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No Solid Foundation for the Use of Ochre-based Compound Adhesives at Le Moustier

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Abstract

Adhesives are commonly sought-after residues due to their current use as a proxy for early human cognitive traits. Identifying adhesives is a complex task due to the preservation bias associated with the archaeological record and the organic nature of the residues. Although important information concerning prehistoric behaviour is being inferred from glue identification and use, it is rarely identified through molecular and elemental compositions. In a recent study by Schmidt et al. (*Ochre-based compound adhesives at the Mousterian type-site document complex cognition and high investment, Science Advances, 2024, Vol. 10, Issue 8*), they attempted to use such procedures to understand the nature of a black residue found on four out of five artifacts excavated at the beginning of the twentieth century from the upper rock shelter of Le Moustier. They claim that they succeeded in identifying the oldest compound adhesives found in a European context. The presence of compound adhesive in the Middle Palaeolithic has several implications for technological and cognitive human evolution. In this paper, we evaluate the reliability of the argument used by the authors to defend the presence of compound adhesives on the studied artefacts. Upon this evaluation, we encountered several uncertainties that put into question the adhesive identification that the authors claimed. These uncertainties should be addressed before the result from this study can be used for further inferences about past human behaviour.

Keywords Adhesive; Cognition; Middle Palaeolithic · FTIR · Residue analysis

Introduction

In recent years, there has been a notable interest in the identification of residues, driven by the pivotal role adhesive identification plays in discussions surrounding cognitive development throughout human evolution. However, this interest does not solely revolve around recognising adhesives but extends to identifying their composition and production processes in depth. This is because the ability to create and use adhesives has been used to infer cognitive capabilities and technological innovation among early human populations, including Neanderthals. Compound adhesive in particular has received a lot of attention. The identification of compound adhesives, often involving ochre, has led to investigations into the intricate recipes needed for their creation, linking such skills to theories of cognition such as working memory and time investment (Gibson et al., 2004; Lombard & Wadley, 2007; Lombard, 2006, 2007; Villa et al., 2015; Wadley et al., 2004, 2009; Wadley, 2005, 2010; Wragg Sykes, 2015). Researchers have postulated that residues found on South African artefacts provide evidence for the intentional addition of ochre to resin to improve its adhesive characteristics (Wadley et al., 2004). In Europe, the identification of birch tar has received a similar amount of attention as the production process of birch tar has been linked to cognitive aspects of Neanderthals (Groom et al., 2015; Koch & Schmidt, 2022; Koller et al., 2001; Kozowyk et al., 2017, 2023; Pawlik & Thissen, 2011; Schenck & Groom, 2018; Schmidt et al., 2019, 2023). There is some nuance to these studies as certain proposed production techniques are seen as more complex than others (Koch & Schmidt, 2022; Schmidt et al., 2019, 2023).

In a recent article, Schmidt and colleagues (2024) report the presence of a compound adhesive, comprised of ochre

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and bitumen, interpreted as a handle grip at the type site of Le Moustier. The study presents an analysis of five artefacts reported as deriving from the upper rock shelter of Le Moustier. The findings from this analysis are used to advocate for significant investment in time and effort to produce this compound glue, suggesting cognitive complexity among its makers. We contend that the evidence presented is not sufficiently convincing and that several issues arise regarding contextual information, material identification, analysis and interpretation as hafting-related residues. We argue that while the study presents intriguing findings, its reliability is compromised by methodological limitations and interpretational uncertainties.

