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The way we wear makes the difference: residue analysis applied to Mesolithic personal ornaments from Hohlenstein-Stadel (Germany)

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Abstract

Research conducted by Newell et al. (1990) has led to propose that during the Mesolithic, the Upper Danube region was inhabited by a “Fish Teeth Band”, characterized by the use of carp fish teeth and exogenous fossil shells as ornaments. However, technological data on these personal ornaments drastically lacked and especially for the fish teeth associated to the Mesolithic burial of Hohlenstein-Stadel that remained completely undescribed until now. The aim of this paper is to establish how the carp teeth from Hohlenstein-Stadel were modified and worn before being deposited in the burial. High-resolution microscopic analysis identifies usewear traces and a red compound adhering to the surface of the teeth. Structural and elemental analysis of the residue combining light and scanning electron microscopy, infrared spectroscopy, and Raman analysis identify a suspension technique that uses an adhesive composed of an organic binder mixed with charcoal and minerals including hematite, dolomite, and quartz. Ethnological inquiry reveals that this system of suspension, commonly used in traditional societies, is for the first time documented, at Hohlenstein-Stadel, in an archaeological context.

Keywords Cyprinid teeth ornaments . Hohlenstein-Stadel . Pigment . Adhesive . Microscopy . Raman . EDX

Introduction

Research on Mesolithic ornaments has focused on the identification of trade networks through the sourcing of raw material (Cristiani and Borić 2012; Eriksen 2002; Komšo and Vukosavljević 2011; Rähle 1978), regional differences in bead type associations interpreted as possible reflection of cultural boundaries (Newell et al. 1990; Vanhaeren and d'Errico 2006; Rigaud et al. 2010; Rigaud 2011), and analysis of ornaments used as grave goods to assess social stratification (Albrethsen and Brinch-Petersen 1976; Larsson 2006; O'Shea and Zvebil 1984; Rigaud et al. 2010). To provide valuable insights, these studies have to rest on precise economic and technological data to identify behavior involved in the acquisition and transformation of the raw material for beads manufacture (White 2008; d'Errico and Vanhaeren 2002; Vanhaeren and d'Errico 2003; 2005; Bonnardin 2009; d'Errico and Rigaud 2011). With this aim in mind, the present study focuses on a region of Europe, the Danube Valley, that features a copious record of Mesolithic personal ornaments. Research conducted by Newell et al. (1990) has led to propose that during the Mesolithic, the Upper Danube region was inhabited by a “Fish Teeth Band”, characterized by the use of carp fish teeth associated to exogenous fossil shells as ornaments. The only publications describing the fish teeth ornaments from the Upper Danube area included low-resolution photographs of the fish teeth from the Mesolithic camp site of Burghöhle Dietfurt showing a lateral drilled perforation (Taute 1990, Fig. 17), and a recent photo of eleven teeth from the Mesolithic burial site of Hohlenstein-Stadel, published in the catalogue of the Ulmer Museum. In this picture, no modification such as perforations, notches or incisions, are visible on the teeth.

The publication of the grave goods associated to two Mesolithic burials from Vlasac, Serbia, in the Iron Gates region, South Danube, reveals the presence of carp teeth used as ornaments. In their study, Cristiani and Borić record the presence of red pigment staining and residues, tentatively interpreted respectively as glue and remnants of skin or tendons (Cristiani and Borić 2012). These observations corroborate the hypothesis proposed by

Radovanovic (1996: 214) according to which carp teeth may have been attached (appliqués) to the clothes of the deceased. However, no physico-chemical analyses were conducted to verify this hypothesis.

Characterization of microwear patterns and microscopic residues on archaeological stone tools is used as a means to identify worked raw material (Hardy and Moncel 2011; Mercader et al. 2008), tool function (Hardy et al. 2008) and hafting technology (Dinnis et al. 2009; Lombard and Pargeter 2008; Rots et al. 2011; Wadley et al. 2009) of past societies. In spite of the numerous studies conducted on personal ornaments, such methods were rarely used to identify systems of suspension and evaluate the behavioral complexity behind the beadwork manufacture.

Here, we apply an array of analytical techniques to analyze the residues attached to the carp teeth associated to the Mesolithic head burials of Hohlenstein-Stadel, Upper Danube, and identify the main constituents of the adhesive compound used to attach the teeth.

Archaeological context

The Hohlenstein-Stadel cave is located on the south edge of the Lone valley close to the village of Niederstotzingen (Baden-Württemberg, Germany) (Fig. 1). The site consists of a 70-m long gallery that opens to the North in the Hohlenstein cliff within the calcareous massifs of the Swabian Jura (Fig. 1).

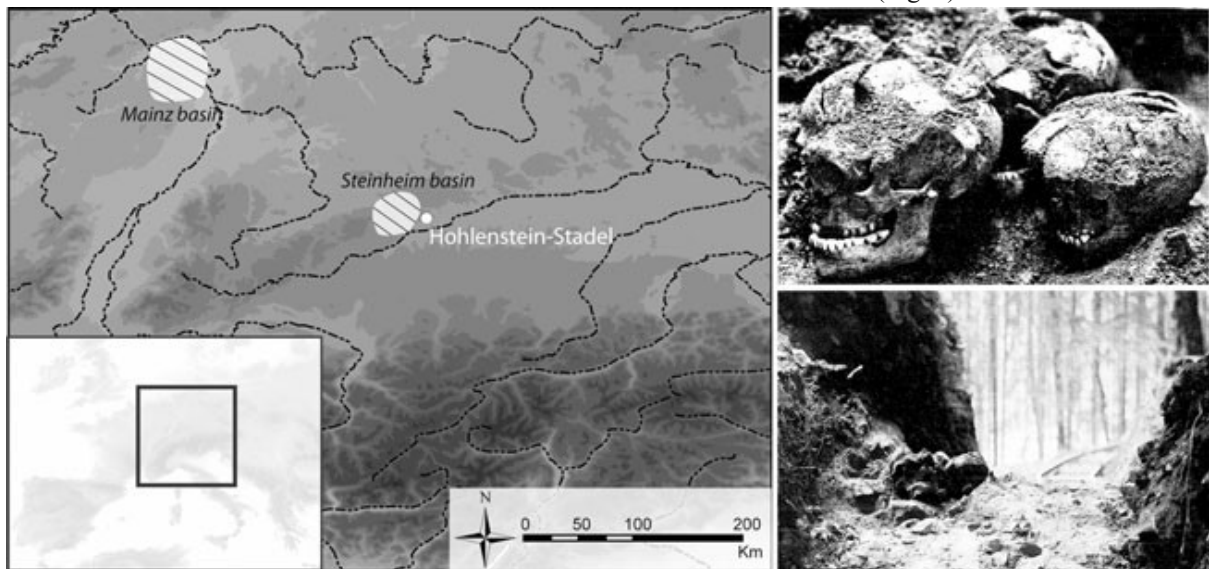


Fig. 1 left Location of Hohlenstein-Stadel archaeological site; right photographs of the skulls at the moment of their discovery (after Wetzel 1938)

