



Ochre-based compound adhesives at the Mousterian type-site document complex cognition and high investment

Patrick Schmidt, Radu Iovita, Armelle Charrié-Duhaut, Gunther Möller,
Abay Namen, Ewa Dutkiewicz

► To cite this version:

Patrick Schmidt, Radu Iovita, Armelle Charrié-Duhaut, Gunther Möller, Abay Namen, et al.. Ochre-based compound adhesives at the Mousterian type-site document complex cognition and high investment. *Science Advances* , 2024, 10 (8), pp.eadl0822. 10.1126/sciadv.adl0822 . hal-04484835

HAL Id: hal-04484835

<https://hal.science/hal-04484835>

Submitted on 29 Feb 2024

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



ANTHROPOLOGY

Ochre-based compound adhesives at the Mousterian type-site document complex cognition and high investment

Patrick Schmidt^{1,2*}, Radu Iovita³, Armelle Charrié-Duhaut⁴, Gunther Möller¹, Abay Namen^{1,5}, Ewa Dutkiewicz^{1,6}

Ancient adhesives used in multicomponent tools may be among our best material evidences of cultural evolution and cognitive processes in early humans. African *Homo sapiens* is known to have made compound adhesives from naturally sticky substances and ochre, a technical behavior proposed to mark the advent of elaborate cognitive processes in our species. Foragers of the European Middle Paleolithic also used glues, but evidence of ochre-based compound adhesives is unknown. Here, we present evidence of this kind. Bitumen was mixed with high loads of goethite ochre to make compound adhesives at the type-site of the Mousterian, Le Moustier (France). Ochre loads were so high that they lowered the adhesive's performance in classical hafting situations where stone implements are glued to handles. However, when used as handheld grips on cutting or scraping tools, a behavior known from Neanderthals, high-ochre adhesives present a real benefit, improving their solidity and rigidity. Our findings help understand the implications of Pleistocene adhesive making.

INTRODUCTION

In archaeology, evolutionary concepts like modern behaviors (1) or complex cognition (2) have been argued for based on tool (3) and raw material behaviors (4), the transformation of materials (5, 6), and composite technology or hafting (7, 8). Adhesives play an important role in arguments about the implications of hafting because, in some instances, Stone Age foragers used elaborate production techniques to make them (9–11). Adhesive making may therefore contain information about innovative behavior, social learning, and cumulative culture (2, 5, 12). In the African Middle Stone Age (MSA) [~300 to 30 thousand years (ka) ago], this debate was fueled by the finding that *Homo sapiens* combined naturally sticky materials with other ingredients, such as ochre (13, 14) or bone fragments and quartz (15), to produce adhesives with properties not otherwise available in nature (16, 17). Some researchers (18) argue that mixing substances following specific recipes requires analogical reasoning and forward planning. If so, compound adhesives might be one of our best indicators of elaborate cognitive processes in human evolution. In Eurasia, Neanderthals also used naturally available adhesives, such as bitumen (19) or tree resins [in one case possibly mixed with beeswax (20)]. In some instances, they made tar by distilling birch bark (5, 21, 22). European *H. sapiens* also used adhesives from the late Aurignacian and Gravettian (<33 ka) on (23–25), but their botanical origin and production techniques are not well understood [an exception is the use of bitumen in Eastern Europe (26)]. The implications of Neanderthal birch tar are still debated [compare (27, 28)], but what is certain is that Neanderthals made the effort to produce it, although other adhesive

substances could simply have been collected in nature [see the discussion in (9, 29, 30)]. Thus, similarly to the modern human record from Africa, the known European adhesive technology documents innovative behavior and even cumulative cultural transmission of techniques (31, 32). In addition, from the Gravettian on and in later periods, there are indications that European *H. sapiens* made compound adhesives using ochre (24, 25), a behavior similar to that of early *H. sapiens* in Africa. However, neither Neanderthals nor early *H. sapiens* present in Europe before 40 ka (33) have been shown to make ochre-based compound adhesives like those known from Africa (34). Therefore, the question whether early European adhesives from the Middle Paleolithic have the same cognitive implications as those made in the African MSA remains unanswered.

Here, we report on the discovery of such ochre-based multicomponent adhesives. For this, we analyze so far unstudied artifacts from the type-site of the Mousterian technocomplex (Le Moustier, France) that are curated at the Museum für Vor- und Frühgeschichte in Berlin (Germany). The Berlin Le Moustier collection was excavated at the upper terrace of Le Moustier shelter by Swiss archaeologist Otto Hauser in 1907. Because these artifacts have never been studied before and even remained individually packed and untouched since the 1960s, they allow unique preservation conditions for organic residues.

RESULTS

We identified five lithic artifacts with traces of red and yellow colorants on their ventral and dorsal sides (Fig. 1). Typologically (35), one is an atypical Levallois flake (no. Va 7136), one an end-notched flake (no. Va 7157.6), one a retouched flake (no. Va 7167.9), one a retouched Levallois blade (no. Va 7158.7), and one a side scraper (no. Va 7157.24). In all cases, red and yellow stains are restricted to one portion of the tools, forming a wall effect that effectively separates the portions of the tools with stains from the clean opposite portions (Fig. 1, but also see figs. S1 to S5). Four of the flakes also show traces of a black residue associated with red colorants (table S1; Fig. 1, C and H; and fig. S6). In one case, black residue appears as a visually

¹Early Prehistory and Quaternary Ecology, Department of Geosciences, Eberhard Karls University of Tübingen, Tübingen, Germany. ²Applied Mineralogy, Department of Geosciences, Eberhard Karls University of Tübingen, Tübingen, Germany. ³Center for the Study of Human Origins, Department of Anthropology, New York University, New York, NY 10003, USA. ⁴Laboratoire de spectrométrie de masse des interactions et des systèmes (LSMIS), Strasbourg University, CNRS, CMC UMR, Strasbourg 7140, France. ⁵Department of Sociology and Anthropology, School of Sciences and Humanities, Nazarbayev University, Astana, Kazakhstan. ⁶Staatliche Museen zu Berlin, Museum für Vor- und Frühgeschichte, Berlin, Germany.

