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Citation

Aleo, A., Jerardino, A., Chasan, R., Despotopoulou, M., Ngan-Tillard, D. J. M., Hendrikx, R. W. A., & Langejans, G. H. J. (2024). A multi-analytical approach reveals flexible compound adhesive technology at Steenbokfontein Cave, Western Cape. *Journal Of Archaeological Science*, *167*. doi:10.1016/j.jas.2024.105997

Journal of Archaeological Science 167 (2024) 105997

Contents lists available at ScienceDirect

Journal of Archaeological Science

journal homepage: www.elsevier.com/locate/jas

A multi-analytical approach reveals flexible compound adhesive technology at Steenbokfontein Cave, Western Cape

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ARTICLE INFO

Keywords:
Use-wear
Hafting
Adhesives
Ochre
Later stone age
South Africa

ABSTRACT

Evidence of different compound resin-based adhesives is present in South Africa from at least 77000 years ago. Ancient glue production is considered one of the oldest known highly complex technologies, requiring advanced technological and mental abilities. However, our current knowledge of adhesive materials, recipes, and uses in South Africa is limited by the lack of in-depth analysis and molecular characterization of residues. To deepen our knowledge of past adhesive technology, we performed a detailed multi-analytical analysis (use-wear, XRD, μ-CT, IR spectroscopy, GC-MS) of 30 Later Stone Age tools with adhesive remains from Steenbokfontein Cave, South Africa. At the site, tools made of various rocks were hafted with compound adhesives, and we identified three recipes: 1) resin/tar of *Widdringtonia* or *Podocarpus* species combined with hematite; 2) resin/tar of *Widdringtonia* or *Podocarpus* species mixed with hematite and another plant exudate; 3) resin/tar without hematite. The studied scrapers were used in hide-working activities, and the studied cutting tools were used to work animal and soft plant matters. All scrapers display evidence of intense resharpening and were discarded when no longer useable. The combination of different methods for residue analysis reveals the flexibility of adhesive technology at Steenbokfontein. Despite the consistent use of conifer resin/tar throughout the sequence, we observed that other ingredients were added or excluded independently of the tools' raw materials and functions. Our results highlight the long-lasting tradition of using adhesive material from conifer species but also the adaptability and flexibility of adhesive traditions. The systematic application of this multi-analytical approach to Pleistocene adhesives will be useful to better characterise adhesive traditions and enhance the debate on the technological, cognitive, and behavioural implications of this technology.

1. Introduction

Evidence for different adhesives, including compound adhesives, is present in South Africa from at least ~77000 years ago (Rots, et al., 2017; Wadley et al., 2009). Compound adhesives consist of multiple ingredients. The main ingredient is the tackifier that provides the stickiness like a resin or tar, and sometimes plasticisers are added to make the adhesive less brittle or more pliable. Examples of the latter are beeswax and fat. Other ingredients like ochre, sand, and fibres are added to increase strength, durability, and pliability (Langejans, et al., 2022). The manufacture of compound adhesives requires considerable technical and cognitive skills, including an understanding of chemical reactions, the use of pyrotechnology, abstraction, recursion, and cognitive fluidity (Wadley, 2010; Wadley et al., 2009). The identification of differences in the composition of compound adhesives in the archaeological record has been viewed in relation to the various raw materials from which tools are made of and tools uses. This evidence highlights the versatility of prehistoric resin-based adhesives (Wadley, et al., 2015; Lombard, 2007).

While there are several reports on adhesive remains from South

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https://doi.org/10.1016/j.jas.2024.105997
Received 20 November 2023; Received in revised form 11 April 2024; Accepted 9 May 2024

African assemblages dated to the Middle Stone Age (MSA) and Later Stone Age (LSA), few of these performed chemical studies for secure identification. Adhesives have largely been studied through microscopy by documenting their distinctive morphologies and systematically mapping of their spatial distribution and association with use-wear traces (e.g., Lombard, 2007; Lombard, 2006; Gibson et al., 2004). However, several studies have emphasised the limitations of interpretation based solely on residues morphology and distribution patterns (Pedergnana, 2020; Pedergnana et al., 2016). Even when adhesive residues are correctly identified, optical microscopy alone cannot securely differentiate between plant exudates of different species (Soriano, et al., 2015). Overall, the paucity of molecularly identified Stone Age adhesive residues limits our understanding of this technology. Without knowing the basic ingredients, additives, loading agents, and production methods of adhesives, current inferences on the complexity of compound adhesives are hard to validate.

The organic components of adhesives are occasionally identified with gas chromatography-mass spectrometry (GC-MS). To date, only five MSA residue samples were analysed by GC-MS: a quartz flake from Diepkloof Rock Shelter (Charrié-Duhaut, et al., 2013) and four segments, one from Rose Cottage Cave, and three from Sibudu Cave (Soriano, et al., 2015) (Fig. 1). *Podocarpus* resin, possibly mixed with bone and quartz, was identified at Diepkloof, while only one of the segments from Sibudu provided evidence of a conifer resin (e.g., *Podocarpaceae* sp.) used to enable hafting. For the LSA, three artefacts with macroscopic residue from Elands Bay Cave (Charrié-Duhaut, et al., 2016) and three microliths from the early LSA of Border Cave (Fig. 1) (Villa, et al., 2012) were chemically analysed. GC-MS identified the residues as an adhesive, either resin or tar, made from species belonging to the *Podocarpaceae* family, likely *Podocarpus elongatus*. In the case of Elands Bay Cave, the adhesive was possibly mixed with organic and inorganic additives such as fat and quartz grains (Charrié-Duhaut, et al., 2016). More adhesive residues form several South African LSA sites were analysed with GC-MS by Veall (2019), revealing the presence of compound adhesives produced from plant exudates, such as conifer resin and pitch and latex, and mixed with organic and inorganic additives such as fat, waxes, and crushed minerals. On the whole the sample size of analysed adhesives per site is limited, complicating a deep diachronic, regional, and technical understanding of adhesive technology.

Other methods of studying both the organic and inorganic fractions of adhesive residues, occasionally used in combination with GC-MS, include scanning electron microscopy (SEM), Raman, and infrared spectroscopy (Wojcieszak and Wadley, 2018; Charrié-Duhaut, et al., 2016; Villa et al., 2015). Although GC-MS is more sensitive and capable of accurate detection of specific organic compounds, these other methods have the advantage of being non-invasive, relatively cheap, and quick (cf. Shillito et al., 2009). Despite the increasing popularity of chemical studies in residue analysis, the lack of a systematic molecular identification of alleged adhesive remains on South African tools, and particularly within assemblages, identified only by means of optical microscopy still represents a drawback in the field.

To gain more information on adhesive production and use during the South African Stone Age and enhance the discussion on the complexity of adhesive technology, we analysed a sample of 30 LSA artefacts with macro-residues from Steenbokfontein Cave, Western Cape (Fig. 1). Despite never being chemically analysed, based on residues' characteristics and distribution and the presence of two previously identified adhesive finds (Jerardino, 2001), the residues are interpreted as adhesive remains. To reconstruct the use-life of the stone tools and their residues and verify their nature, the artefacts were analysed with optical

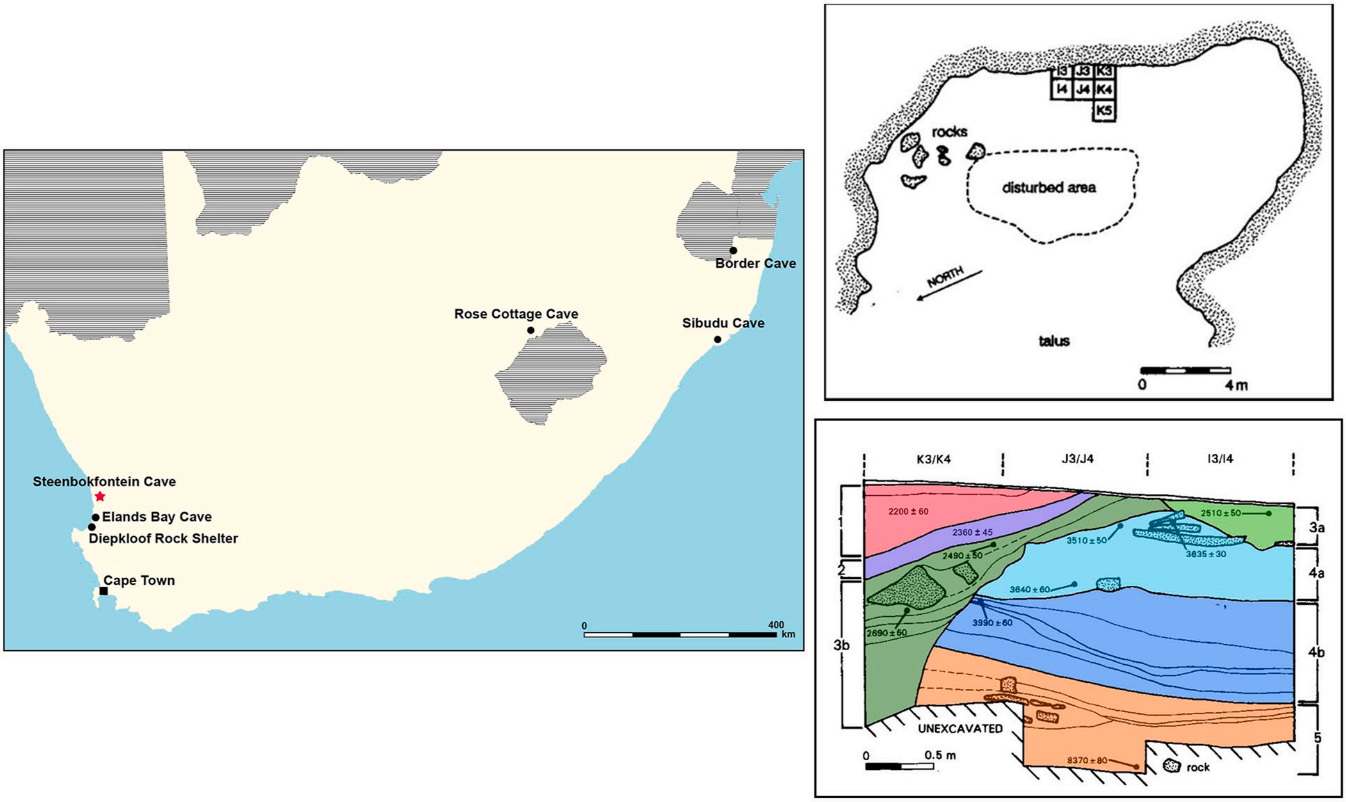

Fig. 1. Location of Steenbokfontein Cave and other archaeological sites mentioned in the text. Site plan and stratigraphic section of the cave. From Jerardino and Swanepoel (1999), modified.

microscopy, spectrographic methods, and chemical analysis. This work represents one of the first comprehensive multi-analytical studies of a large sample of tools with potential adhesive remains dated to the LSA. The integration of optical descriptions and molecular data of use-wear and residues will help document the use of adhesives at the site and illuminate on raw material selection, recipe composition, and potential diachronic changes in adhesive technology. By analysing the Steenbokfontein Cave tools, we lay the groundwork for establishing the regional and geographical continuity of adhesive technology during the South African Stone Age.