The cave was excavated in 1935, and between 1937 and 1939. In 1998, new excavations, directed by Bolus, Conard, and Kandel, focused on the entrance and a small sector inside the cave (Bolus et al. 1999; Kind and Beutelspacher 2009; Wetzel 1938). A new ongoing excavation, directed by Kind (Landesamt für Denkmalpflege Baden-Württemberg im

Regierungspräsidium Stuttgart), started in 2008, has identified Middle Palaeolithic, Aurignacian, Magdalenian and Final Upper Palaeolithic levels. Remains attributed to the Mesolithic, the Neolithic, the Bronze and the Iron Age were recovered at the top of the stratigraphic sequence, considered as disturbed. In 1938, Wetzel and Völzing discovered three human skulls within a large pit at the entrance of the cave (Wetzel 1938). A conventional radiocarbon date of $7,835 \pm 80$ BP (ETH-5732; $7,029$ – $6,499$ cal. BC, 2σ , IntCal 09; Haas 1991) obtained from one of the skulls, attributes the human remains to the Late Mesolithic.

The pit was 70 cm deep, cut into the Aurignacian layers, and presented an oval shape with a north–south longitudinal axis and a diameter varying between 35 and 45 cm. From the top to the bottom (Fig. 2), the sediment filling of the pit consisted of 12 cm of yellow loess including small angular fragments of the cave wall, brown-yellow silt with large cave wall fragments, and red-brown silt close to the bottom of the pit (Völzing 1938; Wetzel 1938).

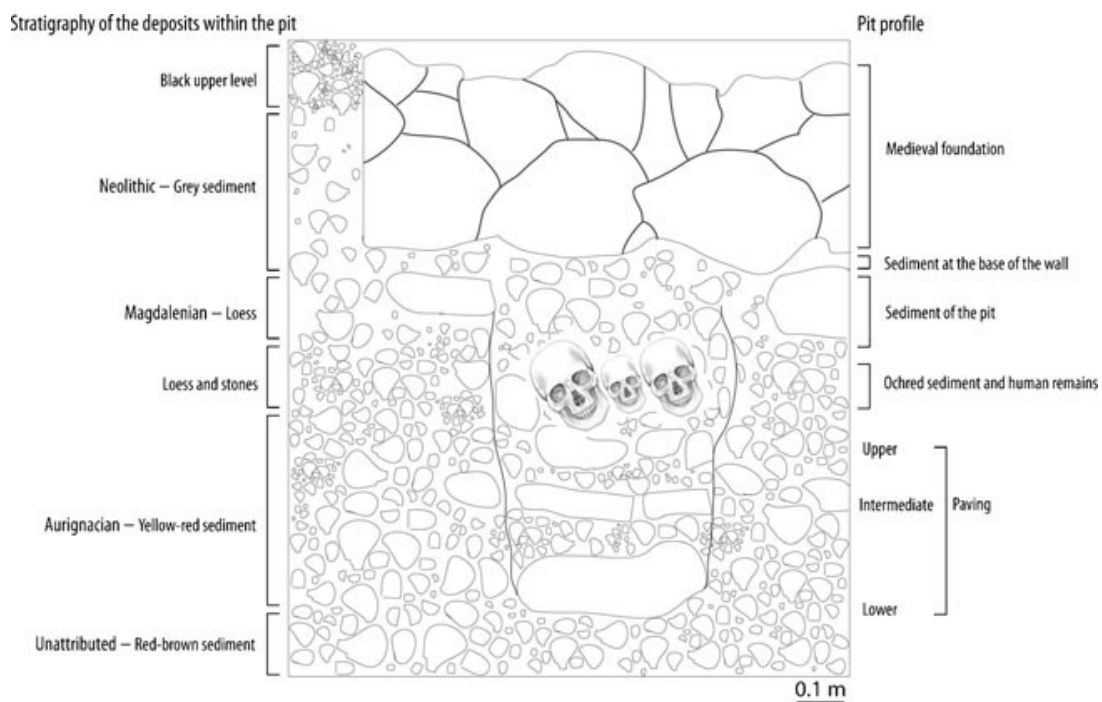


Fig. 2 Drawing and stratigraphy of the head burial pit at Hohlenstein-Stadel (modified after Wetzel 1938)

The human remains were found on top of a succession of flat stones lying on a single intensely ochred larger stone at the bottom of the pit. The human remains consisted of three human skulls each with the mandible, and two or three first cervical vertebrae for each skull (Völzing 1938; Wetzel 1938). Skull faces were oriented to the Southwest (Fig. 1). On the basis of the skull sutures, degree of eruption of the teeth, combined with their wear and some morphological criteria, two skulls were attributed to adults, a man and a woman, the third to a juvenile (Gieseler 1951; Orschiedt 1998, 1999).

Anatomical association of mandibles, cervical vertebrae and crania, and the presence of cut-marks on vertebrae indicate that the skulls were removed from the bodies and deposited in the pit a short time after the death of the three individuals (Gieseler 1951; Orschiedt 1998, 1999). Chopping traumas recorded on the parietal bones of the three skulls suggest that the individuals were injured by violent blows on the head (Gieseler 1951; Orschiedt 1998, 1999). The similarity of the Hohlenstein-Stadel skulls burial deposit with the head burials of Große Ofnet in Bavaria (Schmidt 1912) lead various authors to interpret the deposit as a cult site resulting from intergroup conflicts or as an accumulation of trophy skulls (Orschiedt 1998, 1999; Roksandic 2006; Thorpe 2003).

The only grave goods at Hohlenstein-Stadel consist of 12 fish teeth recovered in the sediment surrounding the woman's skull (Völzing 1938; Wetzel 1938). They were attributed to the *Rutilus frisii meidingeri* (Heckel 1851) cyprinids (Rauther 1935; Taute 1990) and interpreted as personal ornaments (Newell et al. 1990; Rähle 1978). Ten pharyngeal Cyprinid teeth of the Hohlenstein-Stadel head burial (Fig. 3) are on display at the Ulmer Museum. Their number differs from the previously published counts and indicates that two teeth were lost since the excavation conducted by Wetzel and Völzing (Wetzel 1938).