*Corresponding author. Email: patrick.schmidt@uni-tuebingen.de

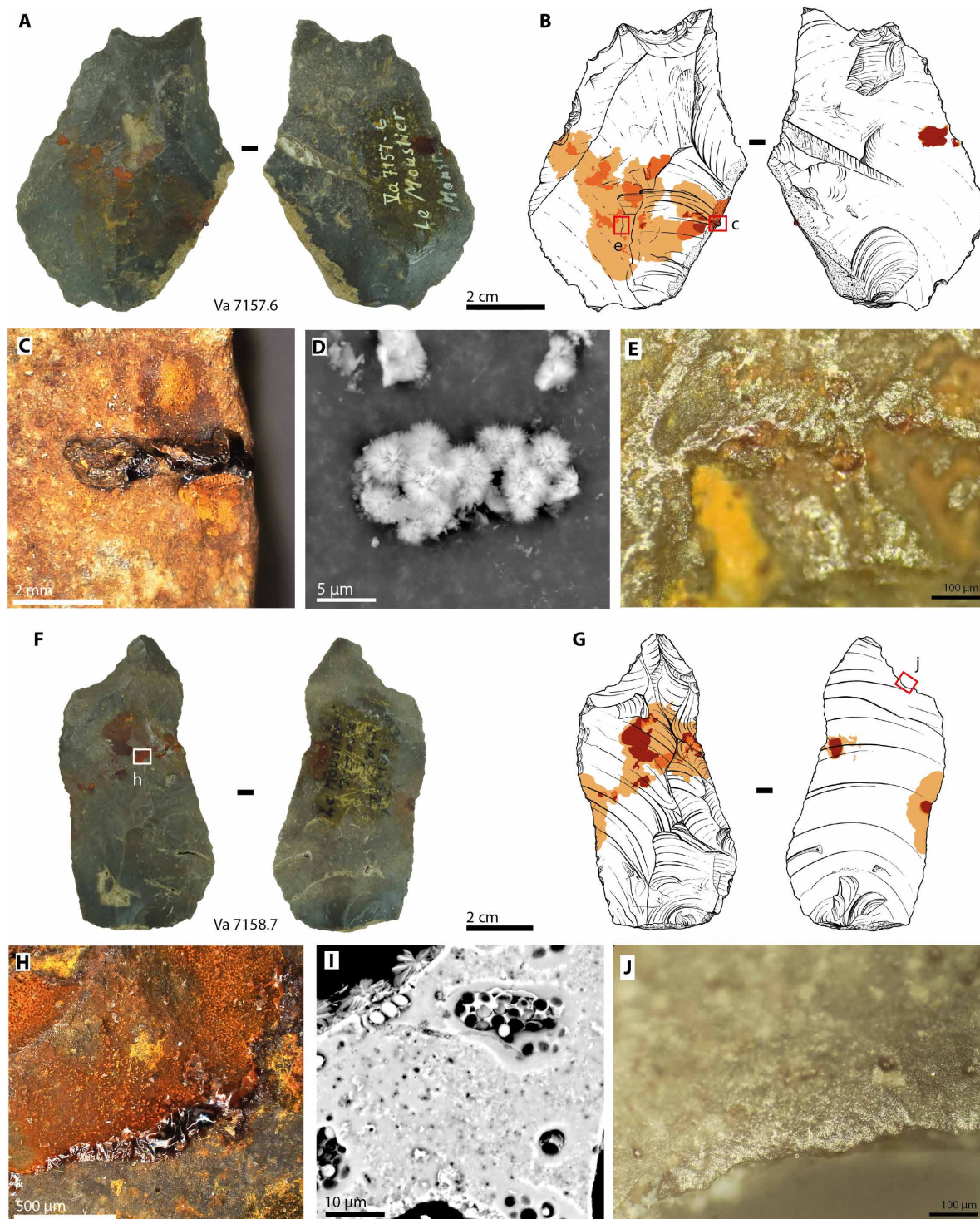


Fig. 1. Photographs, drawings, and details of Le Moustier artifacts nos. Va 7157.6 and Va 7158.7. (A) Photographs of dorsal and ventral surfaces of artifact Va 7157.6. (B) Drawing showing the distribution of residue remains on both sides of the artifact. The red box marks the frame of the detail shown in (C). (C) Detail of artifact no. Va 7157.6 showing an adhesive protuberance. (D) Scanning electron micrograph of spherulites constituting the powder removed from artifact no. Va 7157.6. (E) Micrograph showing polish caused by abrasion in the presumed prehensile portion of the tool. (F) Photographs of dorsal and ventral surfaces of artifact no. Va 7158.7. (G) Drawing showing the repartition of residue remains on both sides of the artifact. (H) Detail of artifact no. Va 7158.7 showing adhesive residues. (I) Scanning electron micrograph of the residue removed from artifact no. Va 7158.7. Note the spherulites on top of the micrograph and air bubbles trapped in the solid adhesive mass. (J) Micrograph showing invasive use wear polish in the presumed active edge of the tool. Drawings by D. Greinert.

shiny, ~2.5-mm-long, and ~1-mm-high protuberance (Fig. 1C). The distribution of colorant stains and their association with black residue are consistent with one portion of the pieces being covered by an adhesive containing red colorant. The opposite portion would then have acted as the active part of a composite tool. To test this hypothesis, we investigated the pieces for traces of wear. We found micro-fractures and localized polishes on the presumed active edges of four tools (Fig. 1J and figs. S1 to S5), documenting that they were used. Moreover, we found bright polish and striations under colorant stains, away from the edges, on dorsal and ventral faces of all five tools (Fig. 1E and figs. S1 and S3 to S5). Parts of the raised topography (the ridges of previous removal scars) are abraded in these zones. Such polishes, which are typical of hafting wear (36–38), are found on relatively large areas across the entire surface of the presumed hafted portion of the tools but not outside of it, and not only at the limit of the residue. We interpret these observations as evidence that the tools were held in an adhesive that still allowed movement of the stone tools or that underwent plastic deformation, with the colorant acting as an abrasive agent. The restriction of polished zones and striations to the presumed hafted parts rules out postdepositional processes as their cause (39). These findings support the hypothesis that the Le Moustier artifacts were part of composite tools. They were assembled by gluing them to shafts or handles using an adhesive, or alternatively, as Neanderthal archaeological examples show (22, 27), the adhesive was molded onto the tools and functioned as the handle itself.

Chemical and structural analysis

We first conducted energy-dispersive x-ray spectroscopy (EDX; using a scanning electron microscope) on powders scraped from the surface of two of the pieces (acc. nos. Va 7157.6 and Va 7158.7; Fig. 1, D and F, and figs. S9 and S10). The powders consist of ~5- to 10- μ m-large spherulites that yield an EDX signal of iron and oxygen. Parts of the powders contain agglomerates held together by a substance between the spherulites that show no discernible structure but air bubbles (Fig. 1I). The EDX spectrum of the agglomerated parts shows sulfur and carbon in addition to iron and oxygen. To investigate what the spherulites and the adhesive itself are made of, we removed 1 mg of a protuberance where the adhesive is still intact (on acc. no. Va 7157.6, Fig. 1C) and analyzed it by transmission infrared (IR) spectroscopy. The spectral signature of the adhesive is consistent with an organic phase mixed with an inorganic filler (Fig. 2). The inorganic phase causes the strongest absorption bands of the IR spectrum. Two relatively sharp bands of the $\delta(\text{OH})$ and $\gamma(\text{OH})$ vibrations of $\alpha\text{-FeO}(\text{OH})$ (goethite) are observed at 890 and 800 cm^{-1} (40). The low-frequency envelope $<700\text{ cm}^{-1}$ is consistent with Fe-O vibrations in $\alpha\text{-FeO}(\text{OH})$ (41). Thus, the inorganic filler of the Le Moustier artifact is unambiguously goethite ochre. The organic fraction of the adhesive causes weak but sharp bands between 3000 and 2800 cm^{-1} that indicate symmetrical and asymmetrical $\nu(\text{CH}_2)$ and $\nu(\text{CH}_3)$ vibrations. The region between 1500 and 1000 cm^{-1} gives insight into the nature of this organic phase. The compound adhesive spectrum presents a weak band at 1458 cm^{-1} assigned to symmetrical $\delta(\text{CH}_2)$ and asymmetrical $\delta(\text{CH}_3)$ vibrations of the aliphatic group. This broad band is known from the asphaltene fraction of crude oil or bitumen (42). There are also a $\nu(\text{CO})$ band at 1125 cm^{-1} and a $\nu(\text{S=O})$ band of the sulfoxide group at 1021 cm^{-1} . The assignment of the 1021- cm^{-1} band to sulfoxide is strengthened by the finding of sulfur using EDX. Both

bands (1125 and 1021 cm^{-1}) are also commonly present in the IR spectrum of asphaltene (42, 43). Sulfur typically accounts for less than 0.1% of natural tree resins and ambers (44), supporting the attribution of the Le Moustier spectrum to asphaltene/bitumen. The interpretation of the region between 1430 and 1330 cm^{-1} , where a symmetrical $\delta(\text{CH}_3)$ vibration would be expected in asphaltene, is not straightforward. This is so because, in some goethite specimens, a hydroxyl bending vibration causes a weak band at 1401 cm^{-1} (45) overlying this asphaltene $\delta(\text{CH}_3)$ vibration. The Le Moustier spectrum also presents a weak but sharp KNO_3 band, either resulting from impurities of the KBr used for pelleting (46) or created by grinding the sample together with KBr powder during sample preparation. Thus, the organic fraction of the sample cannot be observed in this wave number region (the shape of the broad band observed near 1403 cm^{-1} is most likely the result of the convolution of the goethite hydroxyl band and that of the potassium nitrate contamination). Nonetheless, the organic region of the Le Moustier IR spectrum can be best explained by the insoluble fraction of bitumen (asphaltene). This interpretation is strengthened by the absence of a C=O band in the spectrum. Strong C=O bands between 1750 and 1700 cm^{-1} are characteristic of different esters, acids, and aldehydes. They would be prominent in the spectra of plant-derived adhesives like resins and their tars (47, 48), including birch tar (21, 49). The absence of such C=O bands in our adhesive spectrum and the finding of sulfoxide, which is absent in plant-derived resins and tars, make a botanical origin highly unlikely (fig. S7). To corroborate our finding that the Le Moustier residue is made from bitumen, we attempted to conduct gas chromatography–mass spectrometry (GC-MS) on the organic extractable fraction of the 1-mg sample used for IR spectroscopy. The attempt failed, not yielding a signal above the method's detection limit (also see fig. S8). This is not in contradiction with our results obtained by IR. Bitumen is mainly constituted of asphaltenes and contains a soluble fraction of less than 3%, which would potentially be visible in GC-MS (50, 51). Such low soluble content calls for larger GC-MS samples, which are not available on the Le Moustier artifacts. What is certain is that, if the residue consisted of birch tar or tree resin, both substantially more soluble, we would have found a specific di- or triterpenoid signature (52, 53). We did not observe plant-derived terpenoids.

