Hafting of a Neolithic leister: Identification of adhesives from Lolland (Denmark)

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ABSTRACT

Birch tar has been identified as the predominant adhesive used for hafting purposes in the European Mesolithic and Neolithic. Its role in the manufacture of composite tools and weapons comprising wooden, bone and flint components attests its importance during these periods. The discovery of birch tar lumps, some bearing tooth imprints, suggests a broader range of functions beyond its adhesive properties. In this study, we present an analysis of five residues from two sites (Syltholm II and Strandholm I) that have been excavated through the Femern project, with the aim to shed light on the adhesives used in relation to their functions. Through chemical analyses, we show that birch tar constitutes the main component of two lumps and one chewed piece. We also found that birch tar served to haft a bone point within leister prongs, providing new information on its previously unknown role in composite fishing tool technology. These findings have significant implications for our understanding of the functional role and performance of birch tar in aquatic environments.

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Introduction

Adhesives have been produced and used as early as the European Middle Palaeolithic (Grünberg et al., 1999; Mazza et al., 2006; Niekus et al., 2019; Schmidt et al., 2023). Birch tar (also referred to as birch bark tar or birch pitch), is the predominant adhesive identified in the archaeological record throughout prehistory. In more recent periods, adhesive types and their functionality diversified (Nardella et al., 2019; Rageot et al., 2021). Various discoveries of birch tar lumps (Binder et al., 1990; Regert et al., 2000; Vahur et al., 2011), some bearing tooth imprints (Aveling and Heron, 1999; Fuchs and Wahl, 2013; Jensen et al., 2019; Kashuba et al., 2019), attest the importance of this material across diverse Meso- and Neolithic contexts. During these periods, birch tar played an important role as a hafting adhesive for the manufacture of composite tools such as daggers (Bjørnevad et al., 2019; Manninen et al., 2021; Osipowicz et al., 2020), arrows (Larsson et al., 2016), or hafted bone points (Kabaciński et al., 2023; Mirabaud et al., 2015, p. 1007). To our knowledge, no previous studies have been conducted to investigate the role of adhesives in the composite technology of fishing equipment, and only few studies suggest that hydrophobic properties could be of advantage in such conditions (Kabaciński et al., 2023). To address this gap, we conducted chemical analyses to identify the residue on a hafted leister point recovered during the Femern project on Lolland (Denmark) (Stafseth and Groß, 2023). In addition, we included four other artefacts in our study: three nondescript pieces of residue and one chewed piece. The purpose being to chemically characterise and cross-compare similar appearing black substances.

All samples were recovered during the Femern project and come from waterlogged sediments in site complexes 4 and 5 in the former Syltholm fjord (Figure 1), dated to the Late Mesolithic and/or Neolithic. As the finds come from lacustrine environments that underwent changing depositional processes (erosions, accumulations, etc.) they cannot be easily contextually dated. Due to the potential intermixture of sediments, only direct dating can provide correct age determinations for the single finds. The chewed piece is one of the most prominent finds from the Femern project and a previous study revealed that it contains human aDNA material from a female