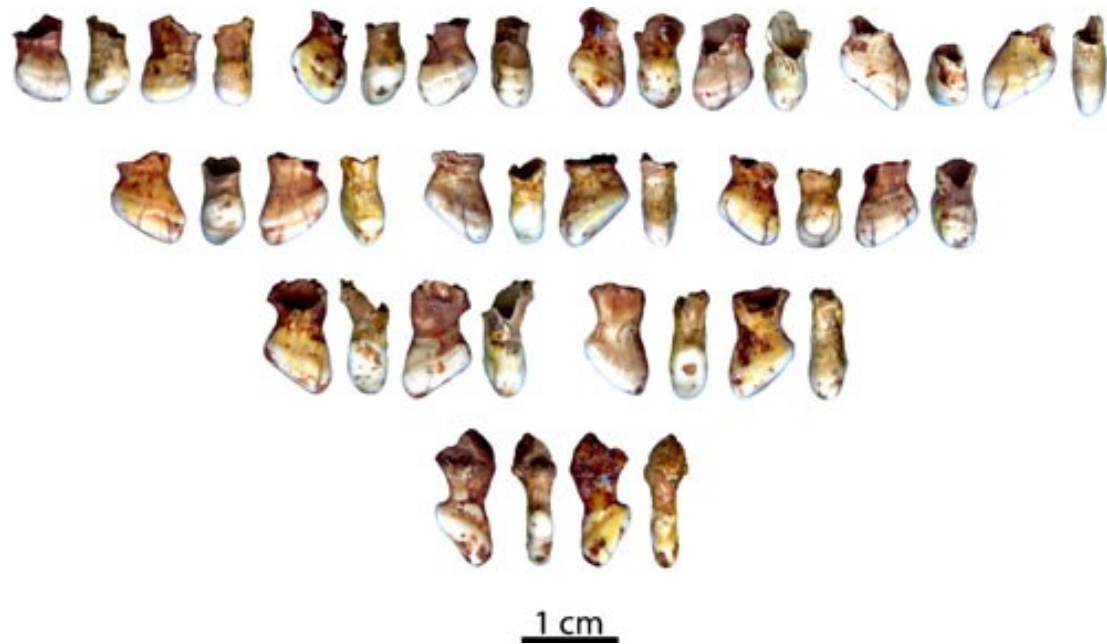


Fig. 3 Four aspects of the ten cyprinid pharyngeal teeth from the Hohlenstein-Stadel head burial. Numbers correspond to those in Table 1 and Fig. 6

A microscope Motic SMZ-168 equipped with a digital camera Jenoptik ProgRes-CT3 was used to record taphonomic and anthropogenic modifications on the surface of the teeth, including location of use-wear and presence of a pigmented compound.

The FishBase (<http://www.fishbase.org/home.htm>) was used to identify the current nomenclature of the fish species.

Two fragments of pigmented compound adhering to the root of two teeth, 1 mm² in size were sampled for elemental and mineralogical analyses. Microscopic observation has revealed that the bony ankylosis of the teeth, where the pigmented compound was present, were consolidated with varnish. To avoid contamination, the sampling of the compound was conducted under the microscope in area where no varnish was present.

One sample was analyzed with a confocal Raman microspectrometer SENTERRA (Bruker Optics, Ettlingen, Germany) equipped with a 532 nm exciting line (0.02–20 mW). Spectra were obtained on mineral grains between 100 and 1,555 cm⁻¹ with a resolution between 3 and 5 cm⁻¹ and were automatically compared with Ruff database (Downs 2006). The other sample was submitted to Energy Dispersive X-ray analysis (EDX) performed using a Quanta 200 scanning electron microscope (SEM). Images were obtained by secondary electron (SE) and back-scattered electron detectors (BSE). Micro-FTIR spectra from one compound fragment of compound were acquired using a Nicolet 6700 FTIR spectrometer (Thermo Scientific) coupled to a Nicolet Nic-Plan infrared microscope equipped with a mercury cadmium telluride (MCT) detector. Sample areas of 200 μm diameter were analyzed in reflectance mode using a 15× Replachromat Cassegrain objective (Spectra-Tech). Spectra were acquired at a spectral resolution of 4 cm⁻¹ and by averaging 200 scans.

Results

The Hohlenstein-Stadel cyprinid teeth

Identification

The current valid name of the fish species from which the teeth were extracted is *Rutilus meidingeri*, (Heckel 1851). Until recently, the species existed in the entire region of the Danube, the lakes of Bavaria and Austria and

the western part of the Caspian Sea, as well as in the Black Sea, with the exception of its southern part (Hensel 1979; Mikschi and Wolfram-Wias 1999). It disappeared from German waters about 60 years ago (Freyhof 2002).

Pharyngeal fish teeth are located behind the pharynx (Fig. 4a) (Zheltenkova 1949). They are involved in the seizing, masticating and swallowing of food. For cyprinids, the teeth are arranged in a single row of five to six teeth on each of the two pharyngeal bones (Fig. 4b, Chebotareva et al. 2009; Kottelat and Freyhof 2007). Teeth are ankylosed to the jaw and are formed of a bony neck, a crown and a main cusp (Fig. 4c).

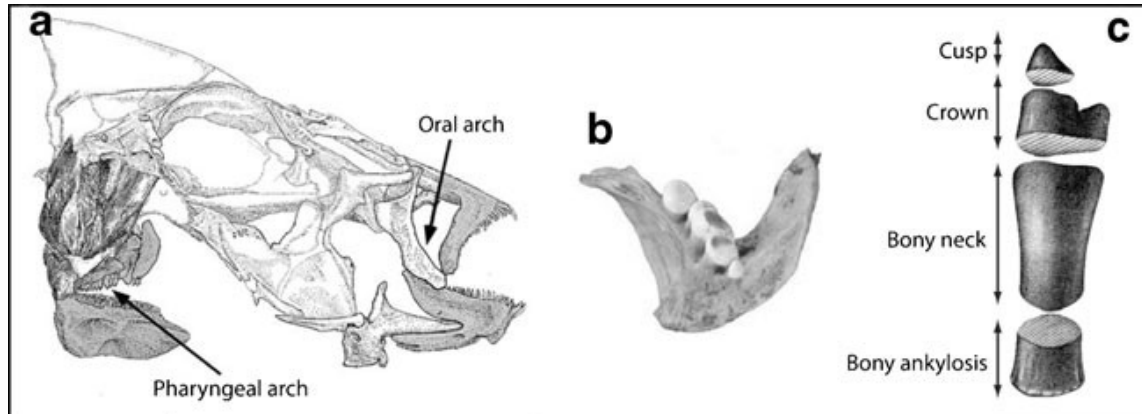


Fig. 4 Anatomy of cyprinid head bone (a), hemi pharyngeal bone (b) and teeth (c) (a, after Fraser et al. 2009, b from <http://fl.biology.usgs.gov>, c modified after Barel et al. 1976)

The Hohlenstein-Stadel fish teeth include six teeth from a right pharyngeal bone and three teeth from a left pharyngeal bone. One tooth cannot be lateralized because of fresh breakages (Table 1).

