in quartz, almandine, and periclase. Note the lack of fine-grained iron oxides as compared to Camilo Mina and Sagitario (La Mina) ochres. (D) Monkey Dust: a Ti-enriched particle embedded in a larger particle of almandine.
SI-Fig. 1e: Results from core scanner analysis of cave ochre from La Mina (LMR) and Camilo Mina (CM), and surficial ochre from La Mina (LM) (LMR-PBC = Pu’bix cavern; LMR-S1, S2, S2 = terra rossa soils). RAD = radiographs. RBG = optical imaging. Fe and As = µ-XRF counts. INC/COH = ratio of incoherent to coherent scattering, indicative of relative organic content and porosity of the samples.
SI-Fig. 2: Conceptual diagrams of calcite raft and ochre formation.
(A) Conceptual diagram illustrating the types of calcite rafts deposits and their relationships with water depths.
(B, C) Probable origin of ochre deposits found in Sagitario, La Mina, Camilo Mina, Monkey Dust.
(D) Alternate origin for the pit ochre (soil-pipe) depicted in B and C.
SI-Fig. 3: Cairns and Fire Pit Feature at La Mina. Heating of the ochre on the limestone cave floor has caused a circular stain (~ 40 cm in diam.), which is close-by a stone-marker cairn. Note to the right of the circle, the generally smooth stone surface is punctuated by a lenticular island created by the cairn and a large stone, between which are smaller stones. This appears to be a trail in which the larger stones protected the smaller clasts from being kicked aside. Repeated treading has worn through the sediment on the cave floor. Also shown in Fig. 3f (main text). Images A-C are grabs from SFM 3D models of the cave floor. Photo credits: S. Meacham (CINDAQ).
SI-Fig. 4: Characteristics of non-resinous and torchwood woods. (A) An example of cellular detail exhibited in cross section (XS) by a typical non-torchwood charcoal sample identified as the genus Zanthoxylon. Note the presence of a growth ring terminus, short radial chains of pores or vessel elements, thin-walled wood fibers, and non-resinous, narrow vascular rays. This type of wood anatomy is characteristic of the Rutaceae or Citrus Family. (B) An example of cellular detail found in XS by a typical torchwood charcoal sample identified as the genus Protium copal. Torchwoods are characterized by the presence of dramatically expanded vascular ray cavities, long radial multiples of pores or vessel elements, thick-walled fibers, and resinous vascular rays which have expanded into the typical cavities. This type of wood anatomy is typical of the Burseraceae or the Torchwood Family. Images have a magnification of 515x.

SI-Fig. 5: Hyperlinks to 3D Models and Video of Features in La Mina. Image credits to authors (S.M., F.D., C.L.M.) from Centro Investigador del Sistema Acuífero de Quintana Roo AC (CINDAQ) and (D.R.), Cultural Heritage Engineering Initiative (CHEI), University of California, San Diego.

SI-Fig. 5a: Interactive 3D model of mining area at La Mina, seen in Fig. 2d (main text). https://sketchfab.com/3d-models/dm-m4-area-abbcecc9d5430b90f9ad1d5c1cf512

SI-Fig. 5b: Interactive 3D model of cairns seen in Fig. 3e (main text). https://sketchfab.com/3d-models/ndm-z-area-6db9cad8a3b3474f9f3477f6426ba495

SI-Fig. 6: Map of Sistema Sagitario. Due to large file size, image is provided through the following secure link to Figshare repository: https://figshare.com/s/136e4d349ee401170783
SI-Fig. 7: Passage broken through a forest of speleothems. (A) Wide enough for a single person to pass, this feature is located between Entrada Pu`bix and the intensively excavated passages of La Mina, about 100 m from the surface entrance. (B) The thin stalactites may have regrown after being broken, but larger stalagmites show intensive breakage (indicated stalagmite is ~10-15 cm in diameter). Photo credits: F. Devos (CINDAQ).
SI-Table 1: Radiocarbon dates from calcite rafts. Calcite raft sheets found under overhangs and overlying mined surfaces in Sagitario (La Mina). See SI-Fig. 2a for depositional context.

| SITE Sample | Station | Depth (m) | Description | D-AMS # | \(^{14}\)C Age (yBP ±1σ) | Reservoir Corrected | Age Cal yBP (2σ) |
|-------------|---------|-----------|-------------|---------|--------------------------|---------------------|------------------|
| SAGITARIO (LM) | | | | | | | |
| B6 | 3 | 9.8 | Thin continuous sheet of calcite rafts - ceiling @ 8.2m | 27463 | 8276±34 | 7009±34 | 7937-7760 |
| B7 | 4 | 10.6 | Thin continuous sheet of calcite rafts - ceiling @ 7.1m | 27464 | 8283±38 | 7016±38 | 7941-7757 |
| B9 | 1 | 9 | Thin continuous sheet of calcite rafts - ceiling @ 8.6m | 27465 | 8410±37 | 7143±37 | 8022-7879 |
| B10 | 2 | 10.3 | Thin continuous sheet of calcite rafts - ceiling @ 10.2m | 27466 | 20816±73* | … | … |
| SITE Sample | Station | Depth (m) | Description                                                                 | D-AMS # | $^{14}$C Age (yBP ±1σ) | Age Cal yBP (2σ) |
|-------------|---------|-----------|-------------------------------------------------------------------------------|---------|------------------------|------------------|
| SAGITARIO (LM) |         |           |                                                                              |         |                        |                  |
| P06         | 7       | 10.7      | Charcoal concentration on ledge on cave wall near a quarrying pit              | 30619   | 10,013±45              | 11,720-11,290    |
| P07         | 5       | 11        | Mound shaped accumulation of charcoal below layer of calcite framboids        | 30620   | 9,118±42               | 10,400-10,210    |
| P10         | 7       | 11        | Detrital charcoal on cave bottom near the concentration P06                  | 30621   | 9,041±47               | 10,280-10,152    |
| P14         | 8       | 11        | Charcoal concentration on floor beside large “cauldron” in limestone with charcoal | 30623   | 10,123±57              | 12,020-11,420    |
| P16         | 15      | 9.2       | Large fragments of detrital charcoal originating from upslope concentration and close to a large rock cairn placement | 30624   | 9,728±51               | 11,240-10,870    |
| P11         | 1       | 9         | Detrital charcoal in cave bottom just downslope from P12                    | 27467   | 9,650±43               | 11,200-10,790    |
| P12         | 1       | 7.8       | Lens-shaped charcoal concentration imbedded in speleothem flowstone on sidewall | 27468   | 9,555±37               | 11,090-10,720    |
| P13         | 17      | 9.3       | Charcoal concentration below flowstone                                       | 30622   | 9,685±67               | 11,230-10,790    |
| P21         | 17      | 9.3       | Charcoal concentration below flowstone - replicate of P13                   | 30625   | 9,812±74               | 11,410-11,080    |
| P22         | 18      | 8.9       | Thick bed of charcoal 5-10 cm thick below flowstone                           | 30626   | 9,845±55               | 11,390-11,180    |
| CAMILO      |         |           |                                                                              |         |                        |                  |
| CM S15      | 23.4    |           | Grand Canyon Passage - in excavation pit                                     | 34385   | 9,544±40               | 11,090-10,720    |
| CM S11      | 23.3    |           | Grand Canyon Passage - in excavation pit                                     | 34386   | 9,850±41               | 11,330-11,200    |
| MONKEY DUST |
|-------------|
| MD 5a       | 7.5 | Charcoal concentration below flowstone | 34384 | 10,146±45 | 12,033-11,611 |
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