2. Materials and methods

2.1. Site introduction and materials

Steenbokfontein Cave is located about 200 km north of Cape Town and about 2.5 km east of the nearest shoreline on the west coast of South Africa (Fig. 1). Excavations at the site were undertaken between 1992 and 1997, and seven occupation layers were identified thus far. Radiocarbon analysis dates this sequence to between 2005 and 9530 cal BP (Jerardino, 2022). Despite the relatively small volume excavated from this coastal site (6.75 m^3), Steenbokfontein Cave has provided unique and key observations to understand the Holocene cultural sequence of the central west coast (Jerardino, et al., 2013). Roughly 11000 flaked stone artefacts were recovered, of which 368 are formal tools. The lithic technocomplex of Steenbokfontein Cave is characterised as a Wilton and microlith final LSA assemblage, with scrapers being the most frequently identified retouched pieces (Jerardino, 2013; Lombard et al., 2012). Lithic raw materials are dominated by quartz and quartzite, which are ubiquitous locally, and exotic rocks such as silcrete, hornfels, and cryptocrystalline silica (CCS) are present in lower percentages (Jerardino, 2013). The highest percentages of exotic lithic raw materials are present in layers 4b (2σ: 4580–4155 cal BP, 10.4%) and 5 (2σ: 5480–9030 cal BP, 13.5%). This temporal trend is also reflected in several other sites within a 20 km radius (Jerardino, et al., 2021; Jerardino, 2013) suggesting that mobility was increasingly restricted to the coast and its foreland in later occupations during the accumulation of layers 4a to 1 (2σ: 3990–2020 cal BP) (Jerardino, et al., 2013). This coastal landscape includes sandstone outcrops and ravines 25 km south or inland where large shrubs and trees grow (Cartwright, 2013).

The Wilton technocomplex is one of several microlithic tool production sequences in the LSA. The microlithic tool production in the LSA started around 40000 years ago, contrasting with the preceding MSA, during which larger stone artefacts were produced (Lombard, et al., 2022; Lombard et al., 2012). Research shows that LSA lithic miniaturization during the Robberg dated to about 18000–12000 BP was the result of technological efficiency decisions with high adaptive payoffs, including bipolar bladelet production (Pargeter and Faith, 2020). This is likely to also have been the case for the Wilton technocomplex, but additional research must confirm this. Unfortunately and with few exceptions, the function of Wilton and post-Wilton microliths in southern Africa has received little attention when compared to much older lithic industries (e.g., Lombard, 2020). These few studies show that microliths were used for different purposes such as wood working (Binneman, 1983) or as insets in hunting composite tools (Lombard and Parsons, 2008). While these and many other microliths were hafted, thumbnail-sized and larger artefacts may not have needed this form of fixture and could have been handheld for hide-scraping.

Steenbokfontein Cave yielded two unique adhesive finds: a stone adze embedded in a large adhesive lump and a cigar-shaped resinous object. Both artefacts were found in layer 1, which roughly dates to 2200 cal BP (Jerardino, 2001). Additionally, macroscopic mastic residues or staining were observed on 30 retouched stone tools from all the stratigraphic units, but they were chemically characterised. Of these tools, two are from layer 5, 10 are from layer 4 (4a and 4b), 10 are from layer 3 (3a and 3b), two are from layer 2, and six are from layer 1 (SOM Table S1). The material is curated at the Department of Archaeology, University of Cape Town (South Africa). We collected morphometric data from all the tools and inspected them with a stereo microscope and a Dino-Lite Edge Digital Microscope (AM7915MZT) to describe the residues.

Of these 30 tools, we selected 13 for in-depth use-wear analysis and molecular identification of residues considering chronostratigraphy, tool morphology, and raw materials (Table 1). In-depth analyses were performed at Leiden University and Delft University of Technology (the Netherlands). The selection allows us to document possible diachronic changes in adhesive technology and directly link adhesives to tool technological aspects (e.g., tool type, tool use, and rocks material properties). All the selected artefacts display macro-residue or black staining clearly delimited to an area and always observed on both sides of the tools (Fig. 2).

2.2. Methods

As the first step of analysis, we examined all the tools with adhesive residues (N = 30) with a stereomicroscope and a Dino-Lite Edge Digital Microscope (AM7915MZT) to describe the morphological features of residues. Thirteen tools were subsequently selected for in-depth non-destructive and destructive analyses (Table 1).

Non-destructive analyses consist of high power optical microscopy for functional analysis, X-ray diffraction (XRD), X-ray micro-computed tomography (μ-CT), Fourier-Transform Infrared microspectroscopy (micro-FTIR) and attenuated total reflectance (ATR-FTIR) (SOM S1). Optical microscopy provides evidence on the use-life of the objects and their residues (Van Gijn, 2010). XRD and μ-CT provide information on the inorganic components of the adhesive mixtures. XRD was performed on 11 tools to verify the presence of additives, such as ochre (e.g., Rosso et al., 2016). We excluded SBF2 and SBF27 because the residues are covered with soil particles and contaminations, which would prohibit confident identification of additives. Four tools displaying millimetres-thick residue were selected for μ-CT to analyse the internal structure of the adhesives and confirm the presence of additives (cf. Niekus et al., 2019). Micro-FTIR and ATR-FTIR were used complementarily to identify organic components in the adhesives (Chen, et al., 2022; Helwig et al., 2014). Six tools, which were not sampled for GC-MS, were analysed with micro-FTIR in reflectance mode to gain information on the nature of the residues. Additionally, ATR was performed on a residue sample removed from SBF14 to compare the results of ATR with those obtained in reflectance mode. Excluding ATR, these analyses are non-invasive and do not require destructive sampling. Due to the risk of accidental damage to the artefact, ATR was not used for *in-situ* residue characterisation.

Destructive analysis consists of GC-MS (SOM S1). Despite its destructive nature, GC-MS is the most precise method to characterise unknown organic residues in archaeological samples (cf. Langejans et al., 2022). GC-MS allows the identification of material-specific biomarkers which are used to fingerprint unknown mixtures (Evershed, 2008). Eight tools were analysed for GC-MS following previously

Table 1
Overview of the 13 stone tools selected for in-depth analysis. CCS: crypto crystalline rock.

ID	Layer	Square	Tool type	Mastic	Mastic Stained	Raw material	Age cal BP	Analysis
SBF2	5	K3	Scraper	Yes		Silcrete	c. 5240	Traceology, μ-CT, GC-MS
SBF4	4b	I4	Scraper		Yes	Quartz	c. 4390	Traceology, XRD, FTIR
SBF5	4b	I3	Scraper (convex)	Yes		CCS	c. 4390	Traceology, XRD, μ-CT, GC-MS
SBF9	4b	J3	Retouched piece	Yes	Yes	Quartz	c. 4390	Traceology, XRD, FTIR
SBF10	4b	I4	Scraper		Yes	CCS	c. 3810	Traceology, XRD, FTIR
SBF14	3b	K5	Scraper (boat-shaped)	Yes		Quartz	c. 2770	Traceology, XRD, μ-CT, ATR, GC-MS
SBF15	3b	K3	Scraper (boat-shaped)		Yes	Quartz	c. 2770	Traceology, XRD, GC-MS
SBF23	3b	H3/I3	Adze	Yes	Yes	Silcrete	c. 2770	Traceology, XRD, GC-MS
SBF17	3a	I3	Scraper (boat-shaped)	Yes		Quartz	c. 2545	Traceology, XRD, GC-MS
SBF20	3a	I3	Scraper (boat-shaped, broken)	Yes		Quartz	c. 2545	Traceology, XRD, FTIR, GC-MS
SBF21	2	K3	Scraper	Yes	Yes	Quartz	c. 2340	Traceology, XRD, FTIR
SBF24b	1	K4	Scraper (boat-shaped)	Yes	Yes	Quartz	c. 2170	Traceology, XRD, FTIR
SBF27	1	K3	Scraper (boat-shaped)	Yes		CCS	c. 2170	Traceology, μ-CT, GC-MS