No.	Length (mm)	Width (mm)	Recent breakage	Cracks	Fractured bony neck	Use-wear	Pigmented compound	Sampled
1	9.92	6.64	-	-	+	+	+	-
2	10.37	7.81	-	+	+	+	+	-
3	12.05	8.1	-	+	+	+	+	-
4	10.19	9.89	-	+	+	+	+	-
5	10.69	9.34	-	+	-	+	+	-
6	10.29	8.81	-	+	-	+	+	+
7	11.69	8.75	-	+	+	+	+	-
8	14.36	9.34	+	+	+	+	+	+
9	12.86	9.23	-	-	-	+	+	-
10	16.04	8.73	-	-	+	+	+	-

Table 1 Morphometric, taphonomic, and archaeological data recorded on the cyprinids pharyngeal teeth from the head burial of Hohlenstein-Stadel. + presence, - absence.

Preservation

The teeth are well preserved. Seven specimens present longitudinal cracks and one shows a recent breakage on the bony ankylosis and the neck. A layer of varnish partially covers their surface in places, which hampers the recording of anthropogenic modifications for those areas.

Technological and use-wear analyses

No anthropogenic modifications affecting the shape of the fish teeth, such as perforations or grooves, were observed. Irregular fractures on the bony ankylosis were registered on seven out of ten teeth (Table 1).

Use-wear was recorded on all specimens (Fig. 5c, f, o, r, v). Located around the bony ankylosis, it consists of a clear smoothing of the natural features associated with an intense shine.

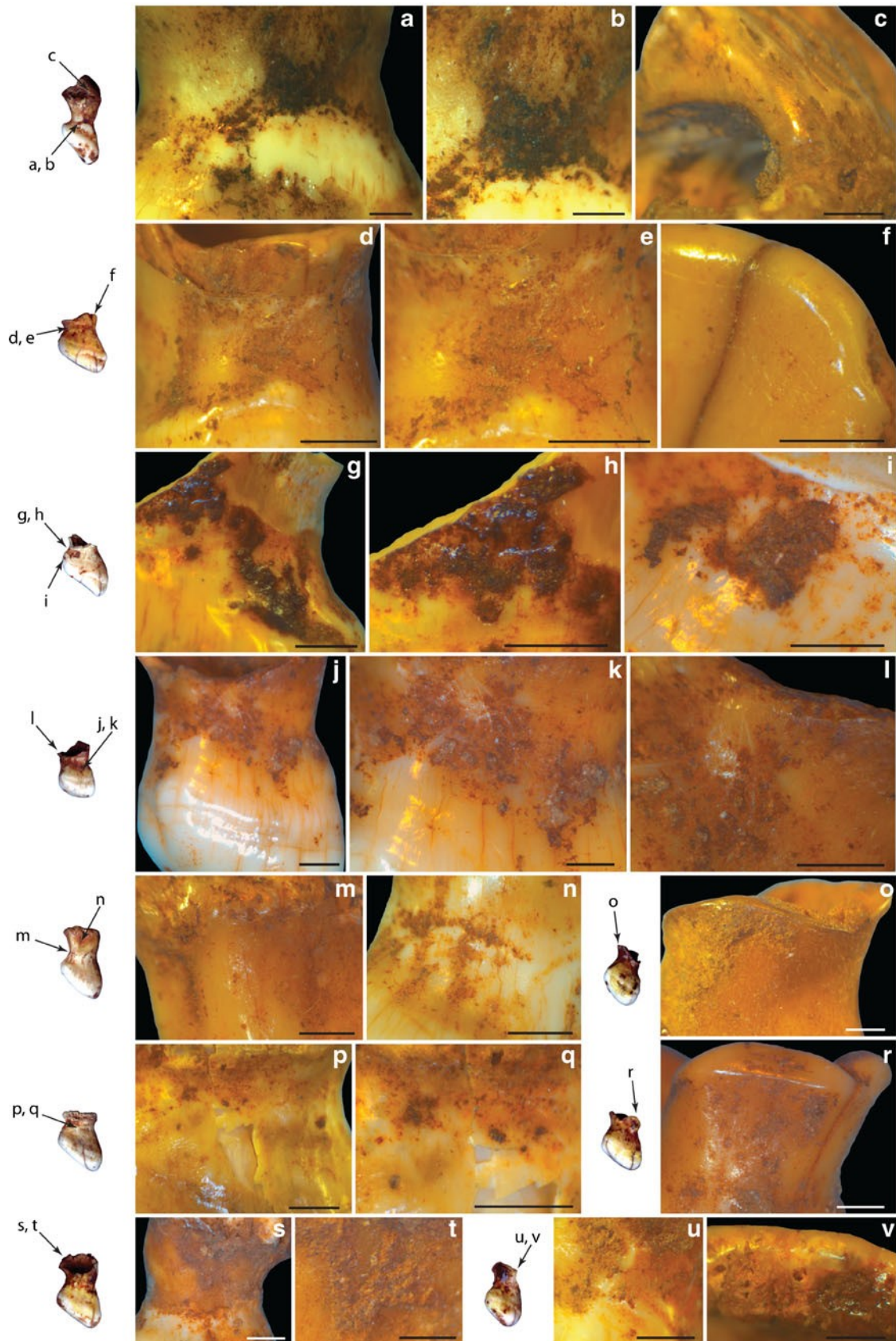


Fig. 5 Residues of a red compound (a, b, d, e, j, k, m, n, p, q, s, t, u) and traces of use-wear (c, f, l, o, v) present on the cyprinids' pharyngeal teeth from the head burial of Hohlenstein-Stadel (scale 1 mm)

Red compound

A hard half-millimeter-thick residue of a red compound, firmly adhering to the tooth and concentrated around the neck, were found on all specimens (Fig. 6). Cyprinids teeth naturally show a natural internal hollow cavity extended from the bony neck to the cusp. Absence of sediment in the cavity is probably due to cleaning during excavation or restoration.