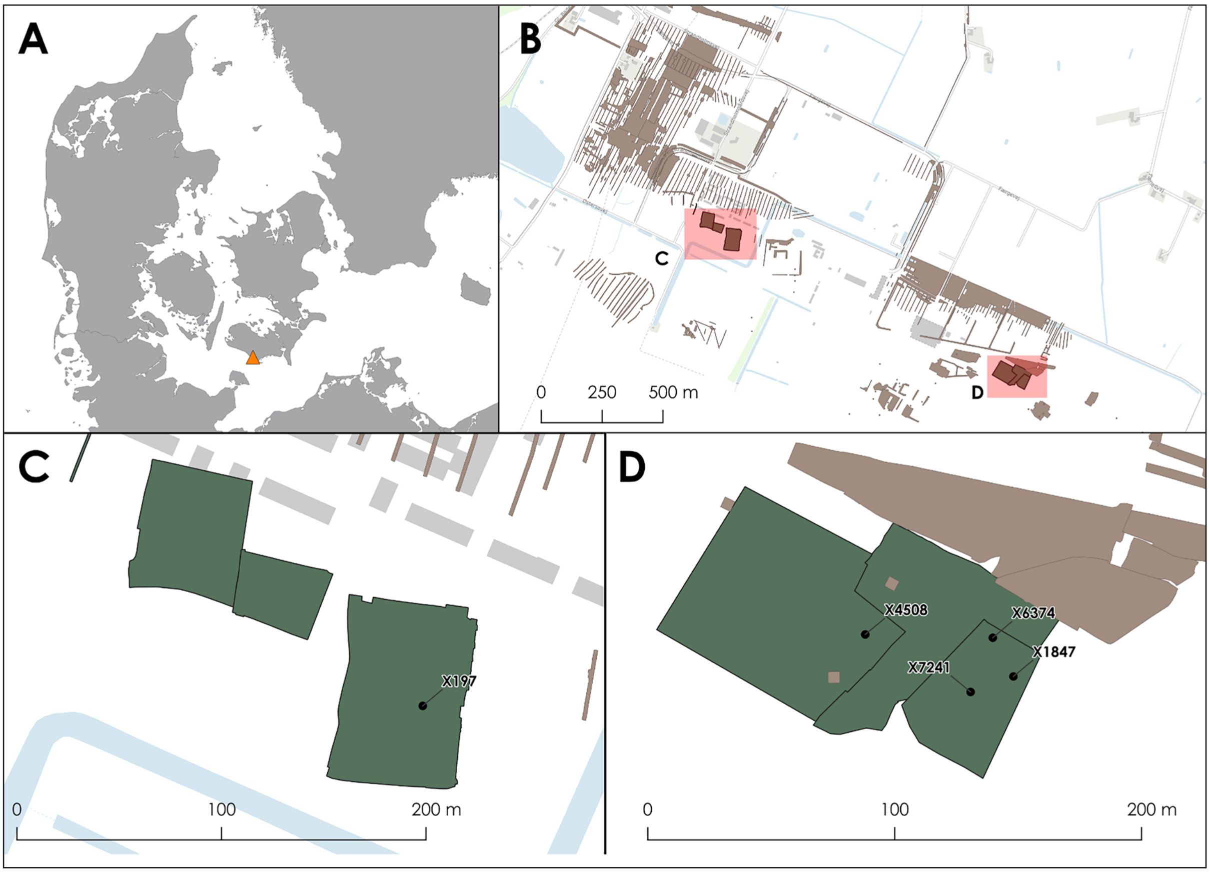

Figure 1. A) Location of the excavations from the Femern project. B) All excavation trenches from the project (brown) and sampled sites (green). C) Excavation trenches of the site Strandholm I (MLF00909) and find locations of the sampled artefacts. D) Excavation trenches of the site Syltholm II (MLF00906) and find locations of the sampled artefacts.

individual, dating to 3930-3710 cal. BC, from the site Syltholm II (MLF00906-II) (Jensen et al., 2019). Our findings of additional artefacts therefore help strengthen our understanding of the adhesives used at this site.

Materials & methods

Archaeological samples

We sampled residues from five different archaeological artefacts. These include a black substance adherent to the bone point found in situ with wooden leister prongs (MLF00909-II X197), three non-descript pieces/lumps (MLF00906-I X4508, MLF00906-II X6374 and MLF00906-II X7241) and one chewed piece (MLF00906-II X1847) (Figure 2). Two of these artefacts have been directly dated to the Late Neolithic (2020-1780 cal. BC for MLF00909-II X197; Måge et al., 2023, supp. mat.) and Early Neolithic (3930-3710 cal. BC for MLF00906-II X1847; Jensen et al., 2019).

Chemical characterisation

The samples were prepared using protocols adapted to the analysis of adhesive materials (for details on the extraction protocol, see Rageot et al., 2021, 2019). In brief, sample powders were solvent extracted using dichloromethane and derivatised with N,Obis(trimethylsilyl)trifluoroacetamide (BSTFA) and pyridine. We used two internal standards (Tetratriacontane and Hexadecane). The extracted and derivatised samples were analysed using a Shimadzu GC 2010 PLUS gas chromatograph equipped with an Agilent J&W HP-5MS GC Column (30 m length x 0.32 mm diameter x 0.25 μm film thickness). Mass spectra were recorded using a Shimadzu QP2010 ultra mass spectrometer and spectral acquisition spanned the range of m/z 50-950. A blank solvent sample was run to assess in-