To gain information on the association of bitumen and goethite, we analyzed the adhesive protuberance on piece no. Va 7157.6 visually with a stereomicroscope. It is mostly opaque and black, but one portion, where it is broken, gives insight into its interior. Evenly distributed goethite spherulites can be seen there (Fig. 3D). To observe the inner structure of the Le Moustier residue, we recorded a micro-computed tomography (microCT) scan of the protuberance. On CT images, it shows no discernible goethite inclusions, as expected at the 11.5- μ m resolution of our scan. Trapped air bubbles can be seen in the adhesive mass (Fig. 3, A to C). Around the bubbles, the adhesive's structure appears mostly homogeneous with a few zones, where goethite distribution seems to be slightly lower. This is consistent with goethite being homogeneously mixed with bitumen. The adhesive mass has an overall brightness (organic part and ochre filler together) of 1539 Hounsfield units (HU). Assuming a roughly linear relationship between gray values in HU and density in our CT scans (54, 55), the overall density of the adhesive can be calculated from its brightness value compared to the brightness value of the adjacent flint (1391 HU, for a known flint density of 2.58 g/cm^3) as 2.85 g/cm^3 . Assuming a density of 1.01 g/cm^3 of

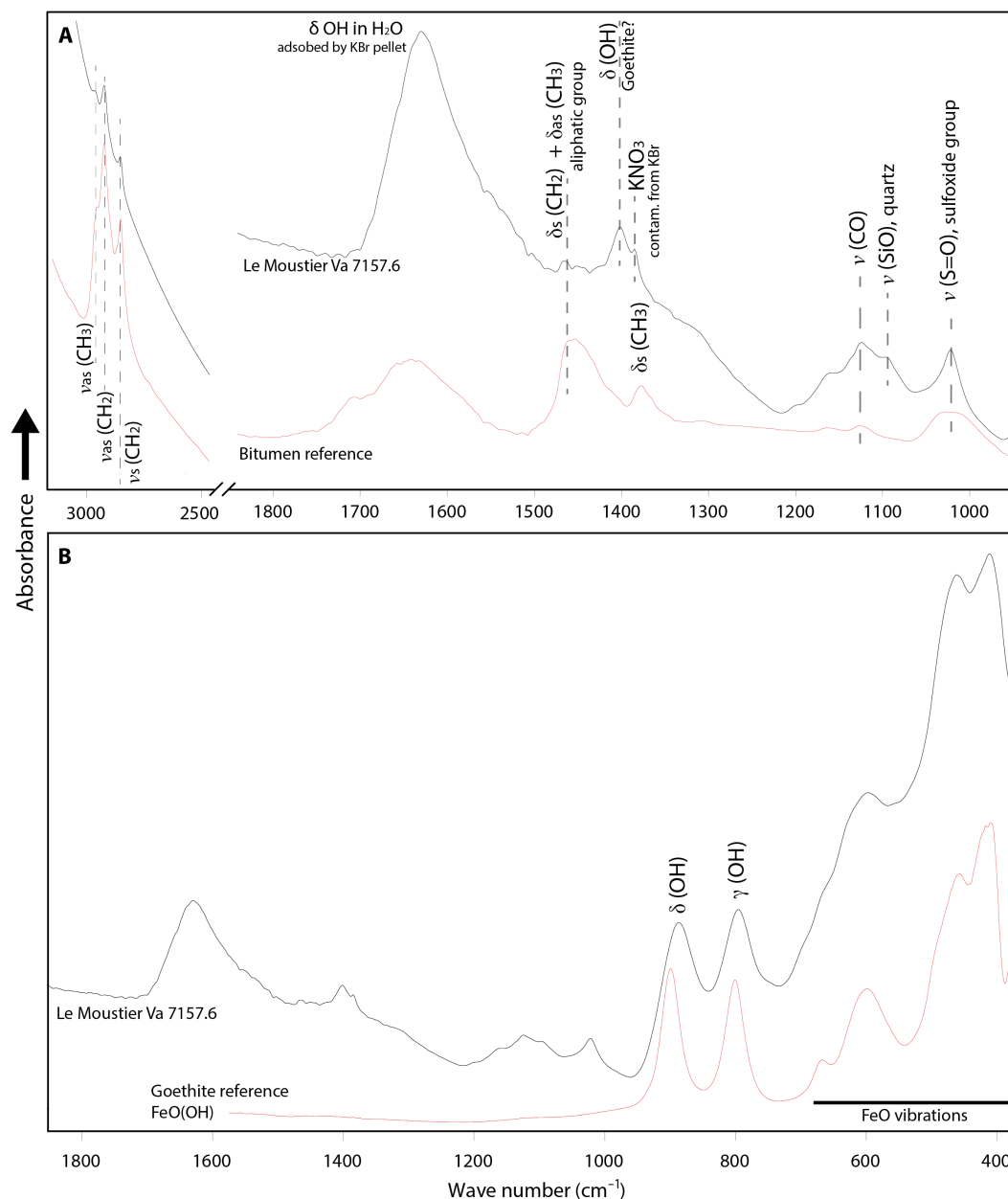


Fig. 2. Transmission IR spectra of the Le Moustier adhesive (removed from artifact no. Va 7157.6) compared to reference spectra of known substances. The 0.3 g–weighing KBr pellets contained 1 mg of sample. **(A)** Comparison with a bitumen reference spectrum. **(B)** Comparison with a Goethite [$\alpha\text{-FeO(OH)}$] reference spectrum. Note that the wave number region below $\sim 1000\text{ cm}^{-1}$ of the Le Moustier spectrum can be explained by goethite being mixed with the adhesive. The wave number regions 3000 to 2500 cm^{-1} and 1800 to 1000 cm^{-1} are best explained by the insoluble fraction of bitumen (asphaltene). Note the absence of a C=O band between 1750 and 1700 cm^{-1} in the Le Moustier spectrum, which would be characteristic for plant-derived adhesives.

bitumen (56) and a density of 4.3 g/cm^3 of $\alpha\text{-FeO(OH)}$ (57), the adhesive's overall density of 2.85 g/cm^3 implies that the compound adhesive was mixed from 55 and 45 wt % bitumen.

Experimental testing of the ochre-bitumen mixture

The proportion of filler to plastic component of the Le Moustier residue appears to be high in view of previous experiments conducted with inert filling loads in adhesive mixtures. Several experimental studies conducted on adhesives of different compositions show that ochre loading agents are beneficial if they account for 28% (16), 7.5

to 33% (58), or 10 to 30% (59, 60) of the compound adhesives. A few experimental studies report data on two-component adhesives containing larger parts of inorganic filler than plastic material (2, 17), but these studies investigated ochre mixed with *Vachellia* gum. To understand the usefulness of 55% goethite ochre in bitumen, we conducted lap-shear tests to measure adhesive strength (61), using naturally available bitumen (from the Massif Central region, France) and pure goethite ochre (from the Lessini Mountains, Italy). Natural bitumen can be collected in bituminous lakes as viscous liquid or as hardened air-dried mass on rock walls from where it seeps out

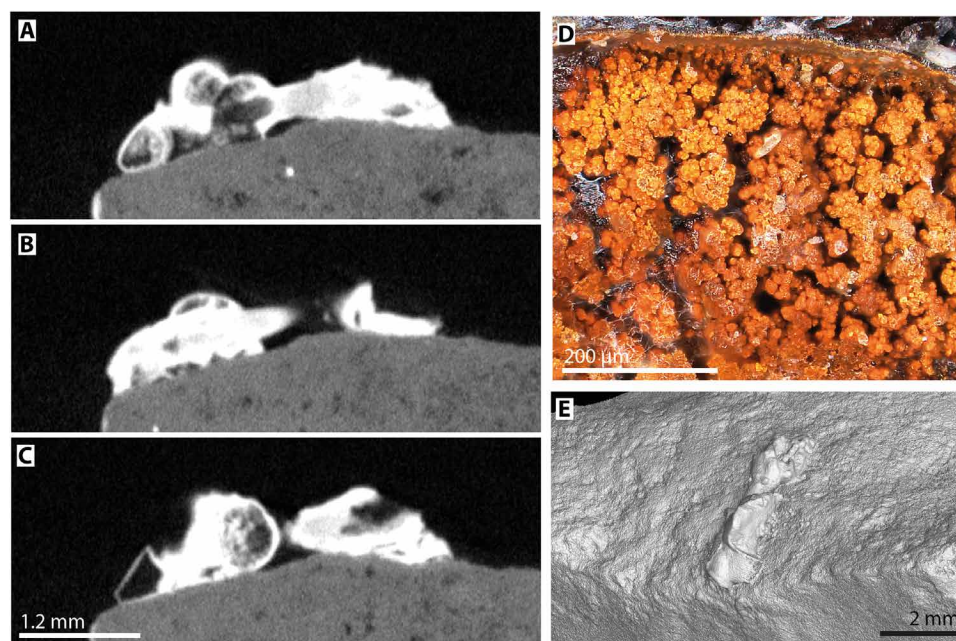


Fig. 3. Inner structure of the adhesive protuberance on artifact no. Va 7157.6. (A to C) MicroCT scans through the long axis of the protuberance. Mark the air bubbles trapped in the adhesive. Around air bubbles, the adhesive appears homogeneous, indicating that goethite is evenly distributed. (D) Stereo-micrograph showing goethite spherulites in the compound adhesive. (E) Iso-surface recorded by the microCT scanner, showing the outer shape of the adhesive protuberance.