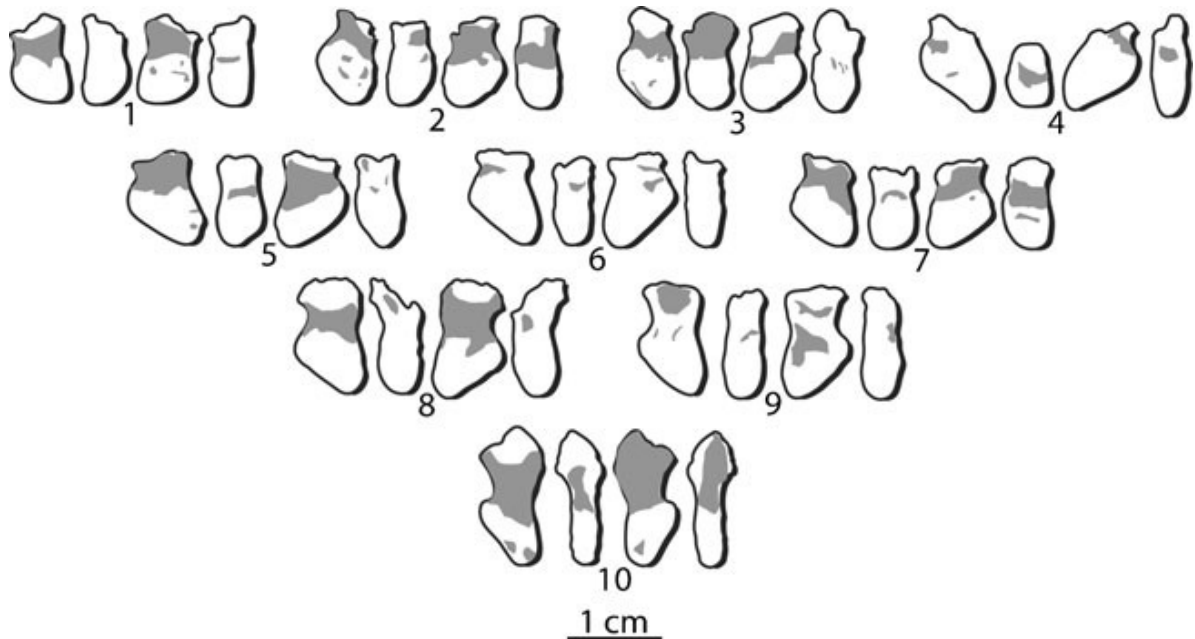


Fig. 6 Location of the red deposit (in gray) on the teeth recovered at the Mesolithic site of Hohlenstein-Stadel

Optical and SEM analyses of this residue reveal a heterogeneous texture composed of a porous matrix made of coarse angular red, white, and black grains cemented by thin red powder (Fig. 7).

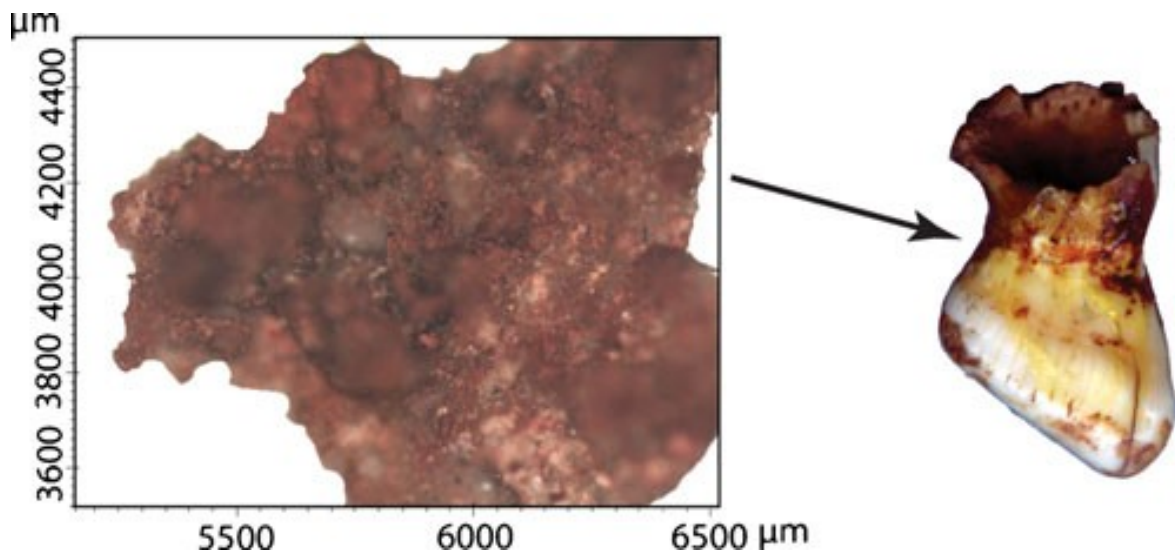


Fig. 7 Photo of the residue sampled on the teeth no. 8

Micro-Raman analysis of the sample from tooth no. 8 (Fig. 3, Table 1) identifies the red grains as hematite (Fe_2O_3), the white grains as dolomite ($\text{CaMg}(\text{CO}_3)_2$), and the black grains as charcoal (Fig. 8). Analysis of the thin powder resulted in intense fluorescence hampering the Raman signal.

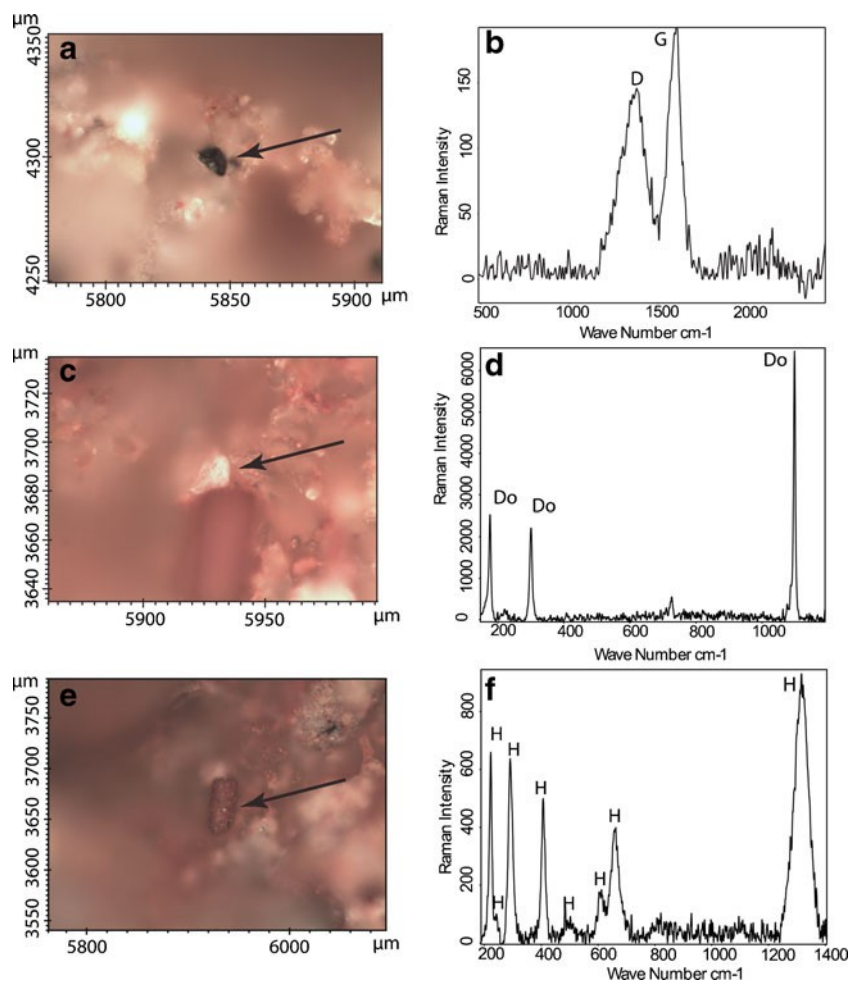


Fig. 8 Close-up views (a, c, e) and Raman spectra (b, d, f) of the grains included in the pigmented compound adhering to a Mesolithic cyprinidss tooth no. 8 from Hohlenstein-Stadel. The black grain (a) corresponds to a charcoal fragment as indicated by the G graphite band and the D amorphous carbon band (b); the white grain (c) to dolomite as shown by the Do bands (d); and the red grain (e) to hematite identified by the H bands (f) (reference spectra after Downs 2006 and Tuinstra and Koenig 1970)

