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ATR-FTIR spectroscopy analysis of mineral-preserved textiles from Viking Age women's graves in Vinjefjord, Norway

Abstract

Recently excavated, mineral-preserved textile fragments recovered from three largely undisturbed Viking Age inhumation female graves in Vinjefjord, Mid-Norway were analysed by Fourier transform infrared spectroscopy (FTIR) equipped with attenuated total reflection (ATR) to identify fibre type and potential dyes. The textiles were found in intimate contact with copper-alloy garment accessories and originate from a boat burial and a simple oval pit burial at Skeiet in Vinjeøra and a burial chamber at Hestnes in Valsøyfjord. Five textiles were selected for analysis. Three were reliably identified as cellulose-based or hair/wool. The degree of mineral preservation of the remaining two samples was too advanced to establish a conclusive identification. The wool-identified sample tested positive for the plant-based dye indigo. The application of FTIR analysis to archaeological textiles to identify both fibre type and dyestuffs, is not uncommon. However, its application to Viking Age period textile remains recovered from excavations in Norway has not been previously reported.

Keywords: ATR-FTIR, Vinjefjord, Norway, Viking Age, textiles, dye analysis, indigo

Introduction

Mineral-preserved textile fragments recovered from three largely undisturbed Viking Age female inhumation graves in Vinjefjord, Mid-Norway were analysed by Fourier transform infrared spectroscopy (FTIR) equipped with attenuated total reflection (ATR) to identify fibre type and potential dyes. The textiles were found in intimate contact with copper-alloy garment accessories and originate from a boat burial and a simple oval pit burial at Skeiet in Vinjeøra and a burial chamber at Hestnes in Valsøyfjord all of which were excavated as part of the E39 Betna-Stormyra rescue archaeology project.

The site

The European Route E39 is the primary coastal road connecting western and central Norway, running along the southern shore of Vinjefjord to the main

Norwegian coastal sea route (the *Norvegr*). Between 2019 and 2020 a large scheme of rescue archaeology excavations commissioned by the Norwegian Public Roads Administration was carried out in selected culturally protected areas of the Vinjefjord region in preparation for the final planned upgrade of the E39 road (fig. 1).

The E39 Betna-Stormyra project is the most extensive archaeological excavation of Late Iron Age cemeteries and settlement features carried out in Mid-Norway (Sauvage 2024, 7). Several settlement sites along the southern shore of the long, narrow fjord were investigated. Archaeological remains from the Iron Age and Middle Ages (500 BCE to 1537 CE) were discovered in three separate locations. These were situated on the farms Skeiet and Fjelnset at Vinjeøra (innermost in the fjord) and Hestnes along Valsøyfjord (a smaller side fjord to the south). The Skeiet site

consisted of two areas: Skeiet 1 and 2. Skeiet 1 contained 11 (possibly 12) flattened burial mounds identified by their circular rings. Excavations revealed 18 graves, and three mortuary houses dated to the Late Iron Age, and the graves were richly furnished with a large collection of artefactual grave goods. Skeiet 2 contained Iron Age and medieval settlement features and remains with at least nine identifiable buildings (Sauvage and Lorentzen 2024, 131–133; Sauvage 2024). The Fjelnset site was characterised by poor preservation, methodical robbing and erosion. The Hestnes site yielded mortuary evidence in the form of a levelled burial ground that contained the remains of a richly furnished, decayed wooden Viking Age chamber burial, including items of jewellery, textile fragments, beads, tools and several fragments of clothing (Sauvage 2024).

During post-excavation conservation of the grave goods recovered from Skeiet 1 and the wooden chamber burial at Hestnes, it was discovered that fragmented remains of the textiles once worn by the buried persons were preserved in close association

with metal garment accessories in some graves. A total of 77 textile remains, including some fragments of down feathers, were identified from the three Viking Age female inhumation graves, in particular: Skeiet 1 Graves 2 and 11 and the Hestnes burial chamber (Øien and Heen-Pettersen 2024, 213–216). Skeiet Grave 2 was a simple oval pit burial (circa 900 to 950 CE); Skeiet Grave 11 was a boat burial (circa 800 to 850 CE); and Hestnes was a chamber burial (circa 850 to 950 CE). Øien and Heen-Pettersen (2024) studied and catalogued the technical aspects (for example, fibre, spin direction, thread count) of the textile remains using visual and traditional optical microscopy (OM) techniques. Their further study focused on an evaluation of the funerary clothing and dressing practices related to mortuary rituals during the Viking Age (Øien and Heen-Pettersen 2024).

Textile mineral preservation

A wide range of organic materials, including textiles, are preserved in the archaeological context through the gradual diffusion of metallic cations from nearby

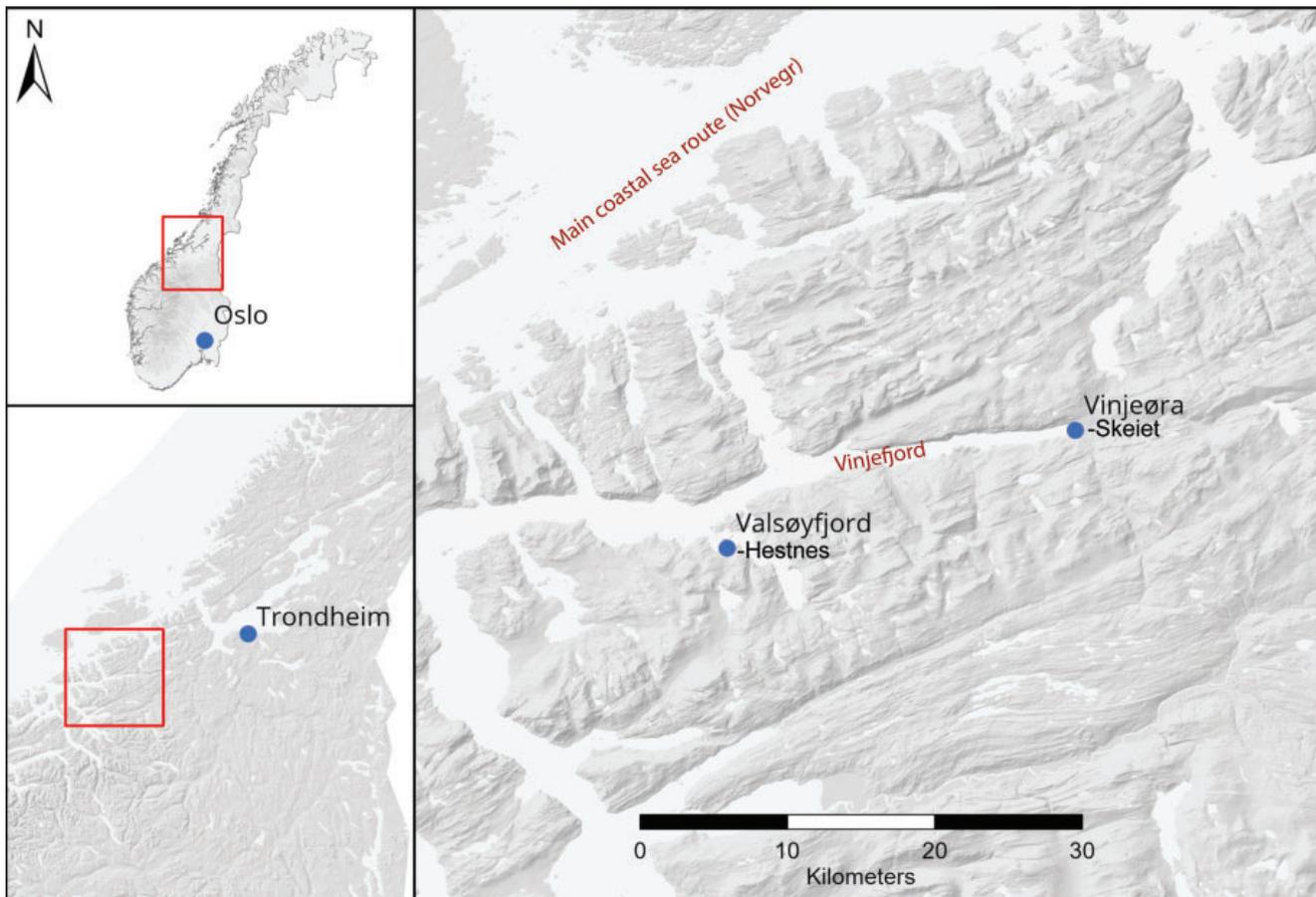
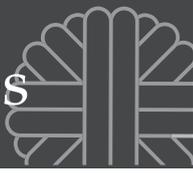