(fig. S11). The viscosity of fresh bitumen is too low for laboratory strength testing. We therefore conducted a cooking experiment attempting to increase its adhesive strength (62). A subsample was removed every 20 min of cooking, half of which was mixed with 55 wt % ground goethite powder. Lap-shear tests were then conducted with the pure bitumen and the bitumen and ochre samples of each cooking step (all values reported in table S2). The maximum strength of pure bitumen adhesives was very low (Fig. 4), with $\tau_u < 0.01$ MPa (i.e., no strength signal could be measured, and the laps slid apart with no measurable resistance; we therefore conducted only a single test with these samples). Cooking for longer produced no change in pure bitumen, i.e., all τ_u values remained < 0.01 MPa. Compound adhesives yielded strength values greater than those of pure bitumen (Fig. 4A). Cooking for 80 min and more resulted in a two- to threefold increase of strength in compound adhesives as compared to pure bitumen: Bitumen cooked for 80 min + 55 wt % ochre had $\tau_u = 0.021 \pm 0.01$ MPa; bitumen cooked for 100 min + 55 wt % ochre had $\tau_u = 0.022 \pm 0.01$ MPa (Fig. 4B), as determined from five consecutive tests with these samples. After 107 min of cooking, we had to stop the experiment because the mass hardened almost instantaneously, becoming a tough solid with no adhesive properties, which could not be used for bonding together laps. The overcooked bitumen had no adhesive properties even when we tried to soften it with a Bunsen burner. We conducted a second lap-shear experiment with air-dried bitumen. Pure air-dried bitumen yielded a significantly higher maximum strength with $\tau_u = 1.49 + 1.07 - 0.81$ (as determined by 10 tests). Mixing in 55 wt % goethite ochre reduced the air-dried bitumen's maximum strength, resulting in $\tau_u = 0.55 + 0.53 - 0.47$ (as determined by nine tests; one test failed). Thus, making compound adhesives from air-dried bitumen and 55 wt % ochre has a negative effect, worsening the bitumen's strength by a factor of 3 (Fig. 4C). When mixed with fresh

bitumen, 55 wt % ochre increases its strength by a factor of 3. Nonetheless, our values measured on fresh bitumen are very low if compared to lap-shear data reported on other archaeological adhesives [which fall in the range of $\tau_u = 0.1$ to 1.5 MPa (9, 62–64)]. We therefore attempted to identify other possible performance gains of a high ochre load in bitumen. It is known from Neanderthal birch tar finds (5, 32) that adhesives were used as handles directly attached to stone tools (as opposed to being used for hafting stone tools to wooden handles or shafts). We therefore molded handles of fresh bitumen to stone tools (Fig. 4F; also see fig. S12). When manipulated, such pure bitumen grips are sticky to the touch and part of the bitumen is removed, leaving sticky stains on the hand, which are difficult to remove (Fig. 4E). When mixed in 55 wt % goethite ochre (Fig. 4G), bitumen grips feel more solid and are not sticky to the touch. When handling such a grip, no bitumen sticks to the manipulator's hands (Fig. 4F). Thus, mixing high ochre loads in fresh bitumen presents an advantage for such composite tools. This type of tool use is supported by paleoanthropological data, as both Neanderthals and Middle Paleolithic *H. sapiens* are known to have habitually engaged in hand movements that involve precision gripping (65, 66). These findings, and the observation that bright polishes and abrasion can be found across the colored part of the tools, make it most likely that the five stone tools were used in such adhesive grips rather than hafted to a rigid handle.

DISCUSSION

The Le Moustier artifacts show that the European Middle Paleolithic adhesive technology was based on very similar processes as that in Africa. In both contexts, early humans deliberately produced some of their adhesives by distillation, from *Podocarpus* conifers in Africa (9) and birch bark in Europe (21, 22). Both also mixed some of their

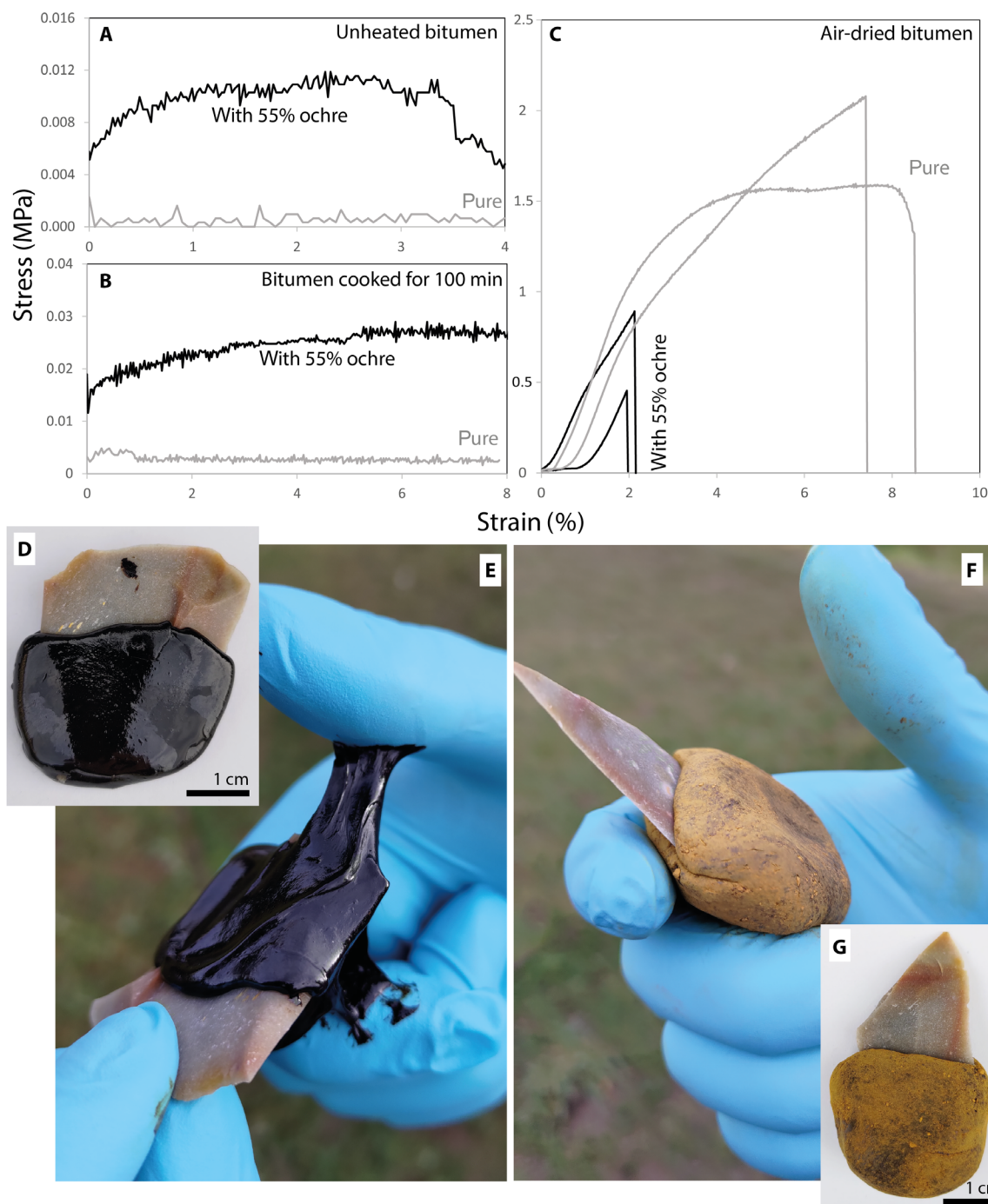


Fig. 4. Experiments investigating the benefit of goethite ochre in bitumen-based adhesives. (A and B) Stress/strain diagrams of uncooked and cooked (100 min) fresh bitumen samples with and without ochre. (C) Stress/strain diagrams of air-dried bitumen with and without ochre. (D and E) Composite tool consisting of a stone tool and a grip made from fresh bitumen. (F and G) Composite tool consisting of a stone tool and a grip made from fresh bitumen and 55 wt % goethite ochre.