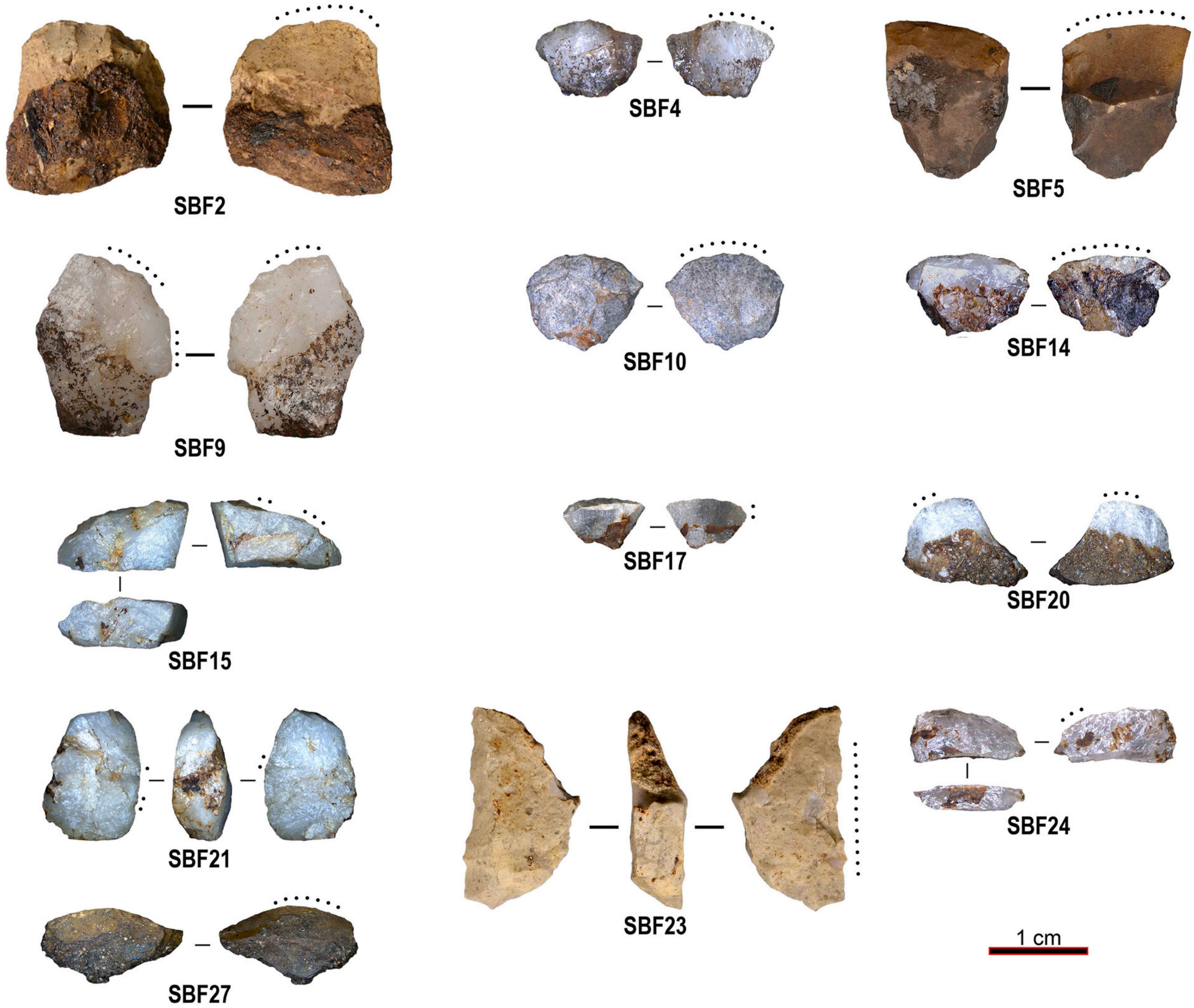

Fig. 2. Tools with residues selected for in-depth analysis. The dotted line indicates the location of wear-traces.

published protocols (Regert, et al., 2006), including two samples (SBF14 and SBF20) that were also analysed with ATR and micro-FTIR respectively. This will help to verify the level of accuracy of spectroscopy results.

3. Results

3.1. Collection overview: typology, raw materials and morphometrics

Most of the analysed tools (N = 24) are typologically classified as scrapers. Two tools are classified as adzes, two as multipurpose retouched tools, and two as utilised flakes. Of the 24 scrapers, 10 are classified as boat-shaped scrapers, eight as convex scrapers, five as generic scraper, and one as a backed scraper. Twenty-three tools are made of quartz, four are made of CCS, and three are made of silcrete (SOM Table S1).

Eighteen tools are complete, while 12 display at least one fracture at one of the extremities. Of the 12 tools with fractures, three are distal fragments, with only the retouched edge preserved (SOM Table S2). These three fragmented tools may have been broken during use or resharpening, but the presence of residues also on the proximal fracture surface (Fig. 2, SBF24) may indicate that they were hafted and used when already fragmented. Complete quartz tools (N = 12) are overall smaller than CCS and silcrete tools (N = 6). The average length/width ratio for quartz tools is 0.89 mm, and the average length/width ratio for non-quartz tools is 1.13 mm. We performed non-parametric Mann-Whitney, and the difference in size is not statistically significant (U = 28.00, p = 0.49).

3.2. Overview of archaeological residues

Considering the whole sample of tools (N = 30), we identified four groups of residues by optical microscopy (Table 2) (Fig. 3). Group 1 is the most common and includes residues documented on 13 tools (Fig. 3.1). Group 2 (Fig. 3.2) includes residues on nine objects; group 3 (Figs. 3.3) and 4 (Fig. 3.4) include residues on three artefacts each. The differentiation of the studied residues into these groups based on their qualitative and morphological characteristics is not strict, and some residues may display characteristics shared with the other groups. These residues are interpreted as organic adhesives, likely a tar or resin or a combination of both, which could have been sourced from trees and other vegetation growing in ravines and outcrops 25 km south or inland from Steenbokfontein Cave (see Cartwright, 2013).

Group 1 residue was recovered primarily in layer 4b (50%), with lower amounts in layer 1 (22%), layer 3b (14%), layer 2 (7%), and layer 5 (7%). Group 2 residue is mainly present in layer 3a (45%), and it is found in lower amount in layers 1 (22%), 3b (22%), and 4b (11%). This distribution reflects that group 1 is predominant in older layers and that in time, its use was superseded by group 2 residues (Fig. 4). This change in adhesive technology may be linked to different production techniques or raw material exploitation.

The residues on two artefacts, SBF1 and SBF13, were not inserted in any of the groups since their distribution and morphological and surface characteristics seem to suggest a post-depositional origin. Residues on both tools are black, granular, cracked, and randomly distributed on the dorsal and ventral surfaces as well as in the fractures. They lack any other characteristics observed in the previously mentioned residues such as the greasiness, the presence of smooth orange semi-translucent inclusion, or the association with thin, flat, semi-translucent orange stains.

Table 2
Qualitative descriptions of residue groups observed on Steenbokfontein tools. Note that residues in group 3 can be assigned to group 1 or 2 based solely on residue's morphological characteristics. Asterisk (*) indicates that those residues were sampled for chemical analysis.

Residue group	Nr of occurrence	Qualitative description
1	13 (SBF3, SBF4, SBF5*, SBF6b, SBF7, SBF8, SBF9, SBF12, SBF15*, SBF21, SBF24b, SBF25, SBF28)	The colour of the residue ranges from black to brown. The residue is usually smooth and matte with cracks on the surface. The limits are sharp and straight, and the residue mostly displays angular terminations. The residue is opaque both when observed in normal and cross-polarised light. Occasionally, thin, flat, orange, semi-translucent/translucent residues are associated with this group. The residue crumbles into small angular fragments.
2	9 (SBF6a, SBF14*, SBF16, SBF17*, SBF18, SBF19, SBF23*, SBF24a, SBF26)	The colour of the residue ranges from brown to orange with a shiny, greasy appearance. The residue is smooth and matte with a rounded shape. When the residue is very thin, it is flat and angular. Cracks are sometimes present on the surface. The residue is opaque and polished when observed in normal light, while semi-translucent spots are visible in cross-polarised light. Occasionally, dark angular inclusions and ochre grains are visible in the residue in cross-polarised light. The residue crumbles into small angular fragments. Thicker lumps of residue are brown, rounded, cracked, and weathered. They are opaque in normal light with some smooth orange inclusions that are semi-translucent in cross-polarised light.
3	3 (SBF2*, SBF20*, SBF27*)	The residue displays on top contaminations from the soil such as sediment grains, charcoal/charred wood, shell fragments, rootlets, etc. The residues underneath the contaminations can either be assigned to groups 1 (SBF2) or 2 (SBF20, SBF27).
4	3 (SBF10, SBF11, SBF22)	The colour of the residue ranges from reddish to light orange with a granular texture. Edges may be either straight and angular or more gradual. The residue is mostly opaque in normal light and opaque with semi-translucent inclusions or semi-translucent in cross-polarised light.