SEM analysis of the pigmented residue adhering to Hohlenstein-Stadel teeth no. 6 (Fig. 3, Table 1) reveals that when observed in BSE mode, the compound is composed of middle atomic weight and small spots of high atomic weight elements. EDX elemental analysis of the matrix identifies silicon, calcium, carbon, iron, aluminum and potassium in decreasing proportions (Figs. 9 and 10).

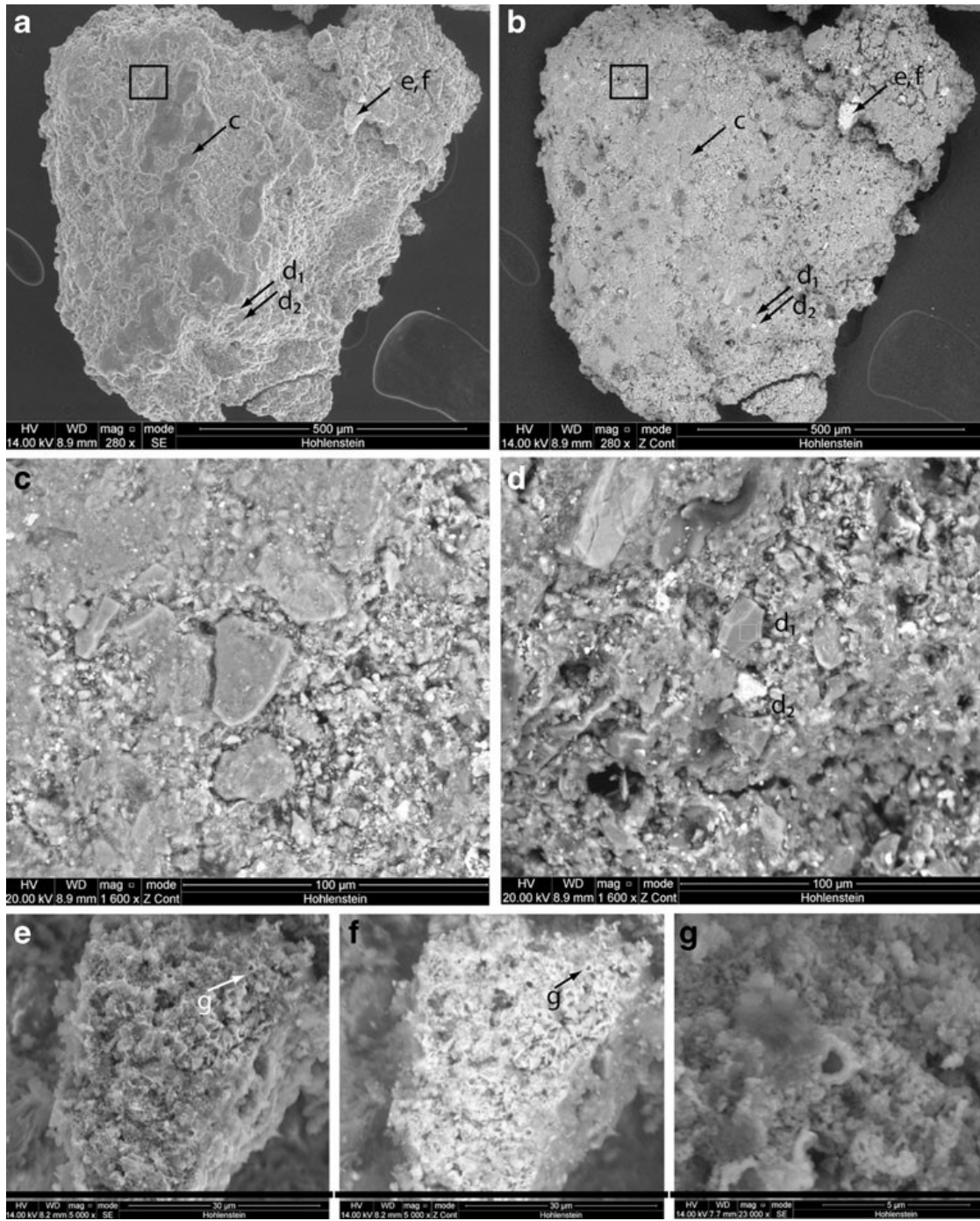


Fig. 9 SEM micrograph in SE mode (a, e, g) and BSE mode (b, c, d, f) of a sample of the pigmented compound attached to the Hohlenstein-Stadel fish tooth no. 6

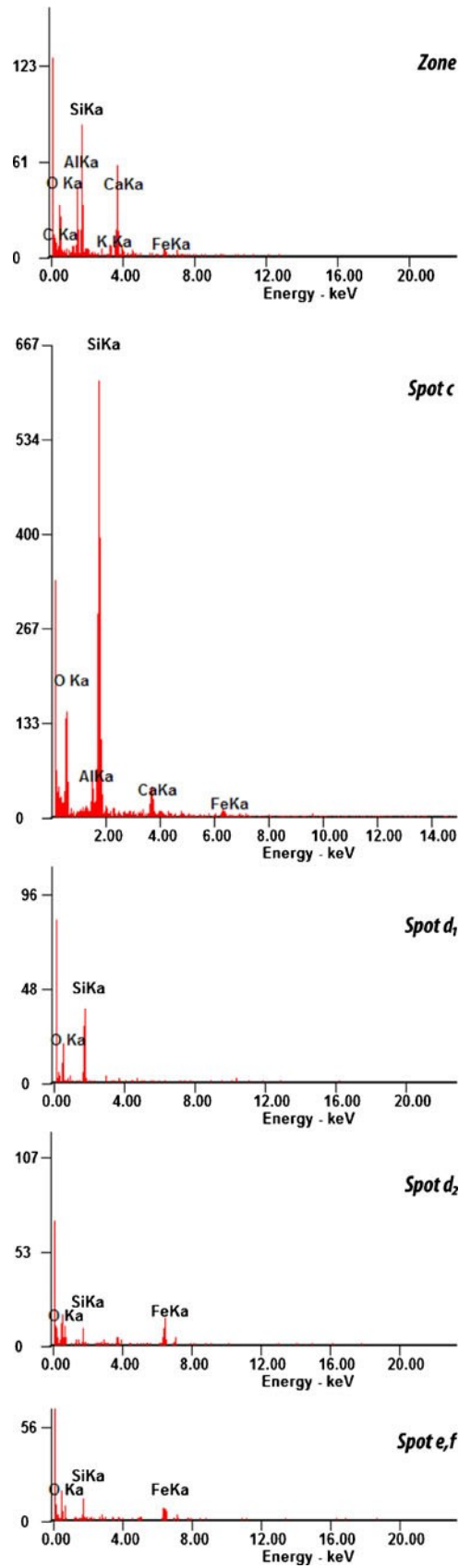


Fig. 10 EDX analyses of selected spots on the residue from Hohlenstein-Stadel tooth no. 6