glues with ochre to make compound adhesives (14, 67). However, what do these behaviors actually imply in terms of cognition or cultural processes? Pioneering work on adhesive technology by Wadley *et al.* (2, 17) suggests that compound adhesives have different implications than single-component ["simple" (34)] glues. This is because specific recipes must be followed, demanding multitasking and the use of abstraction and recursion (18). Others have approached

the meaning of archaeological artifacts by quantitative interpretations of the steps needed for their production and their process complexity (68). There is a variety of methods for interpreting such stepwise complexity. In the case of adhesive technology, they range from graphical representations of compound adhesive production sequences (69) to computer-aided networks that correlate the steps and materials needed for adhesive making (70). Such approaches

apparently allow comparisons among processes but depend on the scientist's initial choice of steps. Because very different production pathways may produce the same finished products and because the actions of Pleistocene hominins do not fossilize, there is no mechanism to verify whether the chosen steps really took place. Therefore, we cannot decide whether the Le Moustier compound adhesives document more or less complex technology than other adhesives, such as birch tar. An alternative approach (12) proposes that adhesive making may have implications for human evolution if it relied on cultural transmission, was more difficult to execute, and/or was more costly than other techniques. In this sense, the Le Moustier adhesives document the willingness to invest more time and raw materials than other contemporaneous tool-making behaviors—i.e., their cost was likely higher. This is so because bitumen, flint, and pure goethite ochre do not occur at the same outcrops but must be gathered and transported to produce the composite tool. The Aquitaine basin, where the site of Le Moustier lies, contains abundant bitumen sources (71) and extensive flint outcrops (72), and goethite ochre is naturally available (73). However, the precise origin of the materials used for the Le Moustier multicomponent tools is not likely within close range of each other. The most prominent outcrop of pure goethite, the Grès de Thiviers (73), lies in a relatively close range to Le Moustier about 50 km to its north, but the closest Aquitaine oil fields, where bitumen outcrops can be found, lie more than 200 km to its south (74), roughly at equidistance to the petroleum sources of the Massif Central in central France. Thus, gathering the raw materials used for the Le Moustier compound adhesives was substantially more time- and effort-intensive than, for example, the collection of birch bark for tar making. In other words, compound adhesives were more expensive to their makers than adhesives like birch tar. The cost associated with these compound adhesives was higher than many other objects made in the European Middle Paleolithic, and their use likely implied cognitive processes, such as forward-planning and imagination (18), which were not required for many other Middle Paleolithic tools. Unfortunately, the context of Le Moustier allows reasonable doubts as to whether the authors of these pieces were Neanderthals. This is so because there are no radiometric dates available for our assemblage and direct dating of the lower shelter at Le Moustier [56 to 40 ka (75)], which is adjacent to the upper shelter from where our adhesives were excavated, situates the site at the end of the Neanderthal presence in Europe. At this time (76), and even before (33, 77), *H. sapiens* incursions into southern Europe make it possible that Neanderthals and *H. sapiens* were present at the same sites.

So, what are the implications if these ochre-based compound adhesives were made by Middle Paleolithic *H. sapiens*? It is known from the African MSA record that very similar ochre-based compound adhesives were used (67). If anatomically modern humans brought this knowledge with them during their Out-of-Africa migration, its presence at Le Moustier would document a remarkably long technological continuity. In this case, the knowledge and skill to produce moldable materials with specific properties that are not available in single-component adhesives might have provided an advantage over the adhesive technology present in Europe at this time. Such a scenario would gain support from a recent proposal that contemporaneous *H. sapiens* at Mandrin Cave used a microlithic technology that relied on hafting (78). If future studies would find an association of the Le Moustier artifacts with early *H. sapiens* incursions [also see (79)], they might become an important argument

in the debate surrounding the latter's technological superiority over more archaic humans [see, for example, (80–82)].

Alternatively, what are the implications if these compound adhesives were made by Neanderthals? It has recently been shown that Neanderthal birch tar making relied on the cumulative cultural transmission of techniques (32). This was proposed to be one of the core criteria for understanding ancient adhesives as proxies of evolutionary processes (12). Another proposed core criterion is the willingness to invest elevated costs in the production of tools (12). If Neanderthals were the authors, the Le Moustier artifacts would constitute the second indication that their adhesive technology can be regarded as highly relevant for studying their cultural evolution. Another implication would result from ochre being used instead of other fillers. It is known that ochre has properties that lend it to its use in both utilitarian and symbolic contexts (17, 83, 84), and some authors argue that its use is never entirely utilitarian (85, 86). Following that argument, if Neanderthals made the Le Moustier compound adhesives and they used the ochre for both its utilitarian and symbolic purposes, the evidence presented here would add weight to arguments in favor of the Neanderthal capacity for symbolic behavior (87–89). In any case, the use of ochre as filler would appear to be present in African *H. sapiens* and Neanderthals, both showing almost identical adhesive technologies in all aspects (the only difference being the types of raw materials used).

This obviously gives rise to the question: Is it possible that Neanderthals made the compound adhesives but that these similarities are the result of acculturation? This might be a likely scenario if contemporaneous *H. sapiens* in this region were known to produce compound adhesives from bitumen and ochre. There are adhesive finds associated with the late Aurignacian of the greater region (23, 24), but these are of unknown composition and either are single-component (23) or contain no ochre [a quartz IR spectrum is presented as evidence of ochre in (24)]. They also postdate the Le Moustier assemblage. Thus, the still fragmentary adhesive find record does not allow us to decide whether acculturation is a likely scenario or not. The roughly contemporaneous find of one artifact containing an adhesive admixture based on coniferous resin and beeswax at Fossellone in Italy (20) most likely cannot shed light on this question either (the authorship of the Fossellone and Sant'Agostino adhesives may also be regarded as ambiguous; no ochre was reported; only 1 of 15 artifacts was proposed to be a compound adhesive; this was based on long-chain fatty acids and alcohols, and the typical wax esters were not reported).

In conclusion, the Berlin Le Moustier artifacts are the oldest compound adhesives that have been found in a European context. We found that high proportions of ochre make bitumen more rigid and prevent it from sticking to the hand. This suggests that the adhesives were used as handles directly attached to stone tools rather than for hafting stone tools to handles. This is in continuity with the behavior known from the European Middle Paleolithic (birch tar handles). Our findings also highlight the importance of the Middle Paleolithic adhesive technology for our understanding of the economy and technology of the foragers of this period. They invested time and effort in making compound adhesives and had the cognitive capacities needed. This technology provided new materials with specific and desirable material properties. This capacity and the willingness to invest in tools that represented an elevated cost documents the complexity of late Middle Paleolithic hominin behavior.

METHODS

IR spectra were recorded from KBr pellets by direct transmission (in a vacuum chamber), using a Bruker VERTEX 80v spectrometer, spectral acquisition between 1800 and 400 cm^{-1} , and a resolution of 2 cm^{-1} . The ~0.3-g pellet contained 1 mg of sample.

CT scans were recorded with a Nikon XT H 320 CT scanner in Tübingen, selecting a resolution of about 11.5 μm (XrayA = 215 mA and ErayV = 215 kV). The reconstructed volumetric data (.vol) were sliced, and the ISO surface of the pieces was generated using the VGSTUDIO MAX version 3.5.1 software.

Lap-shear tests were performed in uniaxial tension and with a speed of 1 mm/min, using a universal testing machine (Instron 4502). All tests were performed at 21°C. Aluminum Laps (100 mm \times 25.4 mm) were mounted in kardanic suspended tensile grips to minimize the bending moment on the samples. Contact zones (12.7 mm \times 25.4 mm, 322.6 mm^2) were abraded with 100 grit sand paper. Tests result in stress/strain diagrams, plotting apparent shear stress τ in megapascal (as obtained from the force applied by the testing machine/bonded area) over strain in percent.

Supplementary Materials

This PDF file includes:

Supplementary Text

Figs. S1 to S12

Tables S1 and S2

References

REFERENCES AND NOTES

1. S. McBrearty, A. S. Brooks, The revolution that wasn't: A new interpretation of the origin of modern human behavior. *J. Hum. Evol.* **39**, 453–563 (2000).
2. L. Wadley, T. Hodgskiss, M. Grant, Implications for complex cognition from the hafting of tools with compound adhesives in the Middle Stone Age, South Africa. *Proc. Natl. Acad. Sci. U.S.A.* **106**, 9590–9594 (2009).
3. L. Backwell, F. d'Errico, L. Wadley, Middle Stone Age bone tools from the Howiesons Poort layers, Sibudu Cave, South Africa. *J. Archaeol. Sci.* **35**, 1566–1580 (2008).
4. T. Minichillo, Raw material use and behavioral modernity: Howiesons Poort lithic foraging strategies. *J. Hum. Evol.* **50**, 359–364 (2006).
5. M. J. L. T. Niekus, P. R. B. Kozowyk, G. H. J. Langejans, D. Ngan-Tillard, H. van Keulen, J. van der Plicht, K. M. Cohen, W. van Wingerden, B. van Os, B. I. Smit, L. W. S. W. Amkreutz, L. Johansen, A. Verbaas, G. L. Dusseldorp, Middle Paleolithic complex technology and a Neandertal tar-backed tool from the Dutch North Sea. *Proc. Natl. Acad. Sci. U.S.A.* **116**, 22081–22087 (2019).
6. K. S. Brown, C. W. Marean, A. I. R. Herries, Z. Jacobs, C. Tribolo, D. Braun, D. L. Roberts, M. C. Meyer, J. Bernatchez, Fire as an engineering tool of early modern humans. *Science* **325**, 859–862 (2009).
7. T. Wynn, Hafted spears and the archaeology of mind. *Proc. Natl. Acad. Sci. U.S.A.* **106**, 9544–9545 (2009).
8. V. Rots, P. Van Peer, Early evidence of complexity in lithic economy: Core-axe production, hafting and use at Late Middle Pleistocene site 8-B-11, Sai Island (Sudan). *J. Archaeol. Sci.* **33**, 360–371 (2006).
9. P. Schmidt, T. J. Koch, E. February, Archaeological adhesives made from Podocarpus document innovative potential in the African Middle Stone Age. *Proc. Natl. Acad. Sci. U.S.A.* **119**, e2209592119 (2022).
10. T. Schenck, P. Groom, The aceramic production of *Betula pubescens* (downy birch) bark tar using simple raised structures. A viable Neanderthal technique? *Anthropol. Sci.* **10**, 19–29 (2018).
11. P. R. B. Kozowyk, M. Soressi, D. Pomstra, G. H. J. Langejans, Experimental methods for the Palaeolithic dry distillation of birch bark: Implications for the origin and development of Neandertal adhesive technology. *Sci. Rep.* **7**, 8033 (2017).
12. P. Schmidt, Steak tournedos or beef Wellington: An attempt to understand the meaning of Stone Age transformative techniques. *Humanit. Soc. Sci. Commun.* **8**, 280 (2021).
13. L. Wadley, M. Mohapi, A segment is not a monolith: Evidence from the Howiesons Poort of Sibudu, South Africa. *J. Archaeol. Sci.* **35**, 2594–2605 (2008).
14. M. Lombard, The gripping nature of ochre: The association of ochre with Howiesons Poort adhesives and Later Stone Age mastics from South Africa. *J. Hum. Evol.* **53**, 406–419 (2007).
15. A. Charrié-Duhaut, G. Porraz, C. R. Cartwright, M. Igraja, J. Connan, C. Poggenpoel, P. J. Texier, First molecular identification of a hafting adhesive in the Late Howiesons Poort at Diepkloof Rock Shelter, Western Cape, South Africa. *J. Archaeol. Sci.* **40**, 3506–3518 (2013).
16. A. M. Zipkin, M. Wagner, K. McGrath, A. S. Brooks, P. W. Lucas, An experimental study of hafting adhesives and the implications for compound tool technology. *PLOS ONE* **9**, e112560 (2014).
17. L. Wadley, Putting ochre to the test: Replication studies of adhesives that may have been used for hafting tools in the Middle Stone Age. *J. Hum. Evol.* **49**, 587–601 (2005).
18. L. Wadley, Compound-adhesive manufacture as a behavioral proxy for complex cognition in the Middle Stone Age. *Curr. Anthropol.* **51**, S111–S119 (2010).
19. T. C. Hauck, J. Connan, A. Charrié-Duhaut, J.-M. Le Tensorer, H. A. Sakhel, Molecular evidence of bitumen in the Mousterian lithic assemblage of Hummal (Central Syria). *J. Archaeol. Sci.* **40**, 3252–3262 (2013).
20. I. Degano, S. Soriano, P. Villa, L. Pollarolo, J. J. Lucejko, Z. Jacobs, K. Douka, S. Vitagliano, C. Tozzi, Hafting of Middle Paleolithic tools in Latium (central Italy): New data from Fossellone and Sant'Agostino caves. *PLOS ONE* **14**, e0213473 (2019).
21. J. Grünberg, H. Gratsch, U. Baumer, J. Koller, Untersuchung der mittelpaläolithischen "Harzreste" von Königsau, Ldkr. Aschersleben-Staßfurt. *Jahresschrift für mitteldeutsche Vorgeschichte* **81**, 7–38 (1999).
22. P. P. A. Mazza, F. Martini, B. Sala, M. Magi, M. P. Colombini, G. Giachi, F. Landucci, C. Lemorini, F. Modugno, E. Ribechini, A new Palaeolithic discovery: Tar-hafted stone tools in a European Mid-Pleistocene bone-bearing bed. *J. Archaeol. Sci.* **33**, 1310–1318 (2006).
23. R. Dinnis, A. Pawlik, C. Gaillard, Bladelet cores as weapon tips? Hafting residue identification and micro-wear analysis of three carinated burins from the late Aurignacian of Les Vachons, France. *J. Archaeol. Sci.* **36**, 1922–1934 (2009).
24. M. Bradtmöller, A. Sarmiento, U. Perales, M. C. Zuluaga, Investigation of Upper Palaeolithic adhesive residues from Cueva Morin, Northern Spain. *J. Archaeol. Sci. Rep.* **7**, 1–13 (2016).
25. F. Javier Muñoz, V. Rubio, C. Gutiérrez, A. Hernanz, M. Menéndez, THE POINTS FROM EL BUXU CAVE (ASTURIAS, SPAIN): First evidence of adhesive as hafting material in the Solutrean. *J. Archaeol. Sci. Rep.* **49**, 103901 (2023).
26. M. Cărciumaru, R.-M. Ion, E.-C. Nițu, R. Ștefănescu, New evidence of adhesive as hafting material on Middle and Upper Palaeolithic artefacts from Gura Cheii-Râșnov Cave (Romania). *J. Archaeol. Sci.* **39**, 1942–1950 (2012).
27. P. R. B. Kozowyk, G. H. J. Langejans, G. L. Dusseldorp, M. J. L. T. Niekus, Reply to Schmidt et al.: Interpretation of Paleolithic adhesive production: Combining experimental and paleoenvironmental information. *Proc. Natl. Acad. Sci. U.S.A.* **117**, 4458 (2020).
28. P. Schmidt, M. Rageot, M. Blessing, C. Tennie, The Zandmotor data do not resolve the question whether Middle Paleolithic birch tar making was complex or not. *Proc. Natl. Acad. Sci. U.S.A.* **117**, 4456–4457 (2020).
29. P. Schmidt, T. J. Koch, C. Tennie, Reply to Paul R.B. Kozowyk: Interpreting the complexity of archaeological adhesives may lead to misconceptions about early humans. *Proc. Natl. Acad. Sci. U.S.A.* **120**, e2300325120 (2023).
30. P. R. B. Kozowyk, Archaeological Podocarpus tar supports the cognitive complexity of Neanderthals. *Proc. Natl. Acad. Sci. U.S.A.* **120**, e2221676120 (2023).
31. M. Tomasello, *The Cultural Origins of Human Cognition* (Harvard Univ. Press, 2009).
32. P. Schmidt, T. J. Koch, M. A. Blessing, F. A. Karakostis, K. Harvati, V. Dresely, A. Charrié-Duhaut, Production method of the Königsau birch tar documents cumulative culture in Neanderthals. *Archaeol. Anthropol. Sci.* **15**, 84 (2023).
33. L. Slimak, C. Zanolli, T. Higham, M. Frouin, J.-L. Schwenninger, L. J. Arnold, M. Demuro, K. Douka, N. Mercier, G. Guérin, H. Valladas, P. Yvorra, Y. Giraud, A. Seguin-Orlando, L. Orlando, J. E. Lewis, X. Muth, H. Camus, S. Vandevelde, M. Buckley, C. Mallol, C. Stringer, L. Metz, Modern human incursion into Neanderthal territories 54,000 years ago at Mandrin, France. *Sci. Adv.* **8**, eabj9496 (2022).
34. L. Wadley, G. Trower, L. Backwell, F. d'Errico, Traditional glue, adhesive and poison used for composite weapons by Ju/'hoan San in Nyae Nyae, Namibia. Implications for the evolution of hunting equipment in prehistory. *PLOS ONE* **10**, e0140269 (2015).
35. F. Bordes, *Typologie du Paléolithique Ancien et Moyen* (Delmas, 1961).
36. S. Beyries, in *La Main et l'outil. Manches et Emmanchements Préhistoriques, Travaux de La Maison de l'Orient*, D. Stordeur, Ed. (MOM Éditions, 1987), pp. 55–62.
37. V. Rots, Towards an understanding of hafting: The macro- and microscopic evidence. *Antiquity* **77**, 805–815 (2003).
38. V. Rots, *Prehension and Hafting Traces on Flint Tools A Methodology* (Leuven Univ. Press, 2010).
39. I. Levi Sala, Use wear and post-depositional surface modification: A word of caution. *J. Archaeol. Sci.* **13**, 229–244 (1986).
40. M. Blanchard, E. Balan, P. Giura, K. Béneut, H. Yi, G. Morin, C. Pinilla, M. Lazzeri, A. Floris, Infrared spectroscopic properties of goethite: Anharmonic broadening, long-range electrostatic effects and Al substitution. *Phys. Chem. Miner.* **41**, 289–302 (2014).