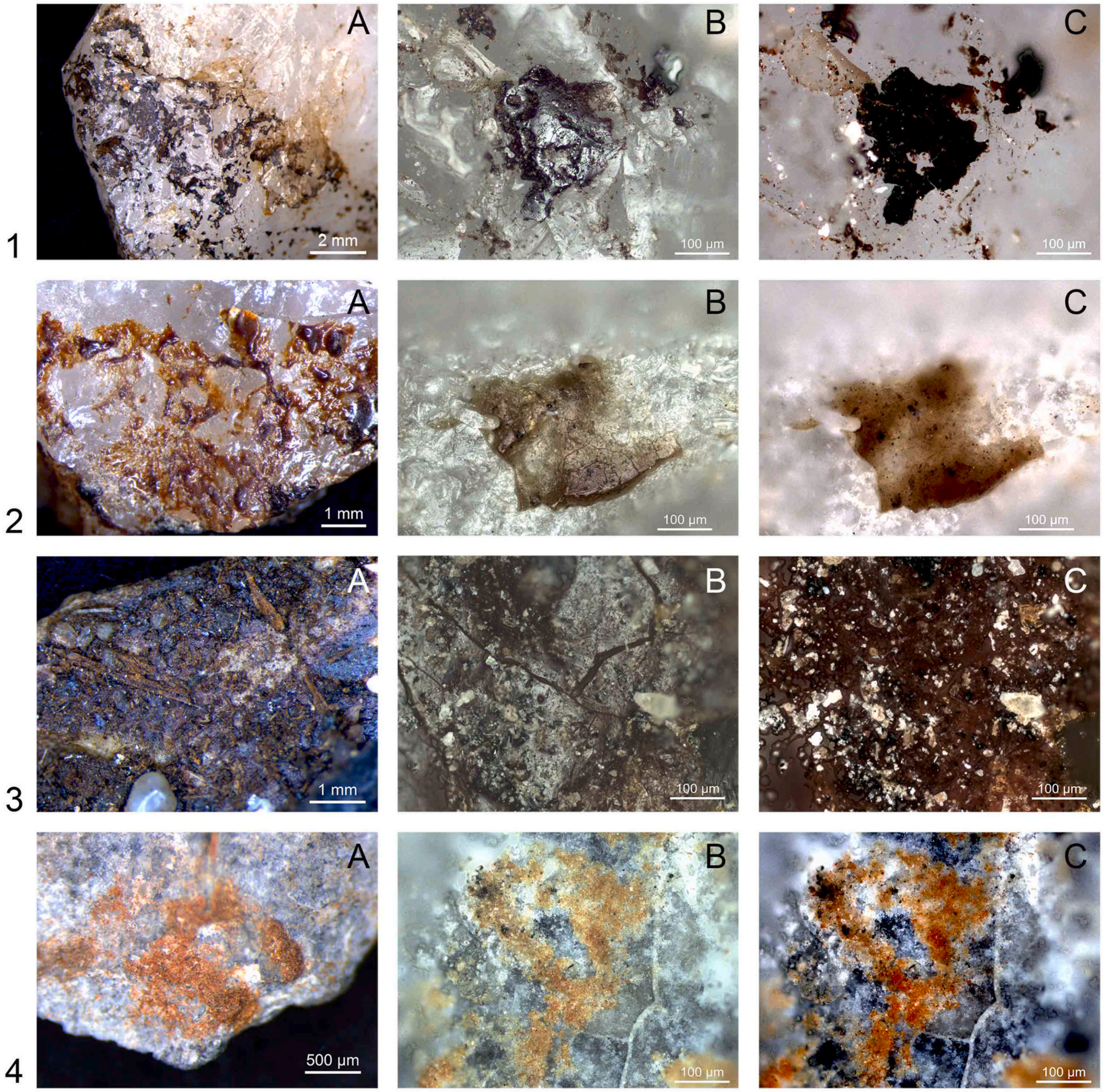

Fig. 3. Different residue groups identified by optical microscopy. A) General view of the residues; B) View of the residues in bright field illumination (magnification 200×); C) View of the residues in cross-polarised light (magnification 200×).

3.3. In-depth analyses

Use-wear traces All the tools analysed (N = 13) display traces of use (Table 3). At least five tools were used with a transverse motion in scraping activities, and five were with a longitudinal motion (Fig. 5). For three tools it was not possible to infer the use-motion. The hardness of the contact materials ranged from soft to medium, with eight tools used on soft material, two on soft/medium material, and three on medium hard material. On eight tools, micro-wear traces allowed a better understanding of the worked materials. For all the tools used in scraping activities (N = 5), hide was identified as the contact material. These tools display light to medium developed edge-rounding and a continuous band of polish along the edge with oblique or transverse directionality (Fig. 5A). One tool (SBF24) displays medium developed edge-rounding and medium developed abrasion of the active edge, suggesting contact with a soft abrasive material, such as dry hide or hide with additives (Fig. 5B). All scrapers (N = 5) display evidence of resharpening of the active edge. Resharpening was identified by the presence of small, overlapping stepped or hinged terminating scars on the dorsal face of the tools and incipient cracks (Fig. 5C and D) (cf. Aleo et al., 2021). Furthermore, one scraper (SBF5) shows on the ventral lateral edge at the haft limit large scars that may be related to de-hafting (Fig. 5E) (cf. Rots and Williamson, 2004). One tool (SBF23) was used to work unspecified animal material due to the rounding of some crystals and isolated spots of rough and greasy polish (Fig. 5F) (cf. Van Gijn, 1990). This tool also displays on the ventral face close, overlapping, step terminating edge-removals with no orientation, likely linked to a different use (Fig. 5G). Two quartz tools (SBF20 and SBF21) were likely used on plant. Both tools show isolated edge-removal, some with longitudinal orientation, in combination with a domed, smooth, and almost ‘fluid’ polish (Fig. 5H) (cf. Aleo, 2022). Lastly, SBF17 displays minimal traces of use likely related to contact with a soft plant, but the available evidence is not enough to reliably infer the contact material.

XRD XRD results (N = 11) allow the identification of additives mixed with the adhesive (SOM Table S3). Hematite (Fe_2O_3) is identified in nine tools, and magnetite (Fe_3O_4) is identified in one. Magnetite is present in

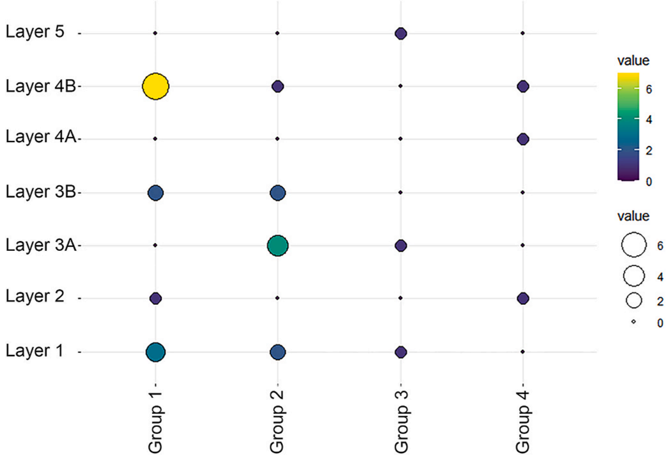

Fig. 4. Ballon plot showing residue types frequency across the different stratigraphic layers.

a sample showing severe thermal damage (SBF10). Experimental work has demonstrated that hematite reduces to magnetite when heated (Lanier, et al., 2009). Hematite signals can come from the minerals the rocks are made of, the burial environment, or the hafting adhesive. Since none of the XRD patterns collected on the rock substrate display hematite contribution (Fig. 6A), we conclude that it was intentionally added to the adhesive mixture. In addition to hematite, the XRD patterns of residue spots of SBF14 display peaks that match with n-paraffin (Fig. 6A). Paraffin wax is detected on both faces of the tool but not on the rock substrate, reinforcing the use of a waxy component, such as beeswax, in the adhesive mixture. However, the XRD pattern of paraffine wax only partially overlaps with the beeswax reference. Hence, the detection of beeswax in SBF14 is likely a misinterpretation. The spectrum also shows the detection of amorphous contribution around 17°, corroborating the organic nature of the residue. Other crystal phases (calcite and halite), which dominate the XRD patterns, are related to the burial environment. Calcite ($CaCO_3$) is a common constituent of archaeological sediment, and halite (NaCl) may relate to soil salinity (El-Ghareb, 2017; Weiner, 2010).

µ-CT scan On the µ-CT scans, the adhesive appears as an amorphous grey matrix with several inclusions that more or less attenuate the X-ray. Since X-ray attenuation is influenced by the concentration and the atomic number of chemical elements, different features can be identified. Cracks that cross through the adhesive and voids, which are the darkest elements, appear in all the scanned residues. Adhesive residues on SBF2 and SBF27 contain several inclusions with an elongated rounded shape interpreted as sediment grains. Some of the inclusions on SBF2 are of biological origin (shells), and one fragment of a biological organism (carbonised plant) is also visible. These inclusions are distributed mostly on top of the residue, so they are likely post-depositional. Both tools display bubbles/voids at the interface between the adhesive and the stone. The adhesive on SBF14 also contains several inclusions, but they are embedded in the matrix. Big angular inclusions are likely quartz fragments since they present the same X-ray attenuation and texture as the quartz raw material of the tool. Fine-grained rounded contaminants evenly distributed within the matrix and characterised by a high X-ray attenuation coefficient are likely iron oxide rich grains (ochre) mixed into the adhesive when it was in a molten state. The residue is no longer fully adhering to the tools and cracks are visible in several locations (Fig. 6B). The residue on SBF4 is very thin. Therefore, no information on its internal structure can be drawn from the scans.

ATR The ATR spectra of SBF14 (Fig. 6C, SOM Table S4) indicate more strongly a gymnosperm extractive, such as a 'cupressaceous resin' (see Tappert et al., 2011). The label 'cupressaceous resin' includes resins originating from conifers of the *Araucariaceae* or *Podocarpaceae* families. The spectra display a broad band around 3300 cm^{-1} attributed to O–H stretching vibrations (ν(O–H)) and a doublet of peaks at around 2920 and 2850 cm^{-1} that correspond to stretching vibrations of the methylene group (vCH2). Typical bands of resins (cf. Martín Ramos et al., 2018) are detected at 1652 cm^{-1} due to ν(C═C) vibrations, at 1230 cm^{-1} associated with δ(C–H) vibrations and at 1710 cm^{-1} strong

Table 3
Overview of the use-wear traces observed on the Steenbokfontein tools.

ID	Macro-wear	Micro-wear	Interpretation
SBF2	-Intense resharpening	- Light edge-rounding - Band of greasy, rough, bright polish	- Scraping hide
SBF4		- Isolated edge-removals - Abrasion - Longitudinal striations	- Cutting medium hard material
SBF5	-Resharpening -Edge-rounding	- Edge-rounding - Band of greasy, rough, bright polish with transverse directionality	- Scraping hide
SBF9		- Edge-removals with longitudinal orientation - Abrasion -Longitudinal striations	- Cutting medium hard material
SBF10	-Resharpening -Edge-rounding -Fire alteration	- Edge-rounding - Band of greasy, rough, dull polish with oblique directionality	- Scraping hide
SBF14	-Some edge-damage	- Abrasion in combination with polish and longitudinal striations	- Cutting medium hard abrasive material
SBF15	-Intense resharpening -Proximal fracture	- Some crystals are rounded - Rough and greasy polish on the crystals with longitudinal directionality	- Longitudinal motion - Soft material
SBF17		- Very few spots of domed, smooth polish with longitudinal directionality	- Motion unclear - Maybe soft plant material but minimal evidence - Likely part of a composite tool
SBF20	-Snap lateral fracture	- Light edge-rounding - Isolated edge removals, some with longitudinal orientation - Domed, smooth, 'fluid' polish with diagonal striations	- Longitudinal motion - Possibly plant material
SBF21	-Few edge-damage	- Light edge-rounding - Light abrasion - Isolated spots of domed, smooth, 'fluid' polish	- Motion unclear - Possibly plant material
SBF23	-Overlapping edge-removals with step terminations	- Light edge-rounding on some protruding crystal - Isolated spots of rough and greasy polish	- Motion unclear - Animal contact material - Edge-damage related to a different use
SBF24	-Resharpening -Proximal fracture	- Light edge-rounding - Medium abrasion - Rough polish	- Scraping hide (dry?)
SBF27	-Intense resharpening	- Light edge-rounding - Band of greasy rough dull polish with transverse directionality - Metal traces → aluminium foil	- Scraping hide