Fig. 1: Location map showing the regional setting in Norway of the E39 Betna-Stormyra project and the Hestnes and Skeiet sites along Vinjefjord (Image: Thea P.B. Christopherson after Kristoffer R. Rantala)



artefacts or, the surrounding environment (Grömer and Grassberger 2018). In fact, it is proposed that finds preserved in this manner can no longer be considered exceptional (Peška et al. 2006, 5). The study of mineral preserved textiles has received more interest in comparison with studies of mineral-preserved leather, for example, in large part because fibre artefacts retain their morphological features and can be more readily visually identified. The permeation process leads to fibres of varying degrees of mineralisation. Preservation ranges from lightly mineralised (“pre-mineralization” (Jia et al. 2024, 10)) retaining close to the original organic and physical properties (for example, a somewhat soft handle) to completely mineralised into a hard or powdery form retaining minor traces of the original organic composition. Attention is now being drawn to previously unidentifiable corrosion features (“ephemeral traces”) in metal corrosion products that are traces of – but no longer appear like – textiles (Davis and Harris 2023, 15–18; Angiorama et al. 2020; Peška et al. 2006, 22–26, 28–31, 39–43).

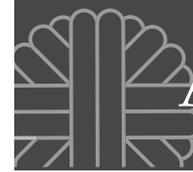
The complex mechanisms of textile mineral-preservation have not been fully ascertained, especially at the nano- to microscale (Reynaud et al. 2020). Several studies have investigated the mineralisation process through experimental degradation in the laboratory (Jakes and Howard 1986; Gillard et al. 1993; Gillard et al. 1994a; Chen et al. 1998). These have provided insights into the preservation process, importantly, that traces of the organic complex can remain in sufficient quantities to allow distinction between protein- and cellulose-based fibres (Gillard et al. 1994a, 138). In fact, Gillard and colleagues concluded that complete mineralisation is probably relatively uncommon (Gillard et al. 1994a, 138).

The near presence of metals such as copper, silver, iron and/or lead (and their alloys) can promote the preservation of organic material, with copper and iron being the most common. Additionally, calcium-rich burial environments contribute to a rarer type of mineral-preservation. Water from the surrounding (burial) environment percolates into and swells the fibres, followed by the transport of soil solutes (for example, calcium) and biocidal metal (for example, copper, silver) cations released during the initial corrosion of closely associated metal artefacts such as garment accessories or tools. If the concentration of metallic cations is high enough, the corrosion products become toxic to many microorganisms and thus inhibit microbial degradation of the fibres slowing down the deterioration process. At the same time, the concentration must be sufficiently low enough to enable the ions to penetrate the fibres. Continued

infilling or impregnation of the fibre structure and simultaneous fibre degradation can proceed to the point where the organic composition is either partially or completely replaced, or a hollow cast of the fibre is formed. These are sometimes referred to as “positive” or “negative” casts respectively. Less-studied forms of textile mineral-preservation are found on stone (for example, Schuurman 2023) and ceramics as well as in soils (Unruh 2007).

Study of mineral-preserved textiles

The primary scientific research method of analysis in the study of textile artefacts is visual observation. The presence of textile remains on metal artefacts has been the subject of observation and description since the 18th century (Chave et al. 2024). Scholarly studies of the appearance of fibre morphology and corrosion products of mineral-preserved textiles was based on visual observation with the naked eye. This was later complemented with the development of the simple light microscope. The systematic visual examination and identification using optical microscopy in the form of the compound microscope, was pioneered by Biek (1963, plates 7 and 10) and was complemented by simple microanalytical methods such as microchemical staining (Biek 1963, 118; Anheuser and Roumeliotou 2003) used to identify the broad fibre type. These methods, now including portable USB digital optical microscopy, continue to be the initial and primary tools of study (Peacock 2024). The increasing availability of scanning electron microscopy (SEM) for investigation of artefacts has provided an increasingly essential tool for the systematic examination and identification of mineral-preserved fibres and associated mineralisation processes, especially where transmitted light will not penetrate fibres with a high mineral content (Janaway 1983; 1985; 1989; Rast-Eicher 2016, 16–17, 22, 33–35, figs. 6–7, 14–15, 32–35). Although a surface technique, its excellent depth of field and greater resolution aid identification if the fibre surface is reasonably well replicated. That said, more recent studies have pushed the identification of ephemeral traces of materials in corrosion products that no longer have the appearance of textiles (Davis and Harris 2023, 15–18; Angiorama et al. 2020; Peška et al. 2006, 39–43). When equipped with a backscatter (BSE) detector and energy dispersive X-ray spectrometer (EDS), microanalysis of the surface elemental composition and distribution can be assessed and mapped simultaneously with visual morphological examination of the fragments (Jakes and Sibley 1984; Chen et al. 1996; 1998; Angiorama et al. 2020). As the interest in mineral-preserved textiles has increased over recent decades, SEM has become



the principal examination method, when accessible. Infrared (IR) spectroscopy, especially Fourier transform infrared (FTIR) spectroscopy, is a widely used vibrational spectroscopic technique that is finding applications to the analysis of the chemical nature of not only historic but also archaeological textiles (Chen et al. 1996; Margariti 2019). The spectrometer measures the absorption wavelengths of an infrared light source that is passed through a material. Specific functional groups and molecular structures absorb specific wavelengths of IR radiation, and the bands of absorbance can be used to identify the composition of the material under study. For textiles this includes fibre group identification, degradation of the organic matrix and presence of dyes. ATR-FTIR is sensitive enough to produce spectra that are distinguishable between fibres from different animal species (McGregor et al. 2018) and plant species (Coletti et al. 2021) when they are modern and in good condition. Gillard and colleagues (Gillard et al. 1993; Gillard et al. 1994a; Gillard and Hardman 1996) investigated the application of FTIR microspectroscopy to modern fibres that had been experimentally mineralised in laboratory-based studies. Their studies showed the presence of measurable amounts of organic component in the mineralised-preserved fibre matrix, and that these traces could remain in sufficient quantities to allow distinction between protein- and cellulose-based fibres. Further, they reported that remnant dye could be detected by FTIR in archaeological textiles; although, mineral-preservation was not specified (Gillard et al. 1994b; Gillard and Hardman 1996). Early in the 1960s, Abrahams and Edelstein (1964) investigated IR spectroscopy for the analysis of solvent-extracted