Presence of organic material in the residue is evidenced by FTIR spectroscopy analysis, which identifies C–H and O–H bands, clearly visible in its domain of absorption (3,200–3,700 cm^{-1}). However, inorganic and organic absorption bands overlap in the first part of the spectrum, preventing a more accurate identification of the organic component (Fig. 11, Table 2).

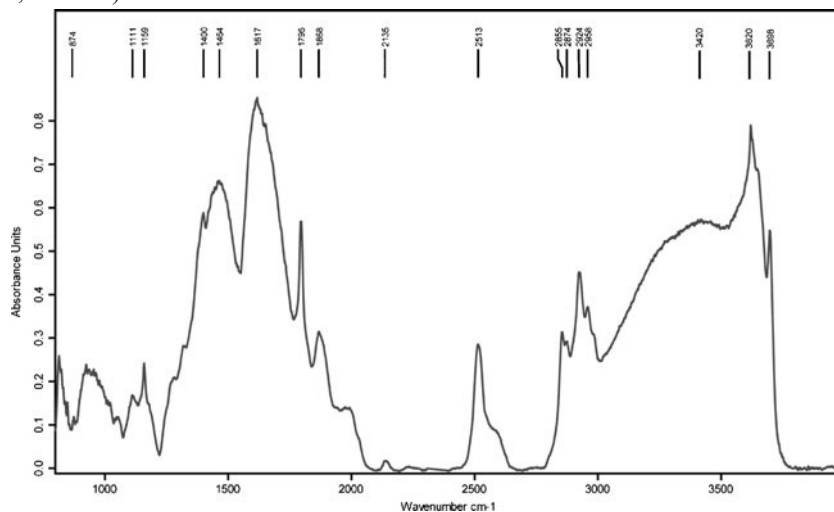


Fig. 11 FTIR analysis of the residue from Hohlenstein-Stadel tooth no. 8

Compound	Assignment	Wavenumber (cm^{-1})
Organic binder	OH bonds	1,618, 3,420w, 3,620, 3,698
	CH ₂ and CH ₃ bonds	2,855, 2,874, 2,924, 2,958
Calcite	CO ₃	874, 1,400, 1,795, 2,513

Table 2 Band assignment of the FTIR spectra obtained on the residue sample no. 8

The compound appears to be composed of a clay matrix containing sorted angular fragments of a silica rich mineral, probably quartz (Fig. 10c, d1) associated with smaller iron rich particles (Fig. 10d2, e, f), identified by Raman spectrometry as containing hematite (Fe_2O_3). High magnification inspection of these particles shows an amorphous structure (Fig. 9e, f) possibly composed of fragmented tests of unicellular organisms (Fig. 9g).

Discussion and conclusion

Irregular fractures recorded on the bony ankylosis of seven teeth indicate that they were extracted from the pharyngeal bone by flexion. Experiments show that these teeth can be easily extracted from a fresh or boiled pharyngeal fish bone (Cristiani and Borić 2012) suggesting that the teeth were acquired shortly after the capture or consumption of the fish.

The consistent location and appearance of the pigmented compound around the fish teeth bony neck suggests that this material was used as an adhesive to attach the teeth.

Our analysis indicates that the compound is composed of a thin matrix, probably clay, containing particles of charcoal, dolomite, quartz and hematite. The sorted angular appearance of the quartz fragments suggests that quartz was finely ground before being incorporated into the mixture. The alternative hypothesis is that the quartz fragments come from the grindstone used to crush one or more elements composing the residue. The abundance and size homogeneity of the quartz fragments better supports the first hypothesis. The presence of carbon in the fine-grain matrix, the high fluorescence produced by our attempt to analyze it with a 532 nm Raman spectrometer, the presence of high proportion of C–H and O–H bands identified by the FTIR analysis, and the adherence of the compound on the teeth, is consistent with the use of an organic binder. Resin, gum, or beeswax are well known

components of traditional glues (Daher et al. 2010; Wadley 2010). However, the FTIR signal does not identify, in our case, clear chemical fingerprints of the organic binder (d'Errico et al. 2012; Maia et al. 2013).

The addition of ochre and charcoal in the preparation of adhesives used for manufacturing composite tools is attested in the Middle Stone Age of Southern Africa (Lombard 2007; Wadley et al. 2009), the Ibéromaurusian (Ighilahriz 1996) and Caspian of North Africa (Beyries 1983), the Mesolithic of Italy (Cristiani et al. 2009), and the Pre-Pottery Neolithic A (PPNA) (Shaham et al. 2010). The use of adhesives in prehistoric beadwork has been occasionally suggested (Bonnardin 2009; Cristiani and Borić 2012), but never demonstrated. Numerous ethnographic examples suggest that this practice may well have been at work in prehistory (Dubin 1999; Verswijver 1986).