Research Context

Given the key role of adhesives in current debates, the identification of adhesives on stone tools has become a crucial endeavour, but it is complicated by the rarity and limited quantity of such residues in Palaeolithic contexts. Many residues have been hypothesised to represent adhesives on visual grounds only, through optical observation (Ambrose, 1998; Clarkson et al., 2015; Fullagar & David, 1997; Gibson et al., 2004; Hamm et al., 2016; Lombard, 2005, 2007; Rots et al., 2011; Thackeray, 2000; Wadley et al., 2004, 2009; Wendt, 1976), with only a minority having been identified through chemical procedures (Boëda et al., 1996, 2008; Koller et al., 2001; Mazza et al., 2006; Villa et al., 2012, 2015; Charrié-Duhaut et al., 2013; Hauck et al., 2013; Niekus et al., 2019, see Table 1) or through procedures that incorporated initial steps towards chemical identification (Bradtmöller et al., 2016; Cârciumaru et al., 2012; Monnier et al., 2013; Pawlik & Thissen, 2011; Rots et al., 2017; Yaroshevich et al., 2013). New studies that involve the chemical analysis of possible remains of adhesives are therefore welcome to increase insight and enlarge the available dataset. However, while the chemical analysis conducted by Schmidt et al. (2024) is a valuable contribution to such a perspective, it is essential to have contextual data for this information to be truly meaningful.

In contrast to previous work that postulates the use of compound adhesives on optical observations alone leaving room for ambiguity, the recent study by Schmidt et al. (2024) tried to integrate chemical analysis to examine the residues on the Le Moustier artefacts. The possibility that this would permit the identification of compound adhesives is an exciting prospect for studying human behaviour. However, we identified several problems within their study which cast significant doubts on their claim that compound adhesives were identified on the artefacts labelled as being found at Le Moustier. Their interpretation essentially relies on the following arguments: residue location, wear traces and the identification of goethite and bitumen. We will demonstrate that each of these arguments is problematic, in addition to the contextual issues with regard to the finds themselves.

Areas That Present Cause for Scepticism

Site Context and Excavation Protocols

Although the Middle Palaeolithic site of Le Moustier has been studied in detail throughout the years, the artefacts in question were not recovered through excavation with modern standards. The isolated artefacts studied here were collected by Otto Hauser when he partially emptied the cave around 1907, and no contextual information is associated with the finds in the museum (also no excavation report). The exact origin of these pieces and their curation history (100 years!) is thus completely unknown. Any claims with regard to their age or link to specific populations are thus unwarranted. It is also known that when excavation does not follow secure and careful protocols, artefacts may be damaged through contact with excavation equipment, subsequent cleaning and handling (Tomasso et al. 2021). Furthermore, essential contextual information is missing to adequately evaluate the residues and post-depositional effects, which is a minimal requirement for present-day residue studies (see also Rots

Table 1 Adhesive substances that have been identified through gas chromatography / mass spectrometry (GC/MS) for the Pleistocene

Site	Country	Chronology	Identified substances	Reference
Inden-Altdorf	Germany	MIS 5	Birch tar	Mazza et al. (2006)
Umm el Tell	Syria	MIS (3-)4	Bitumen	Boëda et al., (1996, 2008)
Königsaue	Germany	MIS 3	Birch tar	Koller et al. (2001)
Zandmotor	The Netherlands	MIS 3	Birch tar	Niekus et al. (2019)
Hummal	Syria	MIS 3	Bitumen	Hauck et al. (2013)
Border Cave	South Africa	MIS 3	Resin, beeswax and egg	d'Errico et al. (2012)
Border Cave	South Africa	MIS 3	Podocarpus pitch	Villa et al. (2012)
Diepkloof Rock Shelter	South Africa	MIS 3	Podocarpus resin	Charrié-Duhaut et al. (2013)

et al., 2016). Moreover, the long curation under unknown conditions creates a long list of possible sources of contamination and destructive cleaning protocols or treatment with chemicals cannot be excluded either. An essential condition for studying prehistoric residues, being the ability to eliminate background noise from modern contaminants and soil, is therefore not fulfilled and without specific information on the soil or storage conditions, it becomes challenging to identify which contaminants are present.