Fig. 2: An example of a copper-alloy oval brooch (NTNU University Museum T28348:2) recovered from the female inhumation burial chamber at Hestnes and covered with layers of textile. Associated textiles were not selected for ATR-FTIR study (Image: Åge Hojem)

dyes in archaeological wool (circa 135 CE) from the Dead Sea. The FTIR attenuated total reflectance (ATR) mode of FTIR spectroscopy involves the pressing of a sample against a crystal window in an ATR accessory. Penetration depth is few to few tens of μm . Analysis is quick and convenient but might necessitate not only the taking of a sample but also deformation of degraded textiles, such as brittle mineral-preserved. Thus, unlike reflectance mode FTIR, ATR mode cannot necessarily be considered a non-invasive and non-destructive technique in all instances.

Today, a variety of advanced analytical methods are applied to the study of archaeological textiles (for example, Margariti et al. 2024). Contemporary methods of analysing mineral-preserved textiles have expanded to include, for example, peptide mass fingerprinting (PMF) measured by matrix-assisted laser desorption ionisation (MALDI) and time-of-flight (TOF) mass spectrometry (Solazzo et al. 2014), microtomography and synchrotron-based X-ray microtomography (μCT) (Iacconi et al. 2023; Jia et al. 2024), and radiocarbon (^{14}C) dating (Margariti et al. 2023). A comprehensive review has been carried out by Bertrand et al. (2025).

The study reported herein is a pilot investigation of the application of ATR-FTIR to identify fibre types and investigate potential dyes on five selected mineral-preserved textile fragments recently recovered from three Viking Age period female inhumation burials in Norway. The aim of this contribution, in addition to presenting the result, is to address the potential for this method in more detail specific for mineral-preserved textile fibres and the dye indigo than reported in other studies. It describes sampling, sample preparation and methodology while also providing a comprehensive literature review of relevant spectra and infrared band assignments to aid identification. The article introduces the archaeological background of the assemblage but does not include a detailed preliminary technical survey of either the selected textile fragments or other textile remains recovered from the burials (fig. 2). Furthermore, it does not present the ensuing discussion about Viking Age women's burial clothing and mortuary rituals at that time (Øien and Heen-Petersen 2024).

Methodology

Materials, recovery and post-excavation

The three female burials were apparently largely undisturbed at the time of excavation (Øien and Heen-Petersen 2024, 199). In cases where textile remains were preserved on metal garment



accessories, the composite objects were recovered in the field as soil blocks. Investigative excavation was carried out in the conservation laboratory at the NTNU University Museum. Detail records were made of the textiles' layering and how they were positioned on the artefacts before the fragments were separated. Fragments that were integrated with surface metal corrosion products and affixed to the accessories were not removed. A traditional visual technological analysis (for example, fibre, spin direction, thread count) was then undertaken (Øien and Heen-Pettersen 2024). Preservation of partial or possible textile features in metal corrosion products on the surfaces of the accessories associated with the textile fragments examined was not investigated (for example, Davis and Harris 2023; Angiorama et al. 2020; Peška et al. 2006, 22–26, 28–31).

Conservation

Before fragments were selected for analysis, the assemblage received light mechanical surface cleaning of soil and other residues from the archaeological context. No chemicals, including consolidants, were employed, with the exception of an Insular brooch (T28276:3, fig. 3e), which was further mechanically cleaned of surface corrosion. The fragile areas on the reverse surface were strengthened with Japanese tissue affixed with 10% Paraloid B72 (an ethyl methacrylate–methyl acrylate copolymer) (w/v) in ethanol.

Condition assessment

The condition, degree and type (for example, positive cast, negative cast, “complete” mineralisation) of degradation/mineralisation of the textile fragments were assessed using visual observation and optical microscopy. The results of the examination were

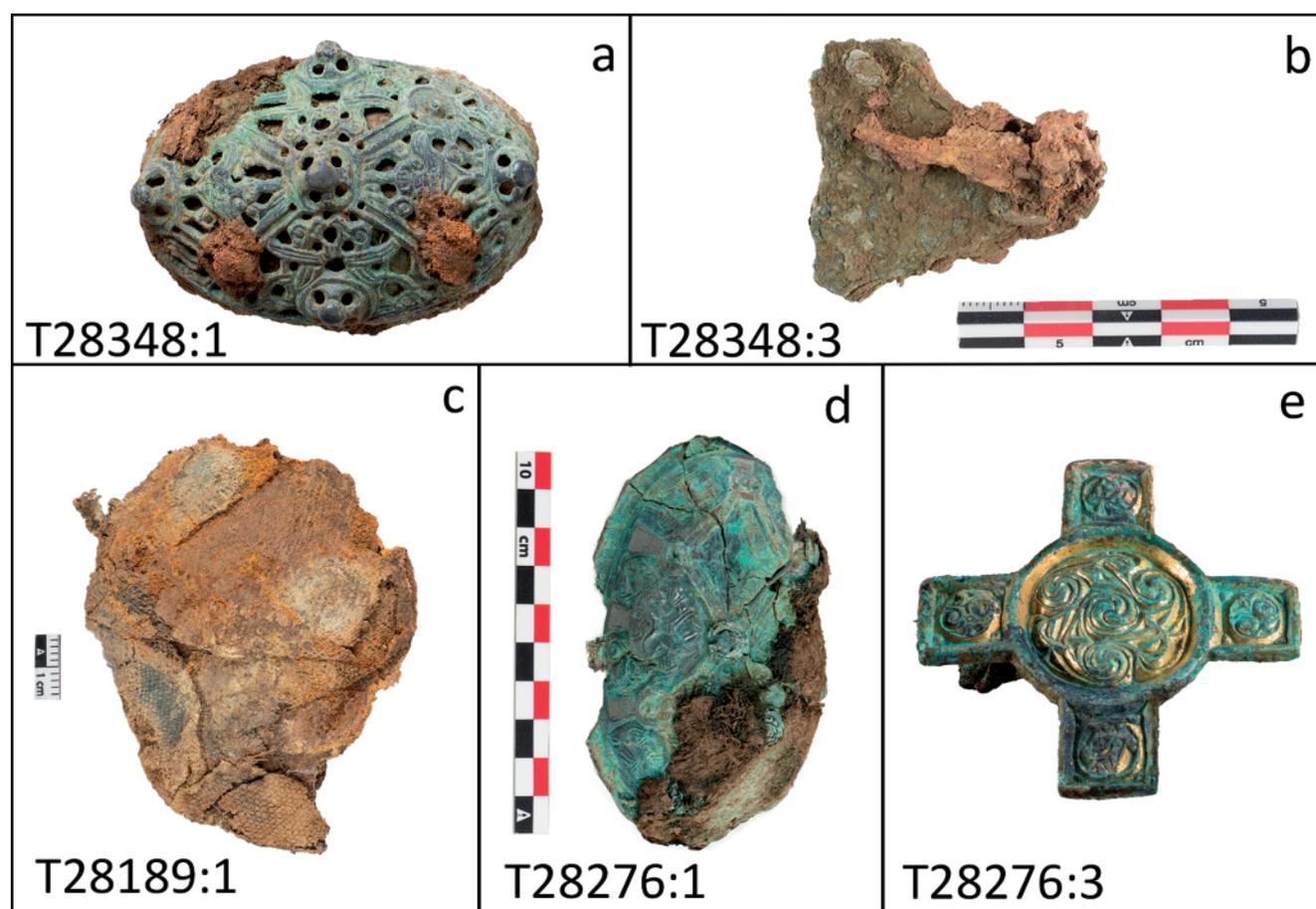
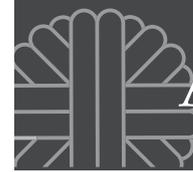