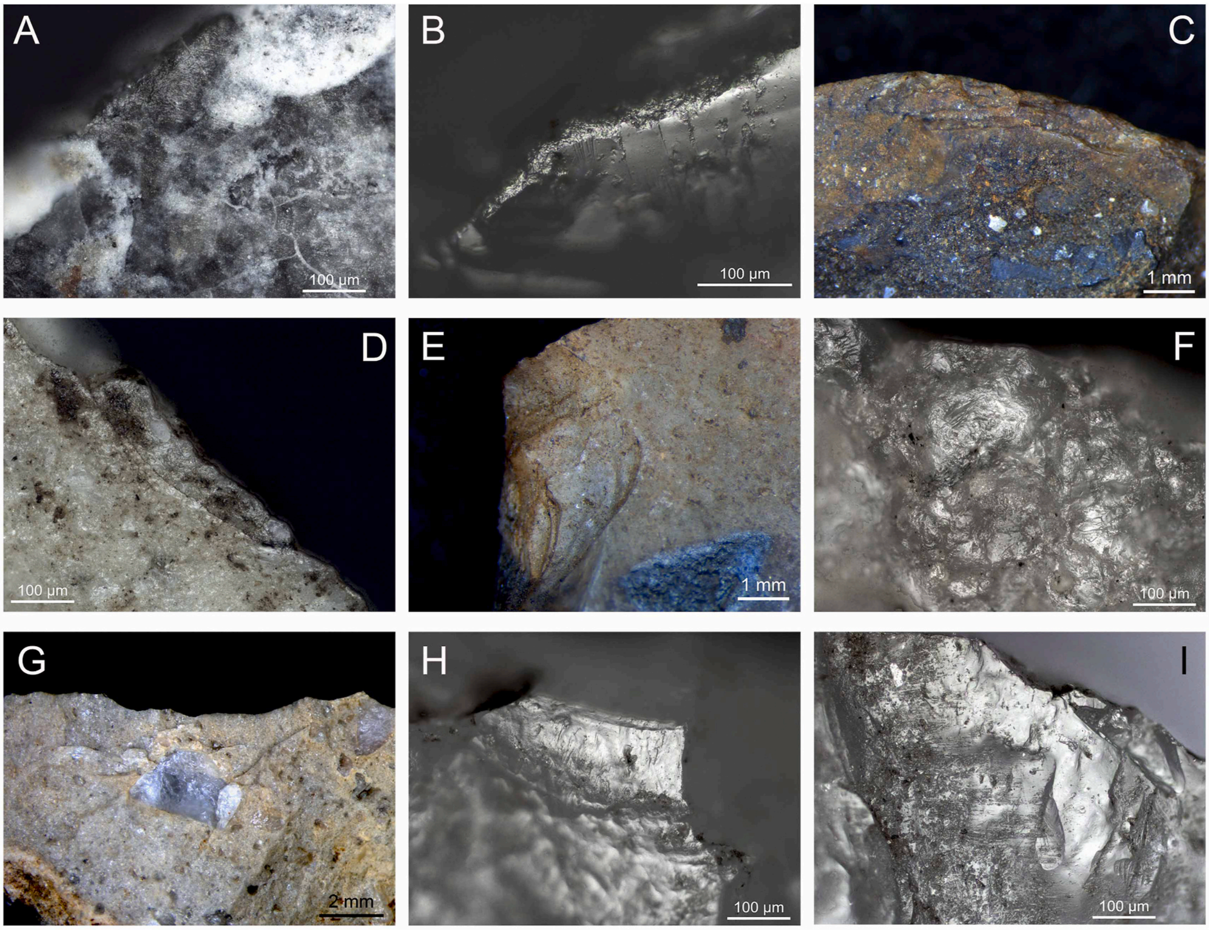

Fig. 5. Selection of use-wear traces documented on the archaeological tools. A) Edge-rounding and continuous band of polish with diagonal directionality from scraping hide (200x); B) Edge-rounding and abrasion from scraping abrasive soft material (300x); C) Overlapping hinge/step terminating fractures from resharpening (16x); D) Incipient crack from resharpening and band of polish from contact with hide (200x); E) Large edge-removal on the lateral edge of SBF5 possibly from dehafting (16x); F) Rounding and greasy polish from contact with soft animal materials (200x); G) Overlapping scars with step terminations on the cutting edge of SBF23; H) Domed, smooth and 'fluid' polish from contact with soft plants (200x); I) Abrasion and longitudinal striations from cutting a medium hard material (200x).

carbonyl (C═O) stretch. The shoulder at 1710 cm^{-1} indicates more strongly abietic acid resins than phenol or ketone group resins. However, the region 1500-700 cm^{-1} shows a stronger resemblance to ketone group resins of trees of the angiosperm clade rather than phenol group resins (Martín Ramos, et al., 2018).

Micro-FTIR The results of the reflectance micro-FTIR (SOM Tables S5–10) are not as clear as the ATR results due to the residue's surface morphology, size, and the significant interference from the underlying siliceous substrate (Monnier, et al., 2017; Prinsloo et al., 2014). For all the analysed tools, there is evidence for a tree extractive, but the clade or family of the tree is unclear. All the spectra except for SBF20 and SBF24 display a doublet of peaks at around 2920 and 2850 cm^{-1} corresponding to stretching vibrations of the methylene group (vCH2) as observed in SBF14. Other typical bands of plant extractives identified include a band around 1650 cm^{-1} due to ν(C═C) vibrations and the one around 1500 cm^{-1} due to ν(C═C) vibrations typical of phenolic resins. In addition to those bands, the micro-FTIR spectra of SBF21 shows a peak at 1450 cm^{-1} and a strong peak at 885 cm^{-1}, attributed to the out-of-plane C–H bending motions in terminal methylene groups, which seem indicative of 'cupressaceous resins' (cf. Tappert et al., 2011).

GC-MS Two samples (SBF5 and SBF15) analysed by GC-MS contain no evidence for archaeological lipids, with only trace amounts of palmitic acid preserved. The remaining six contain evidence for adhesives (SOM Table S11). Identified molecules include saturated fatty acids, hydroxy fatty acids, carboxylic acids, phenolic compounds, diterpenoids, and pentacyclic triterpenoids (Fig. 6D).

Saturated fatty acids range from $C_{7:0}$-$C_{22:0}$, with a predominance of long-chain even-numbered molecules. This wide range of even- and odd-numbered molecules is suggestive of the use of animal and plant products (Pollard and Heron, 2008). Hydroxy fatty acids are both short- (C_6 and C_7) and long-chain (C_{16}, C_{18}, and C_{22}). Dihydroxy fatty acids are less common and include C_8 and C_{10}. The only trihydroxy fatty acid identified is C_{18}, but it is present in each sample. Carboxylic and dicarboxylic acids are primarily short-chain, although two long-chain dicarboxylic acids with C_{16} and C_{18} were consistently identified. Both the hydroxy and carboxylic acids are formed from the degradation of plant biopolymers (e.g., Gandini et al., 2006; Bernards, 2002; Kolattukudy, 2001). Most commonly, they are linked to suberin, which is identified in bark (Kolattukudy, 2001), and their presence is used to suggest the formation of a tar (Ribechini, et al., 2011). However, these molecules also form from cutin, which acts as a waxy substance covering leaves and fruits (Kolattukudy, 2001), so the adhesive may include material from multiple plant parts. Isovanillic acid was identified in one residue (SBF23), and this polyphenol may also be attributed to the degradation of plant biopolymers (Bernards, 2002; Kolattukudy, 2001).

The most prominent class of lipids identified are diterpenoids. The same set was identified in all six samples: sempervirol, 2,3-dehydroferruginol, 14-isopropylpodocarpa-8,11,13-triene-7,13-diol, totarol, and sugiol; dehydrototarol was identified in one adhesive (SBF17). In addition, a series of totarane ketones was identified. These diterpenoids are identified in a limited set of plant families: *Cupressaceae* and *Podocarpaceae* (Cox, et al., 2007; Otto and Wilde, 2001), which both have