Available information on the context of the lower cave of Le Moustier from recent excavations shows that post-depositional processes had a severe effect on the preservation state of the stone tool surfaces, resulting in strongly developed edge and ridge rounding, edge scarring and surface gloss. This study of the context of Le Moustier (Texier et al., 2020) has shown that the accumulation of sediment in the lower part of Le Moustier is a result of periodic flooding, surface runoff and limited rockfall events, resulting in 70% of the studied pieces presenting edge damage. These phenomena by themselves do not explain the 69% (1767/2559) of pieces affected by edge damage. The authors however favour regular trampling to explain this high amount of edge damage observed on the pieces (Thomas et. al 2019). We do not assume that the situation of the lower cave and the upper cave is similar in terms of post-depositional processes. However, given that there are no data available for the upper terrace, there are also no grounds to assume that post-depositional effects or trampling would be absent there. Such surface alterations might override the initial use-wear traces or even be mistaken for functional traces. Also, the impact of these processes on residue preservation and the deposition of taphonomic residues cannot be underrated (Fig. 1). This is why functional analysts treat such site contexts with utmost care.

Post-depositional alteration is neglected in the Schmidt et al. study. This is problematic and only adds to the insecure context of the finds. The effect of post-depositional alterations has been amply described in the literature (e.g. Kamminga, 1979; Levi-Sala, 1986; Plisson and Mauger, 1988; Galland et al., 2019; Burroni et al., 2002; Bustos-Pérez & Ollé, 2024). In our experience, Middle Palaeolithic artefacts in cave contexts are extremely rarely preserved in pristine condition (Fig. 2) and possible alteration needs to be critically evaluated for each stratigraphic level instead of just being ruled out as is done here. Here, the only argument to rule out a post-depositional origin is “the restricted” location of the traces on the stone tools. Restriction of polished zones does not rule out post-deposition processes; in fact, these processes can lead to both polish formation and altered polishes resulting in misleading interpretations (e.g. Levi-Sala, 1986; Michel et al., 2019). Based on what the authors provided it is difficult to exclude alterations of taphonomic or other origin for the traces described (also at least one of the artefacts