Results presented here lead to the conclusion that carp teeth were suspended using an organic glue to attach a link around their neck. Survey of the literature indicates that carp teeth are found at six other Mesolithic sites (Rigaud 2011, Fig. 12, Table 3). Three of them, Burghöhle Dietfurt (BadenWürttemberg, Germany), Falkensteinhöhle (Bavaria, Germany), and Probstfels (Baden-Württemberg, Germany) located in the Upper Danube region (Newell et al. 1990) and three others, Vlasac (Serbia), Kula (Serbia), and Schela Cladovei (Romania), in the Lower Danube. The three carp teeth used at Burghöhle Dietfurt present a drilled perforation on the lateral aspect of the bony neck (Taute 1990, Fig. 17). Peters (1933 cited in Taute 1990) also mentions a lateral perforation on some of the Falkensteinhöhle carp teeth. At Vlasac, half of the carp teeth were perforated by notching the frontal aspect of the bony neck (Borić 2002; Borić and Miracle; 2004; Cristiani and Borić 2012) while the other half was unmodified and probably attached with organic threads as suggested by purported remains of tendons or skin on the bony neck of some teeth (Cristiani and Borić 2012). Unfortunately, no detailed data are available on the suspension technique of the carp teeth from Probstfels, Schela Cladovei, and Kula. In the Upper Danubian area, the site of Burghöhle Dietfurt yielded carp teeth ornaments associated with perforated red deer canines and fossil gastropods attributed to the *Gyraulus sulcatus* (Hilgendorf 1866) and *Gyraulus trochiformis* (Stahl 1824) species (Peters 1934; Rähle 1978, 1981). Carp teeth of Falkensteinhöhle, located in the same region, were associated with perforated red deer and fox canines, a tubular bone bead, shells belonging to the Mediterranean *Columbella rustica* (Coen 1937) species, as well as perforated fossil gastropods attributed to the *Potamides laevis* (Schloth.) and *Pirenella plicata* (Bruguier, 1792) species (Peters 1934; Rähle 1978; Taute 1973–74). At Probstfels, carp teeth were associated with perforated fossil *Potamides laevis* (Peters 1934; Rähle 1978, 1981). In the Lower Danubian area, carp teeth ornaments are associated with *Cyclope neritea* and *C. rustica* shells at the cemetery of Vlasac and to *C. neritea* (Linné 1758) shells at Schela Cladovei.

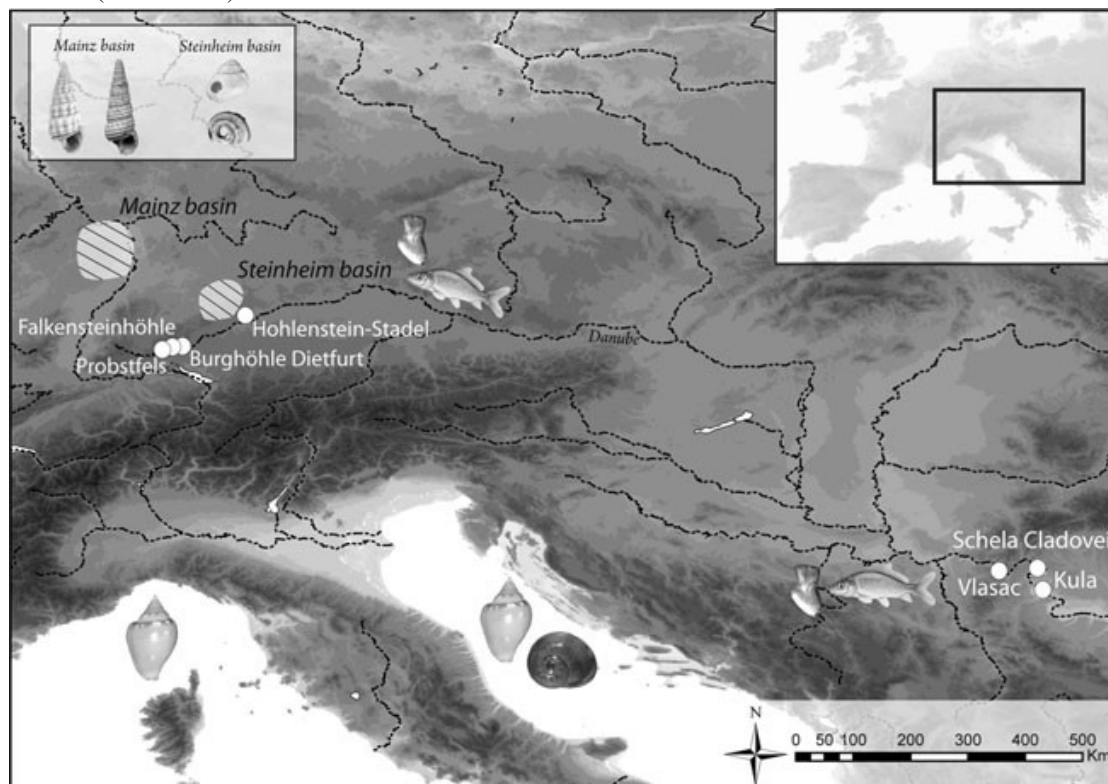


Fig. 12 Synthetic map of raw material used for Mesolithic personal ornamentation along the Danube and their origin

Site	Location	Layer	Attribution	Bead types	References
Probstfels	UD	Camp	Early/Middle Mesolithic	Cyprinid teeth Potamides laevisissimus	Peters 1934; Rähle 1978, 1981
Falkensteinhöhle	UD	Camp	Middle Mesolithic	Columbella rustica Cyprinid teeth Fox canine Pirenella plicata Potamides laevisissimus Red deer canine Tubular bone bead	Peters 1934; Rähle 1978; Taute 1973–74
Burghöhle Dietfurt	UD	Camp	Middle Mesolithic	Gyraulus sulcatus Gyraulus trochiformis Red deer canine Cyprinid teeth	Rähle 1978, 1981
Holenstein-Stadel	UD	Burial	Late Mesolithic	Cyprinid teeth	Völzing 1938; Wetzel 1938
Vlasac	LD	Burial	Late Mesolithic	Columbella rustica Cyclope neritea Cyprinid teeth	Borić 2002; Borić and Miracle 2004; Cristiani and Borić 2012; Srejovic and Letica 1978
Schela Cladovei	LD	Burial	Late Mesolithic	Cyclope neritea Cyprinid teeth	Boroneant 1999
Kula	LD	Burial	Mesolithic	Cyprinid teeth	Radovanovic 1996; Sladic 1986

Table 3 Contextual data on Mesolithic sites yielding Cyprinid pharyngeal teeth ornaments. (UD) Upper Danube, (LD) Lower Danube

Positioning of the carp teeth ornaments on the body also differs between the two areas. Carp teeth ornaments are located on the torso, arms and legs of the deceased in the Iron Gates suggesting they were sewn on a cloth (Cristiani and Borić 2012) while their presence in the sediment surrounding one of the skulls at HohlensteinStadel suggests that they were part of a head-ornament at this site.

In conclusion, the use of cyprinids teeth as personal ornaments in the Mesolithic of the Upper and Lower Danube regions is, in a number of respects, a puzzling cultural phenomenon. It represents a cultural innovation since no previous use of such teeth is attested in prehistory. It appears almost at the same time in two distant regions, which raises the question of the mechanisms (inheritance from an ancestral population, independent invention or diffusion) that may have been responsible for its adoption. It does not reveal, once the objects are analyzed in detail, clear regional trends in the way carp teeth were modified, suspended and worn as one may reasonably expect in the case their use reflected cultural differences between the two regions. In order to choose between these competing hypotheses (inheritance, independent invention, diffusion) we need to analyze other artifact categories from the two regions and put the Mesolithic beads from the Upper and Lower Danube in the context of the Mesolithic bead type variability across Europe.

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