Fig. 3: The five metal artefacts with intimately associated textile fragments that were selected for ATR-FTIR analysis from the NTNU University Museum: a) copper-alloy oval brooch from Hestnes, T28348:1; b) copper-alloy trefoil brooch with iron pin fastener from Hestnes, T28348:3; c) an incomplete, seven-boss, copper-alloy oval brooch from Skeiet 1, Grave 2, T28189:1; d) copper-alloy oval brooch from Skeiet 1, Grave 11, T28276:1; and e) gilded copper-alloy Insular brooch from Skeiet 1, Grave 11, T28276:3 (Image: Thea P. B. Christophersen; a) after Åge Hojem; b) Eystein Østmoe; c) Thea P. B. Christophersen; d) Stian Ingdahl; and e) Åge Hojem)



Site	NTNU Museum number	Find number	Description	Condition	Colour
Hestnes, Female burial chamber	T28348	T28348:6	Seven fragments of tabby-woven textile found on the underside of copper-alloy oval brooch T28348:1	Mineral (Fe) preserved	Light orange brown
		T28348:12	Diamond twill-woven textile fragment found on the underside of copper-alloy trefoil brooch with iron pin fastener T28348:3	Partially mineral (Fe, Cu) preserved	Reddish brown, blue
Skeiet 1, Grave 2 Female inhumation grave	T28189	T28189:1	Several folded layers of a tabby-woven textile fragment that completely covers the top of an incomplete, seven-boss, copper-alloy oval brooch T28189:1	Hard mineral-like to mineral (Fe, Cu) preserved, powdery iron surface corrosion	Yellow brown, blue green areas
		T28276			
Skeiet 1, Grave 11 Female boat burial	T28276	T28276:59	Twill-woven textile fragment found on top of copper-alloy oval brooch T28276:1	Partially mineral (Cu) preserved	Dark brown
		T28276:75	Thread fragments associated with pin fastener on the reverse side of gilded copper-alloy Insular brooch T28276:3	Partially mineral (Cu) preserved	Straw coloured, dark blue green

Table 1: Description of the E39 Betna-Stormyra project textiles and associated copper-alloy garment accessories selected for ATR-FTIR analysis

used to assess the condition of the textiles to choose fragments for FTIR analysis (table 1), and to select an appropriate sampling site.

Selected textile remains

Five of the 77 catalogued textile remains were selected for analysis with Fourier transform infrared spectroscopy (FTIR) with an attenuated total reflectance (ATR) imaging system. As a group, the remains were fragmentary in nature and ranged from loose fibres, individual threads, and narrow fabric loops to woven fragments (of several cm²). Selection was based on the degree of mineral-preservation, fibre group identification, presence of loose fibres, friability, surface integrity, degree of fusion to corroded metal accessory, suspected presence of dye and research interest. The fragments represented the three female inhumation graves and were found in association with copper-alloy oval (tortoise), trefoil, or Insular brooches (fig. 3). Table 1 presents an overview of the selected fragments (table 1).

Sampling

A sample weighing between 5 and 10 mg was taken from each selected textile fragment (fig. 4). The condition influenced the method of sample taking and analysis preparation. From textile fragments with intact woven structure (binding points), one or two threads were removed. Loose textile fibres (T28276:75, fig. 4e) collected during conservation of the Insular brooch (T28276:3, fig. 3e) were used. For the textile find from Hestnes (T28348:6, fig. 4a) that consisted of seven hard, brittle mineral-preserved fragments, a whole fragment was selected since the textile fabric was too brittle for a thread or fibres to be safely removed.

It was possible to take a sample with visible blue dye from the twill-woven fragment (T28276:59, fig. 4d) from Skeiet 1, Grave 11. For the other textile finds, sampling was limited to areas and fibres without visible colour to minimise disturbance and material loss of textile. Prior to ATR FTIR analysis, each sample was further lightly mechanically cleaned of soil. Textile samples without visible