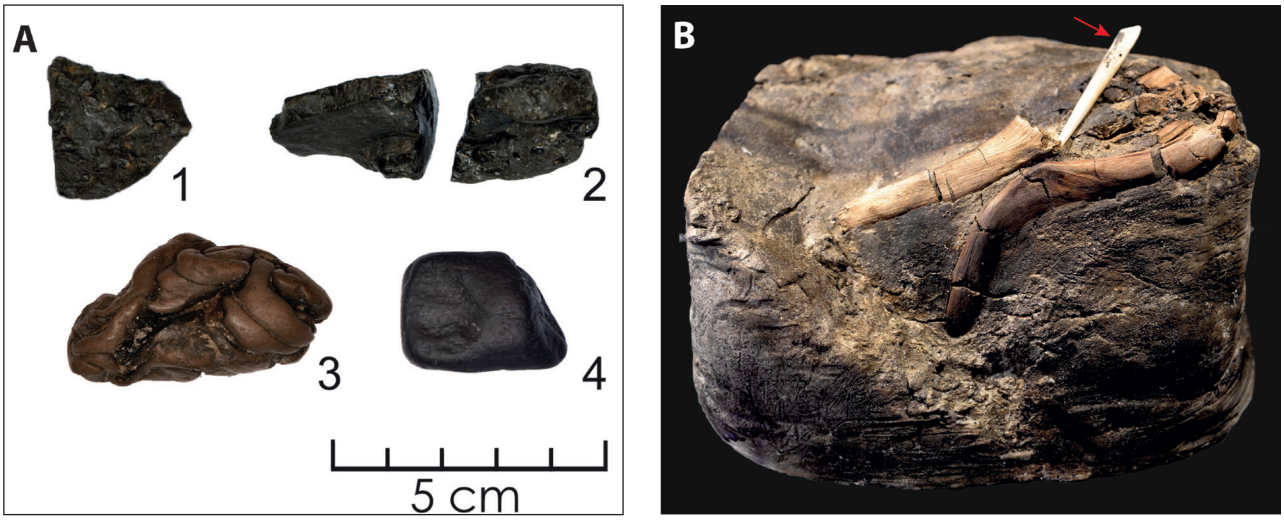

Figure 2. A) Sampled birch tar fragments from the site Syltholm II (MLF00906-II): 1 X6374; 2 X7242; 3 X1847. 4 is a piece of charcoal from Syltholm II (MLF00906-I) X4508, that was initially misidentified as potentially being birch tar. B) A hafted leister point in situ from the site Strandholm I (MLF00909-II): X197. Remains of the birch tar are visible on the proximal part of the bone point (red arrow) (copyright: Museum Lolland-Falster; after Stafseth and Groß 2023, Fig. 1).

laboratory contamination. Compound identification was done using the NIST library and previously published data (Aveling and Heron, 1998; Rageot, 2015; Reunanen et al., 1993).

Results

Four of the archaeological samples contained characteristic compounds indicative of birch tar (Table 1; Figure 3). These include specific biomarkers that can be associated with the molecular composition of birch bark (Aveling and Heron, 1998; Hayek et al., 1990, 1989), natural degradation markers, and molecular markers linked to birch tar production (Rageot et al., 2019; Reunanen et al., 1993). Betulinic acid and allobetulin were present in the leister and chewed tar, but absent in the two lumps that were identified as birch tar. Multiple combinations of diacids and unsaturated fatty acid were also detected. One of the nondescript lumps (MLF00906-I X4508) did not contain any triterpenoid compounds and only showed the presence of two unsaturated fatty acids. These were also detected in the control sample and can hence be regarded as contamination. A list of all compounds detected can be seen in Table 1.

Discussion

Our analysis presents the first chemical identification of birch tar as a hafting agent for prehistoric leisters and active fishing tools in general (see Stafseth and Groß, 2023) in Denmark, and possibly even Europe. While another specimen from the Ertebølle site Næbbet, settlement 24 (Skaarup and Grøn, 2004), shows a similar quality of preservation, no analysis of hafting agents has been conducted to confirm its composition. The identification of birch tar lumps, one bearing genetic material, further underscores both the favourable preservation conditions and the importance of this material in this region. In the context of fishing equipment, it remains unclear whether birch tar had a particular advantage over other adhesives for use in water contexts. Current research has assessed the performance of birch tar as a hafting adhesive (Koch and Schmidt, 2023; Kozowyk et al., 2017; Schmidt et al., 2022, 2021). However, no studies have addressed how these mechanical properties change under water. Still, birch tar has been suggested to yield waterproofing abilities, as shown in the context of coating organic or ceramic containers (Regert et al., 2003; Reunanen et al., 1993), which might also present an advantage for its use as a hafting adhesive in an aquatic environment. Kabaciński et al. (2023) suggest that birch tar on a composite point from Krzyż might have been used both for its