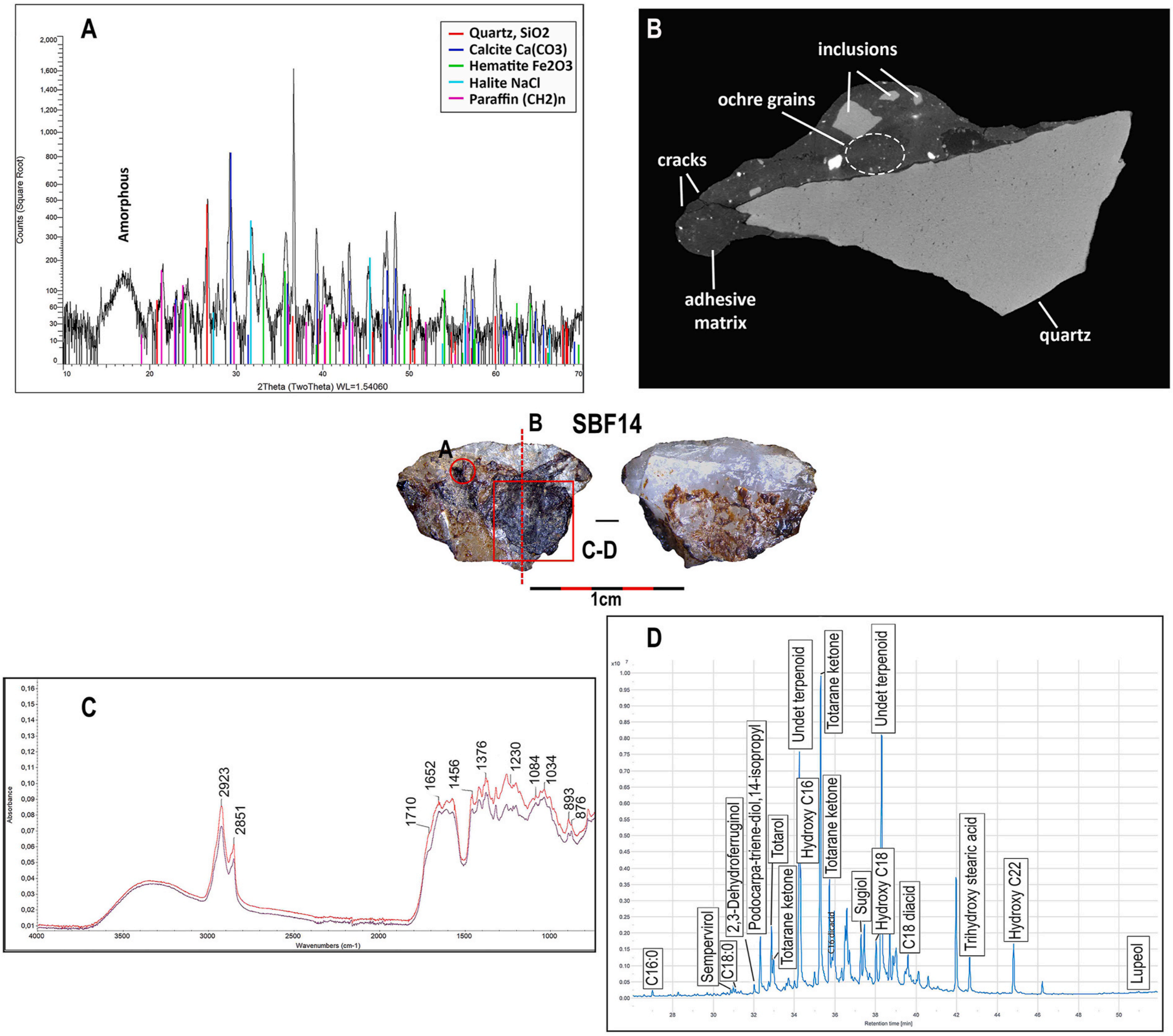

Fig. 6. Summary of results of in-depth analysis on SBF14 residue. A) XRD pattern showing the presence of hematite Fe_2O_3 and paraffine wax $(CH_2)n$ in the residue spots; B) µ -CT scan of the residue showing iron-oxide grains within the adhesives; C) ATR spectra of SBF14. Only peaks assigned to molecular vibrations of adhesives are labelled; D) Partial chromatogram showing terpenoids relevant for the residue molecular identification.

species native to South Africa (Palgrave, 2002). *Cupressaceae* includes different species of *Widdringtonia*, and *Podocarpaceae* includes different species of *Afrocarpus* and *Podocarpus*. Resin may be recovered from the bark of *Widdringtonia* trees as well as from the leaves of *Afrocarpus* and *Podocarpus* trees (Page, 1990a, 1990b). Chemically, these are highly similar (Cox, et al., 2007), so it is unclear which tree species were exploited for their adhesive properties, and it is possible that both were utilised separately or in tandem. Phenolic and aromatic compounds, which form from the degradation of suberin due to intense heating (Robinson et al., 1987) as in tar production, are absent in most of our samples. α,ω–Dicarboxylic acids, which also suggest tar making (Villa, et al., 2012; Ribechini et al., 2011), are conversely consistently identified. The absence of phenolics and aromatics may relate to preservation biases and not exclusively to the use of resin over tar. Therefore, despite some indications in favour of tar, we cannot rule out the use of resin.

In addition, three adhesives (SBF2, SBF14, and SBF17) contain pentacyclic terpenoids. SBF14 and SBF17 contain lupeol, and SBF2 contains α-lupane and lupa-2,20(29)-diene, which form from the degradation of lupeol. Several plant species native to South Africa contain lupeol (Mavundza, et al., 2022; Poumale et al., 2008; Sunita and Abhishek, 2008). Among these, *Euphorbia* is renowned for its latex's adhesive properties (Mwine, et al., 2013). However, in these plant species, lupeol is identified alongside other biomolecules, which are absent from the Steenbokfontein samples, deterring a conclusive identification. Nonetheless, it is clear that in these three adhesives, an additional material was combined with the *Widdringtonia* or *Podocarpus* resin/tar as these do not contain any pentacyclic terpenoids.

4. Discussions

4.1. Function and hafting methods

We documented use-wear traces in different stages of development on all the analysed tools (N = 13). Despite 11 of the analysed tools being typologically classified as scrapers, only five were actually used in scraping activities and particularly for hide-working. Although Wilton scrapers are often assumed to be hide-working tools, functional studies have shown that they were involved in other craft activities, including working wood and bone (Forssman, et al., 2018). The scraping tools we analysed (N = 5) were consistently used on hide; however, our analysis considers only a small number of artefacts, and different uses may emerge from the use-wear study of a larger number of scrapers. Five other tools were likely used to work animal and plant materials with longitudinal motions. Even though we did not observe diagnostic wear

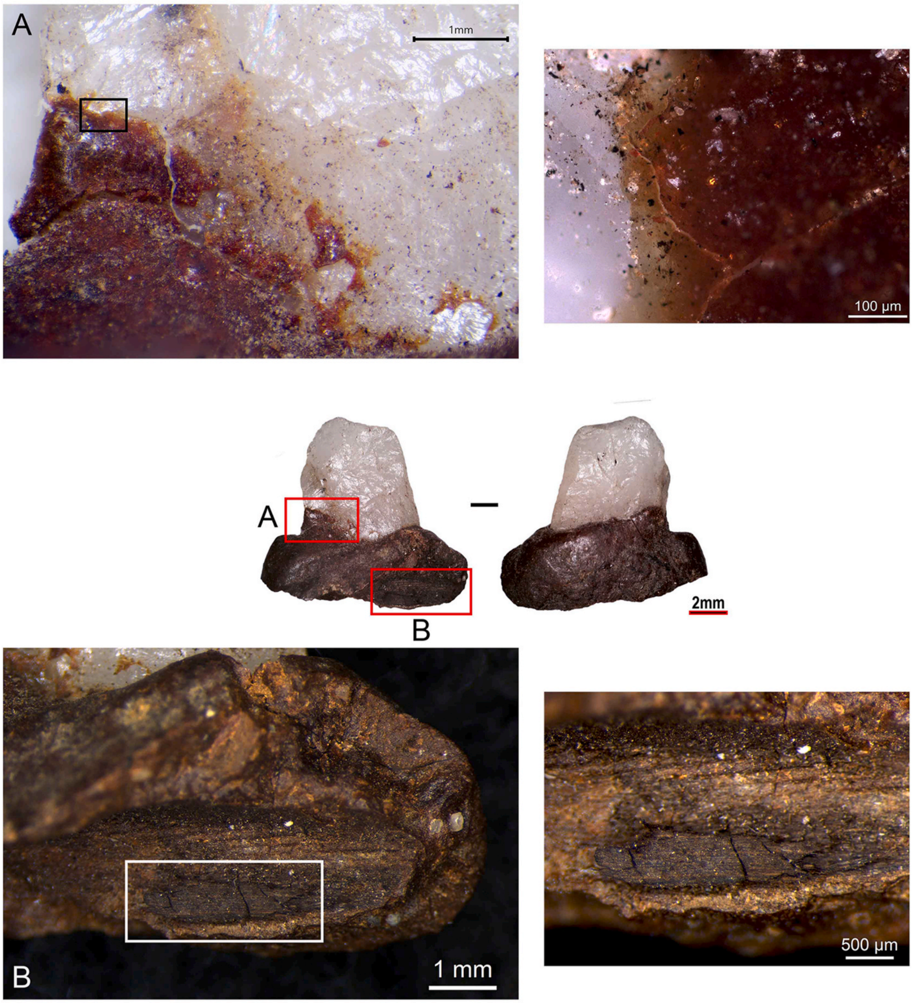

Fig. 7. Quartz microlith set in a large lump of adhesive (SBF16). Wood impressions and wood residues are visible in the inner surface of the adhesive. A) Overview of the residue (30x) and close-up of the residue in cross-polarised light (200x); B) Wood impression and wood residues on the inner surface of the residue (16x) and close-up of a preserved wood fragment from the shaft (32x).

features, we cannot rule out that some of the tools with evidence for contact with animal material were used as lateral barb projections on hunting weapons (de la Peña, et al., 2018; Rots, 2016) and not as hide scrapers. Based on the longitudinal directionality of the micro-traces (abrasion and striations) on these tools, we can however exclude their use as transverse arrowheads. Experimental work has shown that in transverse end-hafted arrowheads, traces are perpendicular to the cutting edge (de la Peña, et al., 2018).

All the scraping tools show clear evidence of resharpening of the working edge. Although end-scrapers are normally subjected to several resharpening episodes during their use-life (Aleo, et al., 2021; Blades, 2003), at Steenbokfontein this may relate to their use as hafted tools. The manufacturing of hafted tools requires more technological investment; therefore, they are often heavily curated and maintained tools (Rots, 2010).

The distribution of residues on most of the analysed scraping tools suggests that they were not inserted in a groove in the haft and then fixed with adhesive. On the contrary, they were likely inserted in an adhesive lump and side-mounted to the handle of a wooden or bone haft with variable inclinations like the specimens from Boomplaas Cave and Plettenberg Bay (see also Porraz and Guillemard, 2019; Deacon and Deacon, 1980). The cutting tools were likely mounted with adhesive to the haft in a parallel lateral position. This hafting method is confirmed by one quartz microlith (SBF16) (Fig. 7). The tool is set in a lump of adhesive with a concave-shaped base. That shape is the result of the adhesive being folded around a wooden shaft, as demonstrated by the presence of wood residues and wood impressions on the inner surface of the adhesive (Fig. 7B). The proximal extremity of the quartz flake does not protrude from the adhesive, confirming that the flake was not in contact or inserted into a socket in the handle.