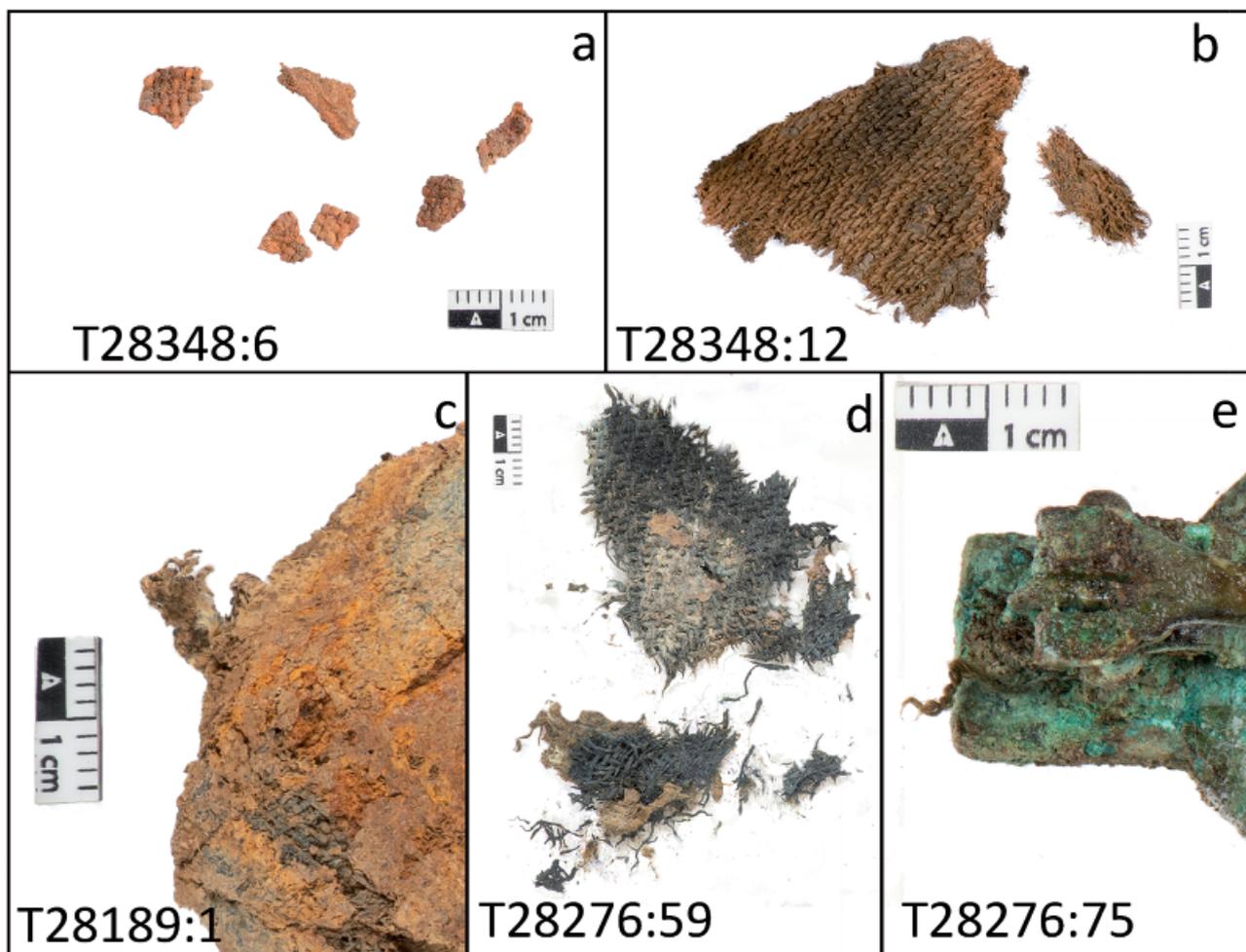


Fig. 4: The textile fragments that were selected for ATR-FTIR analysis from objects of the NTNU University Museum: a) fragment T28348:6 from the underside of copper-alloy oval brooch T28348:1, fig 3a; b) fragment T28348:12 from the reverse side of copper-alloy trefoil brooch T28248:3, fig 3b; c) one thread T28189:1 from the top of copper-alloy oval brooch T28189:1, fig 3c; d) one thread T28276:59 from the top of copper-alloy oval brooch T28276:1, fig 3d; and e) loose fibres T28276:75 from the reverse side of Insular brooch T28276:3, fig. 3e (Image: Thea P. B. Christophersen)

Reference material		Source
Linen yarn	Bockens linen yarn NEL 28/2 ½-BL, 100% linen, semi-bleached flax, undyed	Holma-Helsinglands AB, Sweden
Wool fabric	Unbleached, undyed white vadmél (wadmal)	Røros Tweed A/S, Norway
Blue-dyed wool fabric	Unbleached, undyed white vadmél dyed with indigo fermented in urine	Røros Tweed A/S, Norway
Indigo powder	Genuine indigo, <i>Indigofera tinctoria</i> L. CAS-Nr: 84775-63-3	Kremer Pigments GmbH & Co. KG Germany

Table 2: Modern reference fibre and dye materials used in the study. The reference wool fabrics were prepared for and included in numerous experimental burial degradation studies (Peacock 2004, 189–190; Solazzo et al. 2013, 49–50)

colour were additionally brushed with ethanol to eliminate contamination from other residues from the archaeological burial context.

Analyses and measurements

ATR-FTIR infrared microspectroscopy

IR spectroscopy was carried out with a benchtop PerkinElmer Spectrum 400 Fourier transform infrared (FTIR) spectrometer equipped with a Universal attenuated total reflection (ATR) imaging system with a diamond crystal ATR plate. Infrared spectra were taken of both the mineral-preserved samples and modern materials used as references (table 2). Each sample was positioned to cover the ATR analysis window and pressed down with the pressure arm to ensure good contact with the crystal. The instrument was operated with the associated Spectrum software. Infrared spectra were acquired over the frequency range 4000 to 550 cm^{-1} , and 128 scans accumulated with a spectral resolution of 4 cm^{-1} . Between each analysis, the ATR analysis window was cleaned with ethanol and lens paper and a background spectrum was taken to reduce variations in the spectra from background noise due to varying intensity of the laser and surrounding environmental disturbances (for example, humidity and CO_2 level).

Spectra from the archaeological textile samples were compared against those of the reference samples (table 2), as well as with the Conservation Laboratory's reference spectra library, reference spectra in the IRUG Spectral Database (Infrared & Raman Users Group 2025) and those reported in the literature (tables 3–5). Furthermore, the results were compared with the textile technological analysis of the textile finds reported by Øien and Heen-Pettersen (2024, 13–16).

Findings

Visual preliminary survey

In the visual preliminary survey carried out by Øien and Heen-Pettersen (2024, 13–16), four of the five selected fragments were tentatively fibre-type identified as follows: T28189: plant; T28276:59 animal; T2876:75 plant; T28348:6: inconclusive; and T28348:12 animal. In their survey, approximately 38% of the fibres could not be securely identified.

Condition assessment

All the selected textile fragments are mineral-preserved (copper-alloy and/or iron) in various stages (table 1) ranging from organic-preserved fibres/fabric with limited biological decay to remnant textile

surfaces with mineral-impregnated fibres and binding points. All fibres are preserved as positive casts.

The twill-woven fabric T28276:59 (fig. 4d) is partially mineralised. The threads and fibres are hard, brittle and break easily. In some areas, the weave is pinched, and the threads are no longer oval-shaped in cross-section. The fabric of the fragments has particles of sand/silt between the threads of the weave, which cause breakage when handled. There is plant material scattered on the surface. The colour of the fabric is a dark green blue indicating possible dye, as well as preservation due to intimate association with copper-alloy. In the centre of the largest of several pieces, is a hole where the fabric may have been attached to the oval brooch (T28276:1, fig. 3d). Surrounding this hole are greenish-white copper salts. In this area, the threads are greener in colour and more mineralised. The raw material of the threads was identified as animal fibre in the visual preliminary survey (Øien and Heen-Pettersen 2024, 215). The fragments of down were identified as seagull (*Larus sp.*) and sea duck (Mergini) (Rosvold [in press as cited in Øien and Heen-Pettersen 2024, 206–207]).