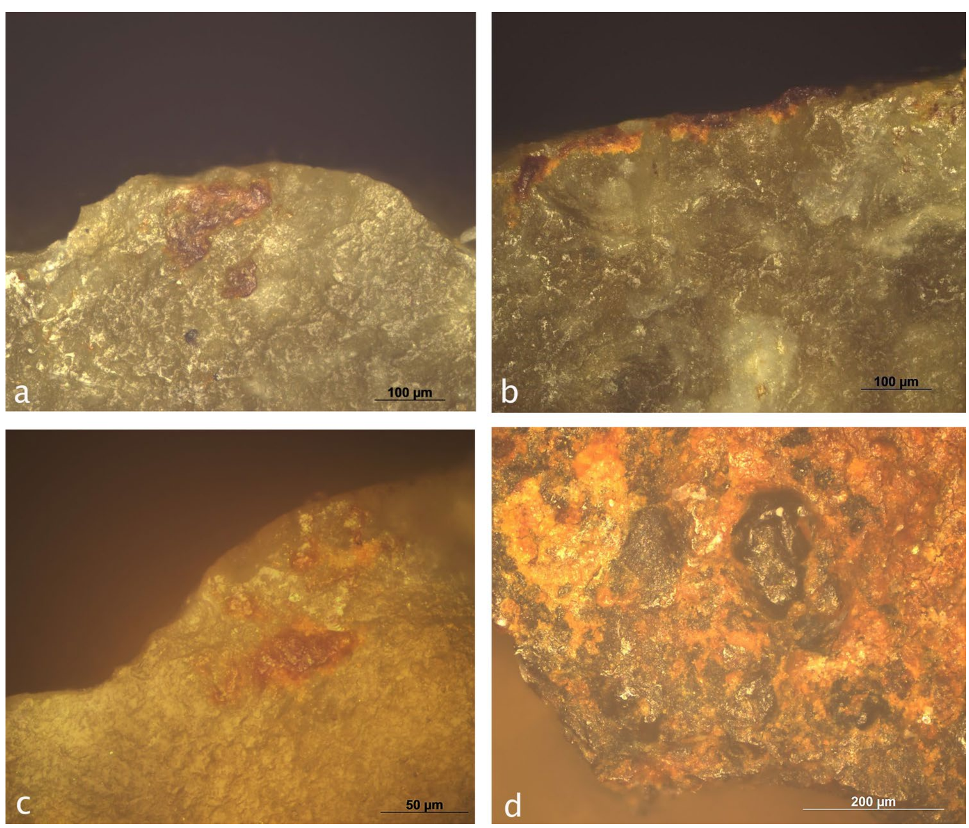

Fig. 1 Examples of natural iron oxide depositions with associated alteration polishes on archaeological artefacts from the site of Ham Aubrug-gestraat (Belgium) (adapted with courtesy from Tomasso & Rots, 2021; pictures taken by S. Tomasso; Zeiss AxioImager reflected-light microscope with an AxioCam ICc5 5-MP digital camera): **a** deposition and polish on the ventral distal right edge of artefact HAM_102301501 with a magnification of × 200 ((LD Epiplan 20 × /0.40); **b** deposition and polish on the damaged ventral distal edge of artefact HAM_102301501 with a magnification of × 200 ((LD Epiplan 20 × /0.40); **c** deposition and polish on the ventral distal left edge of artefact HAM_101506901 with a magnification of × 500 (LD Epiplan 50 × /0.50); **d** deposition and polish on the ventral distal edge of HAM_101606103 with a magnification of × 200 (LD Epiplan 20 × /0.40)