Two of the three artefacts with the adhesive covered in soil particles and contaminants were subjected to μ-CT scanning. Both are characterised by the presence of bubbles and voids at the interface between the residue and the stone. Bubbles may have formed during the de-hafting process. Based on ethnographic accounts (Sahle, 2019), exhausted or broken hafted tools can be placed near the fire to soften the adhesives and facilitate de-hafting. Small bubbles in the adhesive usually form during this process (Y. Shale, personal communication 2023). Wadley (2010) also reported the formation of air-filled hollows under the adhesive surface when the adhesive is heated too rapidly or placed too close to the fire. The tools were then probably discarded while the adhesive was still malleable and sediment particles adhered to it.

4.2. *Evaluating the effectiveness of analytical methods for adhesive residues analysis*

We used optical microscopy to make the first interpretation of residues on stone tools. Based on their location, distribution, and appearance, we interpreted all residues as potential adhesive remains except in two cases (SBF1 and SBF13). Visually, we identified different groups based on the residues' morphological features (see Table 2). Most residues (N = 22) are distributed between groups 1 and 2, which are visually distinguishable and vary in concentration in the archaeological units. Group 1 residues are more abundant in older layers, and their number gradually decreases in younger layers in favour of group 2 residues. Our initial interpretation was that this is linked to a change in adhesive technology over time. Subsequent spectroscopic and chemical analyses on a sample of tools (N = 13) confirmed our interpretation as adhesive remains. But the GC-MS results disputed all the other inferences made during residue morphological description based mainly on the colour and surface attributes. Two residue samples from group 1 do not preserve archaeological lipids, but the results of the sample from SBF2, which can be assigned to group 1, show that it is molecularly similar to group 2 residues. Therefore, the distinction of residues in groups based on morphological attributes does not reflect a difference in the organic components of adhesive mixtures or technology. Black opaque residues were likely exposed to different depositional environments in the cave that affected their surface qualities and, in some cases, the preservation of organic molecules. These results demonstrate that residue analysis based solely on morphological attributes can form misleading interpretations.

We analysed the inorganic fraction of the adhesive mixture by XRD. XRD proved to be a useful tool for the identification of crystalline additives in the adhesive, such as hematite. Hematite was identified in all the analysed residues but one. μ-CT also provided evidence supporting the use of hematite as an additive corroborating XRD results. The μ-CT allowed us to virtually section the residue on the tools to evaluate the presence and distribution of mineral particles within the adhesive. Iron-rich particles evenly mixed in the adhesive can be seen in SBF14 images of the sectioned residue, suggesting they were intentionally mixed into the adhesive. Similar conclusions can be drawn for the other tools as well.

We applied IR spectroscopy (micro-FTIR and ATR) and GC-MS to identify the organic fraction of the residues. FTIR microspectroscopy effectively identified the organic nature of residues. All the residue samples analysed by micro-FTIR and ATR (N = 7) are of plant origin. The FTIR results suggest a tree resin/tar, but the clade or family of the tree of origin is unclear for most of the samples. The ATR results are clearer than those obtained in reflectance mode (micro-FTIR). The ATR spectra of SBF14 strongly resemble the spectra of various extractives from gymnosperm conifer trees (cf. Tappert et al., 2011), as subsequently confirmed by GC-MS. However, residue identification based on spectroscopy alone is challenging mainly due to limitations posed by degradation, natural or anthropic, of the organic component of the adhesives, absence of extensive reference libraries, and weak/noisy reflectance spectra (Monnier, et al., 2017). Nonetheless, IR spectroscopy is a powerful pre-screening method that allows the selection of promising samples for destructive GC-MS and can help narrow down the range of options for identifying unknown organic residues. The precise identification of organic compounds in the adhesive mixture was achieved with GC-MS. For all the residue samples analysed by GC-MS with preserved residues (N = 6), the primary ingredient of the adhesive comes from tree extractives (resin or tar) of the Cupressaceae and/or Podocarpaceae families. Our results do not clearly point towards heated resin or tar production.

4.3. Adhesive materials and additives at steenbokfontein

The combination of GC-MS, XRD, and μ-CT allowed the identification of at least two different compound adhesive recipes at the site: *Widdringtonia* or *Podocarpus* resin/tar mixed with hematite and *Widdringtonia* or *Podocarpus* resin/tar mixed with a different tree extractive containing pentacyclic terpenoids (*Euphorbia* latex?) and hematite (Table 4). Moreover, micro-FTIR for SBF9 indicates that this residue is a tree extractive, but no mineral additives were detected in the XRD pattern of the measured residue spots. Therefore, it is likely that resin/tar or a mixture of plant extractives were also used as hafting adhesive without mineral additives.

Powdered hematite and another plant exudate were added as additives to the main tackifier to enhance the adhesive's material properties, such as tackiness, elasticity, and plasticity (Langejans, et al., 2022). Ochre is a common ingredient in South African compound adhesives (e.g., Wojcieszak and Wadley, 2018; Lombard, 2007; Gibson et al., 2004), and, as several studies demonstrated, it functions to increase the strength and improve the workability of the adhesive and reduce the curing time and hygroscopicity of the adhesive (Kozowyk, et al., 2016; Zipkin et al., 2014; Wadley, 2005). Based on the GC-MS results, another possible additive could have been animal fat. SBF2, SBF14, SBF23, and SBF27 display an odd-numbered fatty acid ($C_{15:0}$), which is typically associated with ruminant animals (Helwig, et al., 2014; Regert, 2011).

Table 4

Overview of the different adhesive recipes identified at Steenbokfontein Cave considering the tool's raw material and stratigraphic position. CCS: crypto crystalline rock. The dash symbol (–) indicates that the compound was not detected. The slash symbol (/) indicates that the analysis was not performed. SBF5 and SBF15 (in grey) do not contain evidence of organic residues.

ID	Layer	Tool raw material	Organic fraction (ATR, micro-FTIR)	Organic fraction (GC-MS)	Inorganic fraction (XRD)	Use
SBF2	5	Silcrete	/	-*Widdringtonia* or *Podocarpus* sp. resin/tar -Plant exudate containing pentacyclic terpenoids	/	Hide-scraping
SBF4	4b	Quartz	Tree extractive	/	Hematite Fe_2O_3	Cutting medium hard material
SBF5	4b	CCS	/	–	Hematite Fe_2O_3	Hide-scraping
SBF9	4b	Quartz	Tree extractive	/	–	Cutting medium hard material
SBF10	4a	CCS	Tree extractive	/	Magnetite Fe_3O_4	Hide-scraping
SBF14	3b	Quartz	Tree extractive (*Cupressaceae*)	-*Widdringtonia* or *Podocarpus* sp. resin/tar -Plant exudate containing pentacyclic terpenoids	Hematite Fe_2O_3	Cutting medium hard material
SBF15	3b	Quartz	/	–	Hematite Fe_2O_3	Cutting soft material
SBF23	3b	Silcrete	/	*Widdringtonia* or *Podocarpus* sp. resin/tar	Hematite Fe_2O_3	Working soft animal material
SBF17	3a	Quartz	/	-*Widdringtonia* or *Podocarpus* sp. resin/tar -Plant exudate containing pentacyclic terpenoids	Hematite Fe_2O_3	Likely soft plant material
SBF20	3a	Quartz	Tree extractive	*Widdringtonia* or *Podocarpus* sp. resin/tar	Hematite Fe_2O_3	Cutting siliceous plant
SBF21	2	Quartz	Tree extractive (*Cupressaceae*)	/	Hematite Fe_2O_3	Likely siliceous plant
SBF24	1	Quartz	Tree extractive	/	Hematite Fe_2O_3	Hide-scraping (dry?)
SBF27	1	CCS	/	*Widdringtonia* or *Podocarpus* sp. resin/tar	/	Hide-scraping

Although animal fat is sometimes reported as an ingredient of adhesives in South Africa (e.g., Charrié-Duhaut, et al., 2016; Lombard, 2006), this is unlikely to be the case. $C_{15:0}$ is documented in tools displaying wear traces of contact with animal materials, and it is absent in the ones (SBF20 and SBF27) used to work plants. Therefore, the presence of animal fat should be seen as contamination linked to tool use and not as an ingredient intentionally mixed into the adhesive.

4.4. Behavioural aspects and implications linked to adhesive technology

The presence of adhesives in the archaeological record is often seen as a proxy for technological complexity. The manufacturing of a completely new material through the distillation of bark or leaves (tar) or the mixing of several organic and inorganic ingredients (compound adhesives) requires advanced cognitive abilities, considerable technical skills, control of fire, and an understanding of material proprieties (Schmidt, et al., 2022; Niekus et al., 2019; Wadley et al., 2009). Adhesives can have a wide range of uses, and adhesive mixtures can be altered by adding and manipulating ingredients to achieve various desired products suitable for different applications. At Elands Bay Cave, for instance, animal fat was likely added to the adhesive mixture used to seal the perforation of an ostrich eggshell flask, but it is absent on the two other samples of hafting adhesives (Charrié-Duhaut, et al., 2016). The deliberate addition of animal fat here likely acted on the proprieties of the adhesive to accommodate a different type of use. Therefore, characterising adhesive traditions and documenting the variations in adhesive recipes and uses is fundamental for discussing: i) the level of understanding of natural material properties of prehistoric populations, ii) their level of technical expertise, and iii) technological flexibility and technological innovations. This is particularly relevant when considering adhesives dated to the Middle Pleistocene and the role played by this technology in the ongoing debate about the cognitive abilities of Neandertals and early modern humans.