The second selected textile find from Grave 11, Skeiet 1 consists of loose, brittle off-white/transparent and blue green fibres (T28276:75, fig. 4e) from the copper-alloy pin fastener on the reverse side of gilded copper-alloy Insular brooch (T28276:3, fig. 3e). In the preliminary visual examination, these fibres were tentatively identified as vegetable (Øien and Heen-Pettersen 2024, 216). The remainder of the textile fragments left on the reverse of the brooch, consist of loose but compressed fibres – perhaps from fragments of a textile fabric caught around one end of the copper-alloy pin fastener. The condition of these fibres varies from off-white in colour and transparent to dark, mineralised and covered in a dark organic layer. These are stiff and brittle and have been more affected by their association with the permeating copper solution.

The textile remains (T28189:1, fig. 4d) from the female inhumation grave at Skeiet 1 consist of a relatively large, multi-layered folded assemblage of a tabby woven textile fabric sitting on top of a large fragment of a copper-alloy oval brooch with an iron pin fastener (T28189:1, fig. 3d). The fabric is covered in orange powdery iron corrosion products. The condition of the fabric varies from hard mineral-like to mineral preserved, but it is not fully mineralised. Much of the surface morphology is unclear. The fabric is of high quality. There are blue-coloured areas that may indicate the presence of dye. There are also greenish-blue areas suggesting mineralisation by corrosion solutions from the copper alloy. Due

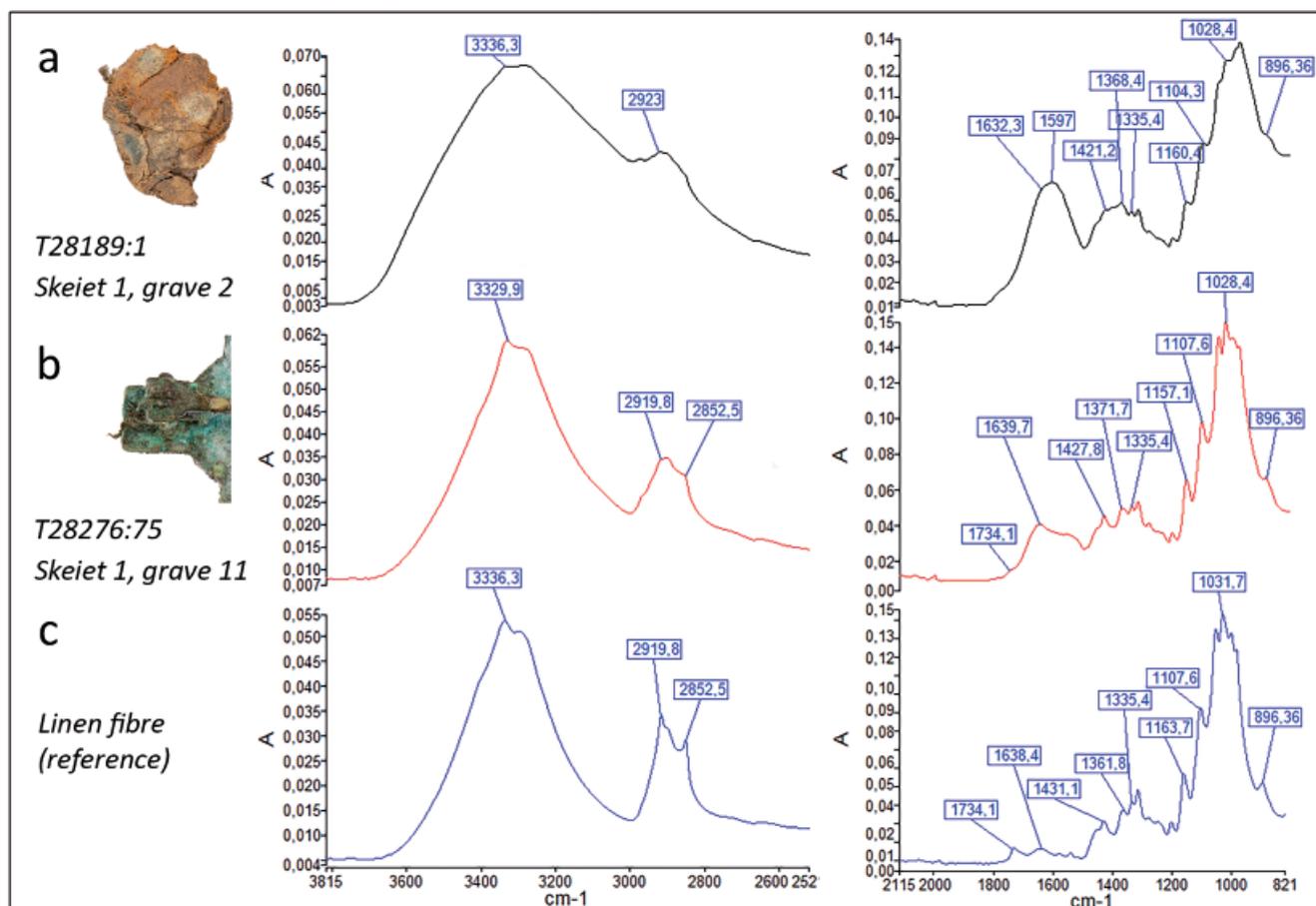
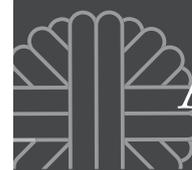


Fig. 5: Infrared spectra for two of the three textile finds from Skeiet 1, kept at the NTNU University Museum: a) textile found on top of copper-alloy oval brooch T28189:1 from Grave 2, see fig. 4c; b) textile fibres T28276:75, see fig. 4e, from the pin fastener on the reverse side of Insular brooch T28276:3, from Grave 11, see fig. 3e; and c) modern linen yarn, reference sample (Image: Thea P. B. Christophersen)

to the poor condition of the surface morphology, the results of the preliminary visual survey for fibre group identification were inconclusive (Øien and Heen-Pettersen 2024, 204–205).

The remaining two selected textiles were recovered from the burial of a woman in a wooden burial chamber at Hestnes. One consists of two larger fragments of a diamond twill woven fabric (T28348:12, fig. 4b) recovered from the underside of a copper-alloy trefoil brooch (T28348:3, fig. 3b). The fabric is predominantly reddish-brown in colour with some undertones of blue, which may indicate the presence of dye. The fabric may be slightly mineralised, but it retains its organic flexible nature. The morphology of the fabric is so well preserved that it was possible to identify it as worsted quality wool in the preliminary visual examination (Øien and Heen-Pettersen 2024, 201, 213). The underside is covered by sand/particles in a thin, dried-out black organic layer that might be human tissue, which was not further investigated.

The final find consists of seven tiny fragments of a mineral-preserved tabby-woven fabric (T28348:6, fig. 4a) that were found on the underside of a copper-alloy oval brooch (T28348:1, fig. 3a). The colour of the fragments is orange-brown, indicating that the textile's preservation is due to close association with corroding iron, probably stemming from the iron pin fastener on the reverse of the oval brooch. The fragments are highly mineralised (that is, solid pieces) but still retain excellent surface details of their fibre, thread and fabric morphology. Such details are less common with iron-preserved fibres. Results from the preliminary visual survey (Øien and Heen-Pettersen 2024, 213), provided an uncertain determination as to whether the fabric consists of animal or vegetable fibres, but leaned more towards animal. There are several areas with a green-coloured under-layer, indicating that the degradation of the textile was initially slowed down due to proximity to the copper-alloy oval brooch before it was permeated by an iron corrosion solution.