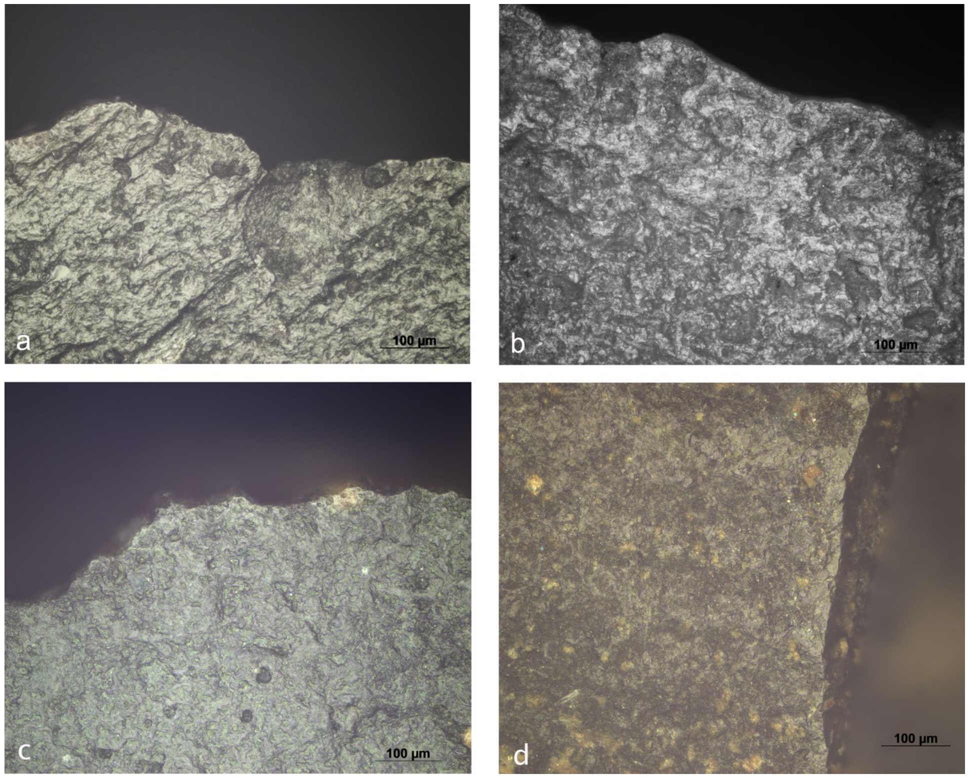

Fig. 2 Examples of alteration polishes as documented on Middle Palaeolithic tools from the site of Ifri n'Ammar (adapted with courtesy from Tomasso, 2024; pictures taken by S. Tomasso; Zeiss AxioImager reflected-light microscope with an AxioCam ICc5 5-MP digital camera): **a** alteration polish and edge damage on the ventral distal left edge of IA90, ×200 (LD Epiplan 20×/0.40); **b** alteration polish and edge rounding on the ventral distal edge of IA541, ×200 (LD Epiplan 20×/0.40); **c** striations and alteration polish on the ventral distal edge of IA1651, ×200 (LD Epiplan 20×/0.40); **d** alteration polish on the dorsal ridge of IA 4373, ×200 (LD Epiplan 20×/0.40)

proved patinated). Confidentially identifying traces as anthropogenic is the prerequisite to interpreting use-wear findings. Based on the published illustrations and explanations, it remains unclear whether the presented use-wear traces are post-depositional modifications, remnant polishes from modern handling due to insufficient cleaning, or use-wear traces. Not a single image shows traces with characteristics typical of use-wear, such as a clear concentration on the edge, a clear impact on the edge or rounding, directional features, etc. (cf. Keeley, 1980; Vaughan, 1985; Rots, 2010; see also below). Elements that could further elucidate the origin of the traces are information regarding the preservation state of the entire assemblage which is not reported and a detailed recording of the surface state of the artefacts. For instance, clear documentation could have been made of the inner surfaces of the artefacts not showing wear evidence and the used portions showing organised wear evidence with directional characteristics. Additionally, comparisons between the observed wear evidence and experimental references on which they rely for their interpretation would have been important. The five artefacts lack information on the depositional context and are isolated from the original assemblage, implying that one should be very cautious with the observed wear. It cannot be verified to what extent the observed wear evidence is also present on large portions of the assemblage. Interpreting the observed wear as being functional without any evaluation of these issues is highly problematic.

Therefore, more information on the preservation of the entire assemblage is needed to rule out whether the observed traces are not the effect of post-depositional processes. One of the ways in which this can be done is by documenting and presenting the well-preserved edges of the artefacts that show the absence of post-depositional processes.

Use-Wear and Hafting Evidence

The authors claim that four out of the five pieces from Le Moustier were used and do not display post-depositional wear that interferes with the use interpretation. They argue that evidence for use is visible on the edges, though they do not provide an interpretation of what this use would be. They mention the presence of micro-fractures and localised polishes and argue that the wear evidence would be distinct for use. It has been abundantly shown that wear traces from use should show a clear impact and concentration on the edge, a clear directional character and an association of different kinds of traces (polish, rounding, scarring, striations) (Keeley, 1980; Semenov, 1964; Vaughan, 1985). The presence of polish does not clarify matters here as alteration is known to result in polish formation (Levi-Sala, 1986; Plisson and Mauger, 1988, see also Fig. 3). The absence of demonstrated and discussed diagnostic features within the wear traces is thus problematic. Observations also do not seem to have relied on an experimental reference framework, which further adds to the problem as such a framework is a