41. G. N. Kustova, E. B. Burgina, V. A. Sadykov, S. G. Poryvaev, Vibrational spectroscopic investigation of the goethite thermal decomposition products. *Phys. Chem. Miner.* **18**, 379–382 (1992).
42. S. Weigel, D. Stephan, The prediction of bitumen properties based on FTIR and multivariate analysis methods. *Fuel* **208**, 655–661 (2017).
43. B. Hofko, L. Porot, A. Falchetto Cannone, L. Poulikakos, L. Huber, X. Lu, K. Mollenhauer, H. Grothe, FTIR spectral analysis of bituminous binders: Reproducibility and impact of ageing temperature. *Mater. Struct.* **51**, 45 (2018).
44. G. Vázquez-Bautista, F. Chalé-Lara, M. Zapata Torres, M. Meléndez Lira, E. Hernández Rodríguez, E. Valaguez Velázquez, Is sulfur the responsible for color of yellow Chiapas Amber? *Superf. y Vacío* **34**, 211201 (2021).
45. H. D. Ruan, R. L. Frost, J. T. Klopogge, L. Duong, Infrared spectroscopy of goethite dehydroxylation: III. FT-IR microscopy of in situ study of the thermal transformation of goethite to hematite. *Spectrochim. Acta A Mol. Biomol. Spectrosc.* **58**, 967–981 (2002).
46. A. Olori, P. Di Pietro, A. Campopiano, Preparation of ultrapure KBr pellet: New method for FTIR quantitative analysis. *Int. J. Sci. Res.* **2**, 1015–1020 (2021).
47. V. Beltran, N. Salvadó, S. Butí, T. Pradell, Ageing of resin from Pinus species assessed by infrared spectroscopy. *Anal. Bioanal. Chem.* **408**, 4073–4082 (2016).
48. J.-B. Chen, Q. Zhou, S.-Q. Sun, Direct chemical characterization of natural wood resins by temperature-resolved and space-resolved Fourier transform infrared spectroscopy. *J. Mol. Struct.* **1115**, 55–62 (2016).
49. A. Dwivedi, A. K. Pandey, K. Raj, N. Misra, Comparative study of vibrational spectra of two bioactive natural products Lupeol and Lupenone using MM/QM method. *Int. J.* **27**, 486304 (2012).
50. R. Fang, R. Littke, L. Zieger, A. Baniasad, M. Li, J. Schwarzbauer, Changes of composition and content of tricyclic terpane, hopane, sterane, and aromatic biomarkers throughout the oil window: A detailed study on maturity parameters of Lower Toarcian Posidonia Shale of the Hils Syncline, NW Germany. *Org. Geochem.* **138**, 103928 (2019).
51. A. Charrié-Duhaut, S. Lemoine, P. Adam, J. Connan, P. Albrecht, Abiotic oxidation of petroleum bitumens under natural conditions. *Org. Geochem.* **31**, 977–1003 (2000).
52. E. M. Aveling, C. I. Heron, Identification of birch bark tar at the Mesolithic site of Star Carr. *Anc. Biomol.* **2**, 69–80 (1998).
53. P. Dietemann, K. V. Miller, C. Höpker, U. Baumer, On the use and differentiation of resins from Pinaceae species in European artworks based on written sources, reconstructions and analysis. *Stud. Conserv.* **64**, S62–S73 (2019).
54. R. T. Mull, Mass estimates by computed tomography: Physical density from CT numbers. *AJR Am. J. Roentgenol.* **143**, 1101–1104 (1984).
55. T. Razi, M. Niknami, F. Alavi Ghazani, Relationship between Hounsfield unit in CT scan and gray scale in CBCT. *J. Dent. Res. Dent. Clin. Dent. Prospects* **8**, 107–110 (2014).
56. E. Yalcin, A. Munir Ozdemir, B. Vural Kok, M. Yilmaz, B. Yilmaz, Influence of pandemic waste face mask on rheological, physical and chemical properties of bitumen. *Construct. Build Mater.* **337**, 127576 (2022).
57. C. Palache, H. Berman, C. Frondel, in *The System of Mineralogy of James Dwight Dana and Edward Salisbury Dana Yale University 1837–1892, Volume I* (John Wiley and Sons, 1944).
58. L. Tydgadt, V. Rots, Stick to it! Mechanical performance tests to explore the resilience of prehistoric glues in hafting. *Archaeometry* **64**, 1252–1269 (2022).
59. P. R. B. Kozowyk, G. H. J. Langejans, J. A. Poulis, Lap shear and impact testing of ochre and beeswax in experimental Middle Stone Age compound adhesives. *PLOS ONE* **11**, e0150436 (2016).
60. P. R. B. Kozowyk, J. A. Poulis, A new experimental methodology for assessing adhesive properties shows that Neandertals used the most suitable material available. *J. Hum. Evol.* **137**, 102664 (2019).
61. J. S. Tomblin, C. C. Yang, P. Harter, Investigation of thick bondline adhesive joints (Report: DOT/FAA/AR-01/33, Department of Transportation, 106, 2001).
62. P. Schmidt, T. J. Koch, C. Berthold, F. Lauxmann, K. G. Nickel, The evolution of strength, elasticity and rupture behaviour of birch tar made with 'double-pot' techniques during tar cooking. *Archaeometry* **65**, 409–422 (2023).
63. P. Schmidt, M. A. Blessing, T. J. Koch, K. G. Nickel, On the performance of birch tar made with different techniques. *Herit. Sci.* **9**, 140 (2021).
64. P. R. B. Kozowyk, J. A. Poulis, G. H. J. Langejans, Laboratory strength testing of pine wood and birch bark adhesives: A first study of the material properties of pitch. *J. Archaeol. Sci. Rep.* **13**, 49–59 (2017).
65. F. A. Karakostis, G. Hotz, V. Tourloukis, K. Harvati, Evidence for precision grasping in Neandertal daily activities. *Sci. Adv.* **4**, eaat2369 (2018).
66. W. A. Niewoehner, Behavioral inferences from the Skhul/Qafzeh early modern human hand remains. *Proc. Natl. Acad. Sci. U.S.A.* **98**, 2979–2984 (2001).
67. L. Wadley, Compound-adhesive manufacture as a behavioral proxy for complex cognition in the Middle Stone Age. *Curr. Anthropol.* **51**, 111–119 (2010).
68. C. Perreault, P. J. Brantingham, S. L. Kuhn, S. Wurz, X. Gao, Measuring the complexity of lithic technology. *Curr. Anthropol.* **54**, S397–S406 (2013).
69. M. Lombard, M. N. Haidle, Thinking a bow-and-arrow set: Cognitive implications of Middle Stone Age bow and stone-tipped arrow technology. *Camb. Archaeol. J.* **22**, 237–264 (2012).
70. S. Fajardo, J. Kleijn, F. W. Takes, G. H. J. Langejans, Modelling and measuring complexity of traditional and ancient technologies using Petri nets. *PLOS ONE* **17**, e0278310 (2022).
71. S. P. Pratt, Geological position of the bitumen used in asphalt pavements. *J. Geol. Soc.* **2**, 80–81 (1846).
72. A. Morala, *Les silicifications des bassins versants de la Dordogne et de la Vézère: Evaluation des ressources lithiques et implications archéologiques* (Paleo, 2017), 110 pp.
73. A. Sempio, A. Gualtieri, Mineralogy of the Grès de Thiviers (northern Aquitaine, France). *Per. Mineral.* **71**, 65–84 (2002).
74. J.-J. Biseau, A. Le Marrec, M. Le Vot, J.-M. Masset, The Aquitaine Basin. *Pet. Geosci.* **12**, 247–273 (2006).
75. H. Valladas, J. M. Geneste, J. L. Joron, J. P. Chadelles, Thermoluminescence dating of Le Moustier (Dordogne, France). *Nature* **322**, 452–454 (1986).
76. J.-J. Hublin, N. Sirakov, V. Aldeias, S. Bailey, E. Bard, V. Delvigne, E. Endarova, Y. Fagault, H. Fewlass, M. Hajdinjak, B. Kromer, I. Krumov, J. Marreiros, N. L. Martisius, L. Paskulin, V. Sinet-Mathiot, M. Meyer, S. Pääbo, V. Popov, Z. Rezek, S. Sirakova, M. M. Skinner, G. M. Smith, R. Spasov, S. Talamo, T. Tuna, L. Wacker, F. Welker, A. Wilcke, N. Zahariev, S. P. McPherron, T. Tsanova, Initial Upper Palaeolithic *Homo sapiens* from Bacho Kiro Cave, Bulgaria. *Nature* **581**, 299–302 (2020).
77. K. Harvati, C. Röding, A. M. Bosman, F. A. Karakostis, R. Grün, C. Stringer, P. Karkanas, N. C. Thompson, V. Koutoulidis, L. A. Mouloupoulos, V. G. Gorgoulis, M. Kouloukoussa, Apidima Cave fossils provide earliest evidence of *Homo sapiens* in Eurasia. *Nature* **571**, 500–504 (2019).
78. L. Metz, J. E. Lewis, L. Slimak, Bow-and-arrow, technology of the first modern humans in Europe 54,000 years ago at Mandrin, France. *Sci. Adv.* **9**, eadd4675 (2023).
79. A. Picin, A. Moroni, S. Benazzi, in *Updating Neanderthals*, F. Romagnoli, F. Rivals, S. Benazzi, Eds. (Academic Press, 2022), pp. 321–347.
80. P. Villa, W. Roebroeks, Neandertal demise: An archaeological analysis of the modern human superiority complex. *PLOS ONE* **9**, e96424 (2014).
81. J. Speth, News flash: Negative evidence convicts Neanderthals of gross mental incompetence. *World Archaeol.* **36**, 519–526 (2004).
82. O. Bar-Yosef, in *Dynamics of Learning in Neanderthals and Modern Humans Volume 1: Cultural Perspectives*, T. Akazawa, Y. Nishiaki, K. Aokie, Eds. (Springer, 2013), pp. 7–20.
83. A. G. Püntener, S. Moss, Otzi, the iceman and his leather clothes. *Chimia* **64**, 315–320 (2010).
84. E. C. Velliki, P. Schmidt, L. Bellot-Gurlet, S. Wolf, N. J. Conard, Early anthropogenic use of hematite on Aurignacian ivory personal ornaments from Hohle Fels and Vogelherd caves, Germany. *J. Hum. Evol.* **150**, 102900 (2021).
85. I. Watts, in *The Cradle of Language*, R. Botha, C. Knight, Eds. (Oxford Univ. Press, 2009), pp. 62–97.
86. R. F. Rifkin, Assessing the efficacy of red ochre as a prehistoric hide tanning ingredient. *Archaeology* **9**, 131–158 (2011).
87. A. Pitarch Martí, J. Zilhão, F. d'Errico, P. Cantalejo-Duarte, S. Domínguez-Bella, J. M. Fullola, G. C. Weniger, J. Ramos-Muñoz, The symbolic role of the underground world among Middle Paleolithic Neanderthals. *Proc. Natl. Acad. Sci. U.S.A.* **118**, e2021495118 (2021).
88. D. Leder, R. Hermann, M. Hüls, G. Russo, P. Hoelzmann, R. Nielbock, U. Böhner, A. Lehmann, M. Meier, A. Schwalb, A. Tröllner-Reimer, T. Koddenberg, T. Terberger, A 51,000-year-old engraved bone reveals Neanderthals' capacity for symbolic behaviour. *Nat. Ecol. Evol.* **5**, 1273–1282 (2021).
89. M. C. Langley, C. Clarkson, S. Ulm, Behavioural complexity in Eurasian Neandertal populations: A chronological examination of the archaeological evidence. *Camb. Archaeol. J.* **18**, 289–307 (2008).
90. E. Lartet, H. Christy, Sur des figures d'animaux gravées ou sculptées et autres produits d'art et d'industrie rapportables aux temps primordiaux de la période humaine. *Rev. Archéol.* **9**, 233–267 (1864).
91. E. Lartet, H. Christy, T. R. Jones, *Reliquae Aquitanicae: Being Contributions to the Archaeology and Palaeontology of Périgord and Adjoining Provinces of Southern France* (Williams & Norgate, 1875).
92. G. de Mortillet, Essai d'une classification des cavernes et des stations sous abri, fondée sur les produits de l'industrie humaine. *Matériaux pour l'histoire primitive et naturelle de l'homme* **4**, 172–179 (1869).
93. G. de Mortillet, Notice sur l'origine du langage. *Congrès international d'anthropologie et d'archéologie préhistoriques* **4**, 286–287 (1869).
94. M. Boursin, Une fouille au Moustier (Dordogne). *L'homme préhistorique* **7**, 193–204 (1905).
95. M. Boursin, L'industrie moustérienne au Moustier. *Congrès international d'anthropologie et d'archéologie préhistoriques* **13**, 287–322 (1906).
96. O. Hauser, Fouilles scientifiques à la Micoque, à Laugerie-Basse et au Moustier. *L'homme préhistorique* **6**, 40–48 (1908).