Wavenumber (cm ⁻¹)	Infrared band assignments	Data source
3360-3260	(OH) free	1-2, 5
2900	(C-H) stretching, associated with polysaccharides	1-3, 6-7
2850	(CH ₂) symmetrical stretching in cellulose	1-2, 6
1735	(C=O) in ester associated with pectin in hemp and flax, but also carbonyl groups in oxycelluloses in degraded cellulose	2-4
1639-1630	Water absorbed in lignin or cellulose	2-3, 5-6
1605-1591	(C=C) associated with lignin	2-3, 6-7
1428-1420	(C-H) in cellulose	1-2, 4-5
1375-1365	(C-H) in cellulose	1-5
1355-1335	(C-H ₂) in cellulose	1-3
1163-1155	(C-C) ring breathing, associated with polysaccharides in cellulose	1-3
1107-1104	(C-O-C) glycosidic ether band, associated with polysaccharides in cellulose	2-5, 7
1029-1025	(C-OH) in cellulose	2-3, 5
900-895	(C-O-C) stretching in cellulose	2-3, 5

Table 3: FTIR wavelength frequency and band assignments that characterise plant fibres. Data abstracted from: 1 – Garside and Wyeth (2006); 2 – Garside and Wyeth (2003); 3 – Margariti (2019); 4 – Kavkler et al. (2011); 5 – Liu and Kazarian (2022); 6 – Schwanninger et al. (2004); and 7 – Raditoiu et al. (2019)

Wavenumber (cm ⁻¹)	Infrared band assignments	Data source
3500-3100	Overlap between regions that are characteristic of (O-H) and of amide hydrogen (N-H). A sharp peak at 3285-3275 is especially characteristic of silk fibres. A wide peak at 3300-3260 is characteristic of wool fibres	1-6, 8-9
3070-2850	Saturated and unsaturated (C=H), and (C-H) region	1-2
1680-1610	Carbonyl oxygen, (C=O), in amide I, typical of wool and silk fibres	1-10
1570-1510	(N-H) in amide II, typical of wool and silk fibres	1-10
1390-1385	CH ₃ in wool	7
1235-1225	(C-N) in amid III, typical of wool and silk fibres	2-6, 8-10
1200-1000	Sulphur oxygen vibration, S-O, in wool. Peaks in this region at 1175-1160, 1124-1122, 1080-1068 and 1045-1035 are assigned to sulphur oxygen bonds in cysteine in keratin	1, 3, 10
1164-1155	(C-C), (C-OH), typical of silk fibres	4
994-990	CH ₃ in silk fibres	4-5
975-970	CH ₃ in silk fibres	4-5

Table 4: FTIR wavelength frequency and band assignments that characterise animal fibres, wool and silk. Data abstracted from: 1 – McGregor et al. (2018); 2 – Margariti et al. (2010); 3 – Belukhina et al. (2021); 4 – Garside et al. (2005); 5 – Liu et al. (2011); 6 – Margariti (2019); 7 – Liu and Kazarian (2022); 8 – Peets et al. (2019); 9 – Parker (1971); and 10 – Mabrouk (2020)



Wavenumber (cm ⁻¹)	Infrared band assignments	Data source
3436	(N-H) stretching vibration in amide hydrogen bond	3
3270-3260	C=O and hydrogen bonds	3
3060-2850	(C-H) in aromatic ring	3
1628-1623	(C=O) and (N-H)	2-4
1586-1585	(C=C) in aromatic ring	1-4
1484-1481	(C-C) in aromatic ring, and (C-H)	1-4
1462-1459	(C-C) in aromatic ring, and (C-H)	1-4
1394-1389	(N-H), (C-N) and (C-H)	1-4
1318-1315	(C-C) in aromatic ring	1, 3-4
1300-1297	(C-H)	1, 3-4
1199-1196	(C-C) in aromatic ring, and (C-H)	3-4
1175-1172	(C-H), (N-H), and (C-N)	3-4
1129-1123	(C-H), (N-H), and (C-N)	1-4
1070-1065	(C=O) and (C-C) in aromatic ring	1, 3-4
1011-1009	(C-C) in aromatic ring, and (C-H)	3-4
880-877	(C-C), δ (C-N), and (N-H)	3-4
755-751	(C-H)	3-4
714-712	(C-C) and (C-N)	3-4
698-642	(C=O), (C-C) in aromatic ring	3-4

Table 5: FTIR wavelength frequency and band assignment that characterise indigo dye. Data abstracted from: 1 – Lee et al. (2014); 2 – Lee et al. (2013); 3 – Ju et al. (2019); and 4 – Baran et al. (2010)

ATR-FTIR

Infrared spectra of two textile finds from the Skeiet 1 site are presented in figs. 5a and 5b, including the fragment (T28189:1, fig. 4c) from the top of the copper-alloy oval brooch from inhumation Grave 2 (T28189:1, fig. 3c) and the fibres (T28276:75, fig. 4e) from the pin fastener on the reverse of the Insular brooch (T28276:3 fig. 3e) from the boat burial (Grave 11). The spectra strongly conform with plant fibres when compared with a known reference of linen textile fibres (fig. 5c). The infrared band assignments of plant fibres are summarised in table 3.

The infrared spectra of both textile finds have a broad peak in the frequency region for O-H bonding in hydroxyl groups around 3330–3290 cm⁻¹, associated with cellulose. In addition, both have peaks in regions typical of polysaccharides in cellulose, with signal from C-H bonding at 2900 cm⁻¹, signal from C-C bonding in ring 1160/1157 cm⁻¹, and C-O-C bonding at 1104/1107 cm⁻¹. Moreover, both have signals in frequency regions typical of hydrocarbons in cellulose at 1335 cm⁻¹, 1368/1371 cm⁻¹, and 1421/1427 cm⁻¹. Both spectra have signals typical of C-OH bonding at 1028 cm⁻¹, as well as C-O-C bonding in cellulose at 901/899

cm⁻¹. Both spectra also had a peak in the region of water adsorbed in lignin and cellulose at 1639/1632 cm⁻¹. The fibres (fig. 4b) from the Insular brooch (fig. 3b) have a weak shoulder around 1735 cm⁻¹ that can be attributed to either ester in lignin or carbonyl in degraded cellulose. The textile fragment (fig. 4a) from the oval brooch (fig. 3a) also shows signs of lignin, with a broad peak at 1597 cm⁻¹ assigned to C=C bond in lignin, and a weak shoulder in the region for ester in lignin or carbonyl in cellulose around 1735 cm⁻¹.