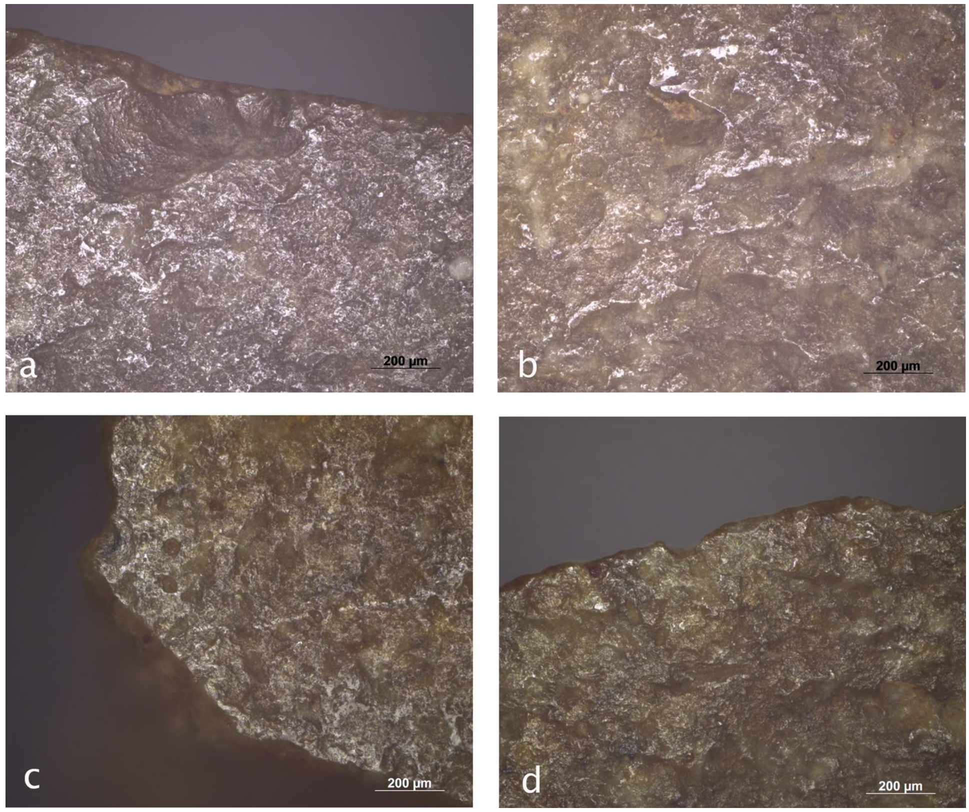

Fig. 3 Examples of alteration wear due to post-depositional processes on archaeological artefacts from Ham Aubruggestraat (Belgium) (adapted with courtesy from Tomasso & Rots, 2021; pictures taken by S. Tomasso; Zeiss AxioImager reflected-light microscope with an AxioCam ICc5 5-MP digital camera: **a** alteration polish and edge damage on the ventral medial left edge of artefact HAM_101606203, × 100 (EC Epiplan-Neofluar 10 × /0.25 HD DIC); **b** alteration polish on the ventral medial surface of artefact HAM_101606203, × 100 (EC Epiplan-Neofluar 10 × /0.25 HD DIC); **c** alteration polish and striation on the ventral medial right edge of artefact HAM_101606203, × 100 (EC Epiplan-Neofluar 10 × /0.25 HD DIC); **d** alteration polish and edge damage on the ventral distal edge of artefact HAM_101606203; × 100 (EC Epiplan-Neofluar 10 × /0.25 HD DIC)

prerequisite for these types of approaches (e.g. Keeley, 1980; Rots, 2010; Vaughan, 1985).

Subsequently, the authors argue that wear related to hafting would be visible under the colourant stains, away from the edges, on dorsal and ventral faces. They describe it as a bright polish with striations even though they also state they did not clean these areas to preserve the residues. Wear observations on uncleaned surfaces are to be avoided especially for pieces with long curation histories. Handling objects results in the transfer of grease and skin flakes on the surface (Cnuts & Rots, 2018; Frahm et al., 2022; Pedergnana et al., 2016) that could obscure any other traces (e.g. Keeley, 1980; Levi-Sala, 1996). Due to the absence of robust cleaning procedures in the study in question, the possibility remains that the observed traces are from handling and more information would be needed to rule this out.

The problems with the wear analysis are crucial as the authors significantly rely on this evidence to isolate a presumed active and non-active part of the artefacts. The authors build their inferences regarding the use of an adhesive grip from the significant polish formation in the hafted area. This is a puzzling conclusion as it is in contradiction with previous work done on prehension and hafting wear (Rots, 2003, 2010) showing clearly that no polish forms for the inferred type of arrangement in the hafted area, aside from isolated and very particular adhesive-related frictional spots and possible scarring. Thus without further data, no convincing argument for hafting can be made for the artefacts in question.