97. O. Hauser, *Le Périgord préhistorique, guide pour les excursions dans les Vallées de la Vézère et de la Dordogne et pour l'étude de leurs stations préhistoriques* (Réjou, 1911).
98. D. Peyrony, Après une grande crue préhistorique de la Vézère. *Revue de géographie commerciale* **4**, 123–127 (1914).
99. D. Peyrony, Le Moustier, ses gisements, ses industries, ses couches géologique. *Revue anthropologie* **40**, 48–76 (1930).
100. A. Hoffmann, Le Moustier und Combe Capelle. *Die altsteinzeitlichen Funde des Schweizer Archäologen Otto Hauser. Museum für Vor- und Frühgeschichte Bestandskatalog, Band 9* (Staatliche Museen zu Berlin – Preußischer Kulturbesitz, 2003).
101. R. Busch, Dr. Alfred Tode Braunschweiger Landesarchäologe i. R. verstorben. *Nachrichten aus Niedersachsens Urgeschichte* **65**, 235–236 (1996).
102. R. Drößler, *Flucht aus dem Paradies. Leben, Ausgrabungen und Entdeckungen Otto Hausers* (Mitteldeutscher Verlag, 1988).
103. R. Drössler, S. Drössler, M. Freyberg, The Swiss archaeologist Otto Hauser. His skeleton findings and assumptions concerning the Evolution of man. *Anthropol. Anz.* **64**, 97–123 (2006).
104. A. Debénath, H. L. Dibble, *Handbook of Paleolithic Typology Lower and Middle Paleolithic of Europe* (University of Pennsylvania Press, 1993).
105. V. Rots, Prehensile wear on flint tools. *Lithic Technol.* **29**, 7–32 (2004).
106. M. E. Mansur, Microwear analysis of natural and use striations: New clues to the mechanisms of striation formation. *Studia Praehistorica Belgica Leuven* **2**, 213–233 (1982).
107. T. A. Del Bene, in *Lithic Use-Wear Analysis*, B. Hayden, Ed. (Academic Press, 1979), pp. 167–177.
108. L. H. Keeley, *Experimental Determination of Stone Tool Uses: A Microwear Analysis* (University of Chicago Press, 1980).
109. G. H. Odell, F. Odell-Vereecken, Verifying the relationships of lithic use wear assessments by 'blind tests': The low-power approach. *J. Field Archaeol.* **7**, 87–120 (1980).

Acknowledgments: Part of the research equipment (Instron, IR Spectrometer) was provided by K. G. Nickel and C. Berthold at Tübingen University's Department of Geosciences, Applied Mineralogy, and the Competence Center Archaeometry Baden-Wuerttemberg (CCA-BW). We acknowledge A. Flicker (Univ. Tübingen) for assistance with the IR analyses and the team of Senckenberg Centre for Human Evolution and Palaeoenvironment at the University of Tübingen (SHEP), namely, G. Ferreira for assistance with the microCT scan, T. Miranda for assistance with the REM, and S. Wolf for support at SHEP. We also thank M. Nykamp (Freie Universität Berlin, Physische Geographie) for providing the initial XRF analysis. Further, we thank E. Leize-Wagner (head of the Laboratory of Mass Spectrometry of Interactions and Systems, Strasbourg, France), J.-L. Schmitt (head of the analysis platform from ISIS), and W. Kazöne (Laboratory of chemical catalysis) for providing access to the GC-MS system (Strasbourg, France). D. Greinert, Staatliche Museen zu Berlin, contributed the technical drawings. We also thank the team of the Museum für Vor- und Frühgeschichte Berlin, namely, P. Schmidt-Reimann and Y. de Bordes, for helping us with handling the artifacts. **Funding:** This work was supported by Deutsche Forschungsgemeinschaft (DFG) grant SCHM 3275/3–1 (to P.S.). We acknowledge support by Open Access Publishing Fund of University of Tübingen. **Author contributions:** Conceptualization: P.S. and E.D. Methodology: P.S., R.I., and A.C.-D. Investigation/formal analysis: P.S., R.I., A.C.-D., G.M., A.N., and E.D. Visualization: P.S., A.C.-D., and E.D. Funding acquisition: P.S. Project administration: P.S. and E.D. Supervision: P.S. and E.D. Writing—original draft: P.S. Writing—review and editing: P.S., R.I., A.C.-D., G.M., A.N., and E.D. **Competing interests:** The authors declare that they have no competing interests. **Data and materials availability:** All data needed to evaluate the conclusions in the paper are present in the paper and/or the Supplementary Materials.

Submitted 27 September 2023

Accepted 18 January 2024

Published 21 February 2024

10.1126/sciadv.adl0822