4.4.1. Recipes in relation to tool materials and function

At Steenbokfontein, we identified several multi-component adhesives made of resin/tar and mixed with powdered hematite and, in some cases, another plant exudate. Adhesives were primarily used at this site for mounting stone tools to handles, with the only exception of a cigar-shaped mastic tool with a still unclear function (Jerardino, 2001). It has been argued that the variability in adhesive types may be influenced by the object's raw material or use. At Sibudu for instance, ochre stains are common on segments made of dolerite and hornfels and are notably less frequent on quartz and crystal-quartz segments (Lombard, 2007). According to Lombard (2007), this discrepancy relates to differences in the raw material, such as roughness, grain size, and porosity. Experimental work also showed that glue performance varies according to different substrates (Tydgadt and Rots, 2022); therefore, it is conceivable that the composition of mixtures varied according to the object's raw material. Furthermore, brittle adhesives may have been preferred for certain activities, e.g., shooting, while robust adhesives were preferred for repetitive tasks, e.g., scraping and cutting, suggesting that the selection of adhesives was task-oriented (Wadley, et al., 2009). However, in our sample, coniferous ochre-loaded adhesives were used both on quartz and non-quartz tools, and these tools were used for diverse activities, showing no correlations between tasks and adhesive recipes.

4.4.2. Diachronic view on recipes

This consistent adhesive recipe is also observed diachronically at Steenbokfontein Cave despite important changes in subsistence and settlement patterns in the Western Cape since about 3500 BP. Settlements shifted from caves and shelters to large open-air sites along the coast, mobility drastically decreased and was limited to the costal margin and foreland, and the diet became more marine oriented soon after about 3200 BP (Jerardino, et al., 2013). The changes in mobility in Steenbokfontein Cave and other local sites are reflected in the frequency of exotic raw materials, which decreases after the deposition of layer 4a (2σ: 3990–3245 cal BP) in favour of local ones (Jerardino, 2013). However, the observed changes in mobility did not affect the procurement of the primary ingredient for adhesive production.

It is also important to observe that while conifer resins/tar is consistently exploited throughout the sequence, the use of additives seems more flexible and less standardised than previously argued. A recent ethnographic work (Fajardo, et al., 2024) showed that traditional adhesives exhibit adaptability in materials, productions, and behaviours. Ingredients can be replaced, left out, and mixed based on their availability (Fajardo, et al., 2024). The same flexibility in adhesive technology also emerges from the analysis of Steenbokfontein Cave adhesives. Despite the near consistent use of coniferous resin or tar as the main component of their mixtures as well as ochre, prehistoric glue makers mixed and substituted additional ingredients to achieve the final product. This variation shows that adhesives were not material-specific or task-oriented, but were likely made of what was available. Another potential explanation for the variability in recipes is that the different adhesive types were produced by different makers or groups who inhabited the cave with slightly different adhesive traditions. This hypothesis could find support from previous interpretations of Steenbokfontein Cave as an aggregation place for different human groups since the mid-Holocene (Jerardino, et al., 2013).

The continuity of the use of coniferous resin adhesives in South Africa is documented in different sites from the west to the east coast and dates to at least ~65000 years ago (Soriano, et al., 2015). Charcoal and pollen of *Podocarpus/Afrocarpus* trees are documented in Elands Bay Cave (Cartwright, et al., 2016), Diepkloof Rock Shelter (Cartwright, 2013), Sibudu Cave (Zwane and Bamford, 2021), and Border Cave area (Scott, et al., 2023) indicating the availability of these species in the environment. Furthermore, the abundance of *Podocarpus* sp. charcoal in some archaeological layers or sites where adhesives were found has been seen in relation to its role in adhesive production (Cartwright, et al., 2016). Despite the availability of other natural sources of adhesives (see Schmidt et al., 2022), most molecular studies of South African Stone Age adhesives indicate the use of an extractive of the *Podocarpus* genus. The preferential use of *Podocarpus* for adhesives by hunter-gatherers groups is seen as a long lasting tradition and adaptation that transcends changes in technology and has been compared to the preferential exploitation of birch tar in western Europe (Charrié-Duhaut, et al., 2016, p. 302). In a recent study, Schmidt et al. (2022) argued that *Podocarpus* tar was preferentially selected over other substances, including *Widdringtonia* resin, for its superior mechanical proprieties, similar to birch bark tar (Kozowyk and Poulis, 2019). While this may be the case, it is for now impossible to molecularly distinguish between *Widdringtonia* and *Podocarpus* extractives. In this regard, modern botanical and archaeobotanical evidence may help narrow down the potential sources of adhesives. In the case of Steenbokfontein, *Podocarpus elongatus* is found today within a ~12 km radius of the cave (Lombard, 2023) and is present in the archaeological deposits of nearby Diepkloof and Elands Bay Cave (Cartwright et al., 2016; Cartwright 2013). Therefore, it was locally available in the Steenbokfontein landscape. On the other hand, *Widdringtonia cedarbergensis* currently grows within a ~40 km radius of the cave (Lombard, 2023). Based on this evidence, it is possible that *Podocarpus* was the source of adhesive at Steenbokfontein although based on the molecular signature of our sample, we cannot rule out *Widdringtonia*. Also, importantly, experiments show significant differential preservation of natural adhesives and particularly between tars and exudates (resins and gums) (Kozowyk, et al., 2020), so it is possible that additional materials were used, but their signature is not preserved.

The systematic application of optical microscopy, spectroscopy, and chemical analysis on a relatively large sample of tools with adhesive residues from all the stratigraphic units allowed us to characterise adhesive technology at Steenbokfontein Cave. Adhesive recipes and use transcend changes in technology, raw materials exploitation, and also subsistence on the west coast of South Africa since the mid-Holocene.

Throughout the entire sequence, adhesive technology involved the systematic exploitation of conifer resin/tar combined with organic and inorganic additives. However, its use was part of a flexible strategy in which different ingredients were mixed to achieve the final product, independent of the tool raw material and function. Such behaviour is difficult to document at other LSA sites where (chemical) analyses were mostly performed on a single or a few objects among those of interest (e.g., Charrié-Duhaut, et al., 2013). For now, this limits the ability to discuss diachronic and regional trends in adhesive production and use and compare between sites. When viewed in light of the wider history of adhesives, applying a multi-disciplinary approach to studying Middle Pleistocene adhesive residues can help capture nuances in adhesive technology. Recent studies on adhesives used by Neanderthals revealed that they used and combined various organic and inorganic sources (Schmidt, et al., 2024; Degano et al., 2019) to haft different tool types, suggesting the flexibility of their adhesive technology in terms of resources exploited and additives. However, most of the studies focusing on adhesives dating to the Middle Pleistocene lack a comprehensive analytical methodology encompassing use-wear analysis, experiments, and molecular characterization of organic and inorganic adhesive ingredients. Hopefully, in the future, the re-examination of old collections and the systematic application of molecular and spectrographic techniques for residue identification will allow a better understanding of adhesive technology in the deep past.

5. Conclusions

We presented the results of a multidisciplinary study on 30 LSA tools with adhesive residues from Steenbokfontein Cave, South Africa. We combined optical microscopy, μ-CT and XRD, IR spectroscopy, and chemical methods for use-wear and residue molecular identification to reconstruct the use-life of these tools and their residues and explore adhesive use at the site. Use-wear analysis shows that the tools were used as hafted scrapers for hide-working and as elements of composite tools for cutting animal and plant matters, although other functions, such as use as barb-projections, cannot be excluded. Tools were side mounted on the shaft using compound adhesives made of extractives (resin or tar) of *Widdringtonia* or *Podocarpaceae* species and powdered hematite or by adding a third unidentified plant ingredient, possibly *Euphorbia* latex. There is also an indication of a third recipe that did not include mineral additives. Throughout the whole sequence, conifer resin/tar is exploited as the main adhesive material, while organic and inorganic additives were added or left out without any observable correlation to the tool's raw material and function. Moreover, we did not observe a chronological trend in the use of the different mixtures. From our results emerge that adhesive technology at Steenbokfontein was flexible in terms of additives and ingredients.

The application of methods for molecular identification of Steenbokfontein Cave residues, in combination with microscopic observation and morphological description, led to a better reconstruction of artefacts use-life and increased our knowledge and understanding of adhesive technology during the Later Stone Age. Our detailed analysis enables us to discuss adhesive technology in relation to tool's function and highlight the absence of diachronic changes in the production and use of compound adhesives at the site. Nonetheless, we documented a flexible use of adhesive ingredients that challenged previous interpretations of glue recipes designed according to the tool's material proprieties and use. The systematic application of this approach to other South African archaeological assemblages will allow us to better understand past adhesive technologies and pinpoint the continuity or breaks of traditions in the manufacturing and use of adhesives. Similarly, applying this approach to Pleistocene adhesives ought to generate new insights into different adhesive traditions and allow us to evaluate possible technological flexibility and variability in the deep past and discuss it in light of the technological and cognitive abilities of different human populations.

Disclosure statement

No potential conflict of interest was reported by the authors.

CRediT authorship contribution statement

Alessandro Aleo: Writing – review & editing, Writing – original draft, Visualization, Investigation, Formal analysis, Data curation, Conceptualization. **Antonieta Jerardino:** Writing – review & editing, Investigation, Conceptualization. **Rivka Chasan:** Writing – review & editing, Visualization, Investigation, Formal analysis. **Myrto Despotopoulou:** Writing – review & editing, Formal analysis. **Dominique J.M. Ngan-Tillard:** Writing – review & editing, Formal analysis. **Ruud W.A. Hendrikx:** Writing – review & editing, Formal analysis. **Geeske H.J. Langejans:** Writing – review & editing, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This project has received funding from the European Research Council (ERC) under the European Union's Horizon 2020 Research and Innovation Programme, grant agreement number 804151). The funders had no role in study design, data collection and analysis, decision to publish, or preparation of the manuscript.

The export of the material was permitted by the South African Heritage Resources Agency under permit number 14109 while the sampling permit was granted by the Heritage Western Cape under reference number 20012303AS0310E. We would like to thank Deano Stynder and Louisa Hutten (University of Cape Town) for helping with the export permit and assistance during the fieldwork in Cape Town. We also thank Annelou van Gijn and Annemieke Verbaas (Leiden University) for helping with the interpretation of use-wear traces on the archaeological tools, Liliana Baron (TU Delft) for assistance during the GC-MS analysis, and Paul Kozowyk (TU Delft) for assistance in interpreting FTIR and ATR data. We thank the Laboratory for Material Cultural Studies (Leiden University) and the Inorganic Systems Engineering Laboratory (TU Delft) for generous use of lab space and equipment.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jas.2024.105997.

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