Inorganic Residue Identification

Wear observations are supplemented by an SEM–EDS analysis of residues from two artefacts. The analysis, however, was not performed on the artefacts themselves as would typically be the case in functional approaches. Assumed residues were scraped off from the artefacts and analysed, but the exact sampling and analytical conditions were not provided. This is problematic because an observation of the residue on the artefact would have allowed the recording of visual characteristics such as smearing which is of functional importance (Cnuts & Rots, 2018) and it would also have allowed to verify possible direct associations with surface features or wear. Depending on the mounting medium that was used, it may have interfered with the EDS result (see Hayes et al., 2019 for information on the subject). The maximum electron penetration depth of the electrons will depend on the density of the material and the applied accelerating voltage (kV) and can be calculated using Castaing's formula (Castaing, 1960). For example, using carbon tape as a mounting medium to fix the powdered residues can explain the high presence of carbon. In addition, the long curation history may have led to the deposition of skin flakes and finger lipids onto the iron residues, which could have been visually verified if the residues had been left on the tool. Such contaminations from manipulation are omnipresent on artefacts with a long curation history and may have contributed to the carbon and sulphur signal (see for example Pedergnana et al., 2016).

The scraped-off powders are said to contain iron and were interpreted as inorganic fillers, consisting of goethite. This interpretation is based on FTIR spectra of the iron oxide deposit containing two relatively sharp bands of the δ(OH) and γ(OH) vibrations of α-FeO(OH) (goethite) observed at 890 and 800 cm^{-1} and low-frequency envelope < 700 cm^{-1} attributed to Fe–O vibrations in α-FeO(OH). While the analysis of goethite through FTIR spectroscopy can be considered a first step in a reliable identification of these inorganic residues, the technique does not distinguish between inorganic and organic residues, and a more accurate identification of goethite requires a multi-analytical approach that includes several methods such as X-ray diffraction (XRD) to specifically target inorganic molecules like iron oxides (Dayet, 2021). Moreover, the fact that goethite is present in the form of spherulites indicates that these residues were most likely deposited through post-depositional precipitation due to the presence of water (Meakin & Jamtveit, 2010) and were not deposited intentionally. In this case, the most likely scenario is that iron-rich groundwater encountered an environment where oxygen was available, in this case at the lithic-sediment interface, causing the iron oxides to precipitate out of solution (Fonolla et al., 2020). The precipitated iron oxides then grew concentrically around a central core to form larger concretions or nodules (spherulites). It is well-known that goethite is naturally present within caves (Broughton 1971; Northup & Lavoie, 2001), especially within fluvial/aqueous contexts where they are formed through precipitation. As no information is available on the precise location and context in which the artefacts have been found in the cave or the soil composition, it is difficult to rule out the post-depositional origin of these goethite residues. Thus, the evidence presented is too weak to claim that goethite is the only viable identification for the residue, and furthermore that it was deposited on the tool as a result of intentional human actions.

Organic Residue Identification

The authors subsequently assume that the organic fraction in the residue is an adhesive and they identify it as bitumen. This identification is based on the presence of peaks within a single FTIR spectrum that were linked with a (modern) bitumen reference sample. The spectrum of the Le Moustier piece and the bitumen reference spectrum have some overlap in the 1458 (aliphatics) and 1021 (sulfoxides), but the archaeological FTIR spectrum has issues with contaminations. Between 1600 and 1700 cm^{-1}, a broad peak is assigned to water contamination from the KBr pellets. Previous studies on the molecular identification of Palaeolithic bitumen demonstrate that weathered bitumen may contain a high number of ketones and carboxylic acids (Cârciumaru et al., 2012). This might result in a broad peak between 1600 and 1700 cm^{-1} in accordance with the C = O groups present (Burger et al., 2016; Monnier et al., 2013). In the FTIR spectra of Schmidt et al. (2024), a similar broad peak between 1600 and 1700 cm^{-1} is present; however, this broad peak is interpreted as OH contamination from the KBr pellet. Moreover, the peak at 1375 cm^{-1} is assigned to KBr contamination by Schmidt et al.; however, the band at 1375 cm^{-1} could also be assigned to sulphones, which are expected to be in bitumen too. The presence of the contamination is troublesome as it occurs in an important region for the interpretation of bitumen and is causing ambiguity in the peak interpretation. Schmidt et al. make no attempt to remove the water contamination by, for example, drying the KBr pellets and remeasuring or taking the second derivative of the spectrum. Without attempts to remove the contamination and in the absence of uncompromised FTIR data, the attribution to bitumen is unreliable.