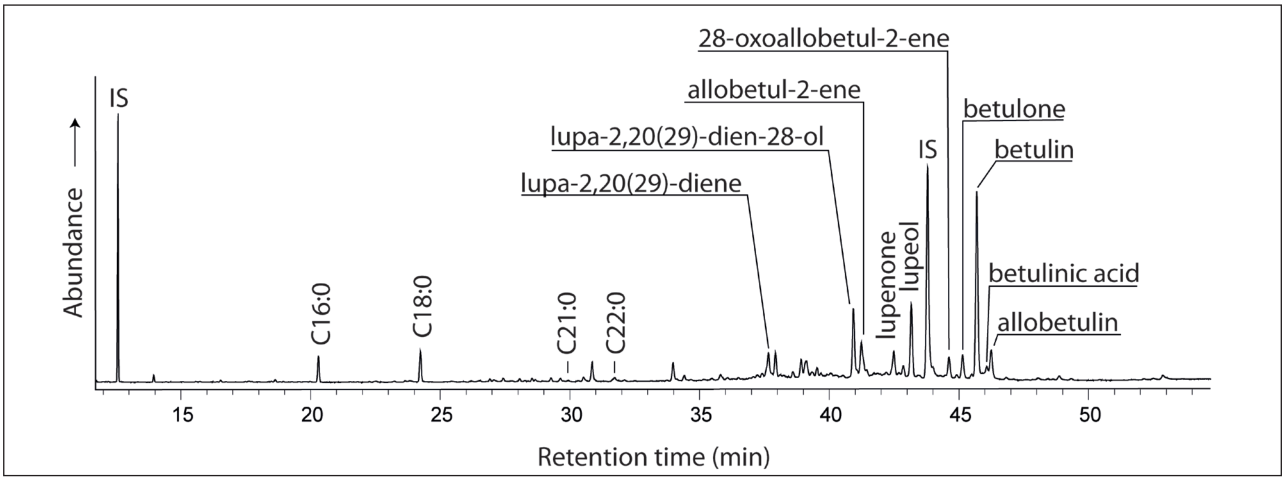

Figure 3. Chromatogram obtained through GC-MS analysis of the residue adherent to the leister bone point (MLF00909-II X197) indicating the presence of unsaturated fatty acids (CX:Y, X = number of carbon atoms, Y = number of saturations) and triterpenoid compounds characteristic for birch tar, IS = Internal Standard.

	MLF00906-II X1847	MLF00906-II X6374	MLF00909-II X197	MLF00906-II X7241	MLF00906-I X4508
Azelaic acid				✓	
Hexadecanoic acid	✓	✓	✓	✓	✓
Heptadecanoic acid		✓		✓	
Octadecanoic acid	✓	✓	✓	✓	✓
Heneicosanoic acid	✓	✓	✓	✓	
Docosanoic acid		✓	✓	✓	
Lupa-2,20(29)-diene	✓	✓	✓	✓	
Lupa-2,20(29)-dien-29-ol	✓	✓	✓	✓	
Allobetul-2-ene	✓	✓	✓	✓	
Lupenone	✓	✓	✓	✓	
Lupeol	✓	✓	✓	✓	
28-oxoallobetul-2-ene	✓	✓	✓	✓	
Betulone	✓	✓	✓	✓	
Betulin	✓	✓	✓	✓	
Betulinic acid	✓		✓		
Allobetulin	✓		✓		

Table 1. List of compounds identified in the archaeological samples from Lolland-Falster. MLF00909-II X197 = leister adhesive, MLF00906-I X4508, MLF00906-II X6374 and MLF00906-II X7241 = nondescript lumps, MLF00906-II X1847 = chewed piece.

adhesive and waterproofing properties, which might also be the case for the leister. Numerous components of other leisters, individual prongs or bone points, have been recovered at the Femern project (Chaudesaigues-Clausen, 2023; Stafseth and Groß, 2023), but whether birch tar was also used to assemble these remains hypothetical for now. To strengthen our understanding of these cultural and technological aspects, it is important for future studies to explore specific categories of artefacts and their association with adhesives (e.g. fishing equipment) on a larger, comparative scale. Through analytical approaches aiming at the identification of organic, but also inorganic materials, we can assess the range of adhesive substances used throughout prehistory, but also reveal unprecedented insights into, for instance, prehistoric technologies (e.g. Kabaciński et al., 2023) or personalities (Jensen et al., 2019). This will enable us to better understand potential differences in the chaîne opératoire, functional purpose, regional preferences and potentially also the social role of birch tar and other adhesive products in different archaeological and historical societies (Little et al., 2022).

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Declaration of interest statement

The authors declare no conflict of interest.

Credit statement

T.J.K is responsible for conceptualisation, investigation, visualisation and writing. A.L. is responsible for supervision, project administration, writing, funding acquisition. D.G. is responsible for writing, visualisation, data curation and resources. B.T.M. is responsible for resources.

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