Furthermore, given the isolated nature of the artefacts and the long curation history, contamination is to be expected. In this perspective, the presence of sulphur also does not strengthen the case because sulphur is a known contaminant, and the presence of sulphur has been detected in natural resin (Bradtmöller et al., 2016; Dinnis et al., 2009; Pawlik & Thissen, 2011), as part of soil contamination (Devièse et al., 2020; Venditti et al., 2019), animal tissue, as well as human skin particles (Pedergnana & Ollé, 2018; Pedergnana et al., 2016). While FTIR is a good technique to quickly identify organic residues, it has more difficulties in identifying weathered and/or contaminated organic residues, especially in the absence of good referential data. When dealing with heavily contaminated and/or weathered organic residue, analysis with (pyrolysis-) gas chromatography-mass spectrometry ((py-)GC–MS) is advisable. Indeed, (py-)GC–MS is the most sensitive technique known as it separates the sample on a molecular basis, making it possible to identify the molecules present and to separate the contamination from the sample (Cârciumaru et al., 2012; Hauck et al., 2013). Especially, py-GC–MS is known to be more sensitive towards the identification of bitumen as it enables the analysis of insoluble molecules (Nardella et al., 2021). Instead of opting for this technique, Schmidt et al. tried to perform GC–MS on the sample, which proved unsuccessful, but such an analysis can only show that the residue is probably not of vegetal or animal origin, it does not prove that it is therefore bitumen.

Finally, it has to be reiterated that the authors did not report any efforts to eliminate the possibility of contamination in the residues of the artefacts. This is problematic given the limited information available regarding the artefacts' curation and handling in the past. This also concerns contamination that was introduced during sample preparation, as shown in the single FTIR spectrum upon which their interpretation is based. Without contamination being ruled out as a factor, the interpretation can therefore not be considered reliable.

Conclusion

We found that the claims presented by Schmidt et al. are unsubstantiated and that alternative interpretations are more plausible and should be taken into account. Context, preservation state, wear and residue identification are critical components required to make a convincing argument that these pieces were used and encased in an adhesive that could give insight into the cognition of their makers. Post-depositional alterations and effects of long-term curation in uncertain conditions cannot be ruled out at this point, given the questionable history of these finds. Hypotheses regarding the finds being potentially associated with either modern humans or Neanderthals seem irrelevant, given the problematic context and stratigraphic origin of the artefacts.

We tried to critically evaluate each argument put forward by the authors to substantiate their claims, and we found flaws in each one of them. Therefore, the presented evidence does not provide solid grounds for arguing that ochre-based compound adhesives would have been used during the Middle Palaeolithic in Europe. This also brings into question any other inferences made by the authors regarding adhesive technology and the cognitive capacities of the makers.

We thus conclude that the Berlin Le Moustier artefacts are not the oldest compound adhesives found in a European context. Bold claims such as the ones presented by Schmidt et al. should rely on robust analytical evidence and secure contextual information.

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Author Contribution V.R. and R.O wrote the main manuscript with all authors (V.R., D.C., J.C., A.L., R.O.) contributing to parts of the text. All authors reviewed the manuscript. V.R. prepared the figures.

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Data Availability The authors confirm that all data generated or analysed during this study are included in this published article.

Declarations

Competing Interests The authors declare no competing interests.

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