The infrared spectrum of the blue-coloured textile fragment from Grave 11 (T28276.59, figure 3d) is presented in fig. 5a. The spectrum is consistent with known references of animal textile fibres, wool and silk (figs. 5b and 5c). The infrared band assignments of proteinaceous animal fibres are summarised in table 4. The blue textile fragment has several signals that are more compatible with wool rather than silk. This is shown by a broad peak in the amide hydrogen bonding frequency region around 3271 cm⁻¹. The spectrum also has characteristic peaks for amide I at 1625 cm⁻¹ and amide II at 1533 cm⁻¹, and a small peak at 1227 cm⁻¹ assigned to amide III. In addition, the sample has signals from methyl group in wool at 1388 cm⁻¹,

and signals assigned to sulphur-oxygen compounds in keratin at 1124 cm^{-1} , 1068 cm^{-1} , and 1039 cm^{-1} .

In addition, the spectrum is also compatible with the known indigo reference, suggesting that the blue colour in the textile is indigo dye (fig. 7c). The infrared band assignments of indigo are summarised in table 5. The blue textile (fig. 7a) has characteristic signals assigned to aromatic rings in indigo with peaks at 1582 cm^{-1} , 1484 cm^{-1} , 1462 cm^{-1} , 1317 cm^{-1} , at 1199 cm^{-1} , as well as 1069 cm^{-1} , a shoulder at 1010 cm^{-1} and a small peak at 698 cm^{-1} . The spectrum also has peaks at 1390 cm^{-1} , 1300 cm^{-1} , 1172 cm^{-1} , 1125 cm^{-1} and 880 cm^{-1} , assigned to nitrogen bonds with hydrogen and carbon, as well as carbon and hydrogen bonds in indigo. Peaks at 1299 cm^{-1} and 755 cm^{-1} are also assigned to indigo carbon-hydrogen bonds.

Some typical indigo characteristics are masked due to overlap in frequency regions where there are also signals from wool fibres in the sample. Some signals may also have shifted frequency due to the effects of signal overlapping. This is the case around 3264 cm^{-1} ,

where a peak appears in indigo that is attributed to carbonyl, C=O, and which is not possible to reliably identify in the sample due to the broad peak in this frequency region from the signal of the N-H bond in wool. This also applies to the peaks between 3068 cm^{-1} and 2877 cm^{-1} that are in the region for hydrocarbons that can be associated with both proteinaceous animal fibre and signals from hydrocarbons in the aromatic ring in indigo. The spectrum has two nearby peaks at 1626 cm^{-1} and 1612 cm^{-1} , which are in the region where there is an overlap of signal frequency for amide I in animal fibres and C=O and N-H bonding in indigo. As both indigo and proteinaceous fibres contain amide hydrogen bonding, the peak at 1390 cm^{-1} (region of bending of N-H bond) in the sample can be attributed to either of these substances. The peak at 1069 cm^{-1} may also possibly overlap with the peak in the frequency region for sulphur compounds in keratin, at $1080\text{--}1070\text{ cm}^{-1}$.

The infrared spectrum of the textile from the chamber tomb at Hestnes that consists of large fragments of

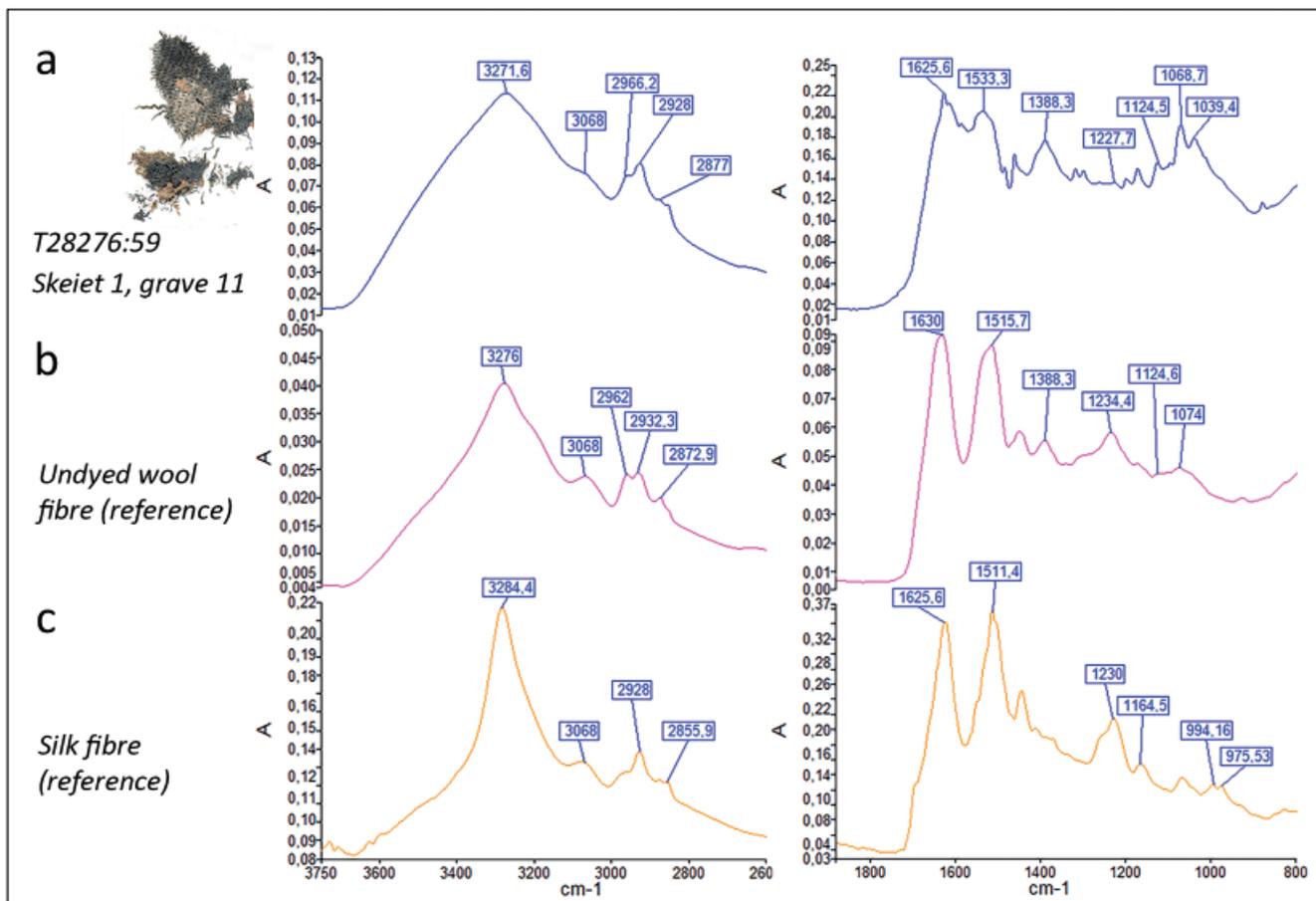


Fig. 6: Infrared spectra for the blue-coloured textile fragment compared to reference spectra for protein-containing textile fibres: a) blue-coloured textile fragment from Skeiet 1, Grave 11, NTNU University Museum T28276:59, see fig. 4d; b) modern undyed wool yarn, reference sample; and c) silk, reference from NTNU IAK Conservation Laboratory's reference library (Image: Thea P. B. Christophersen)

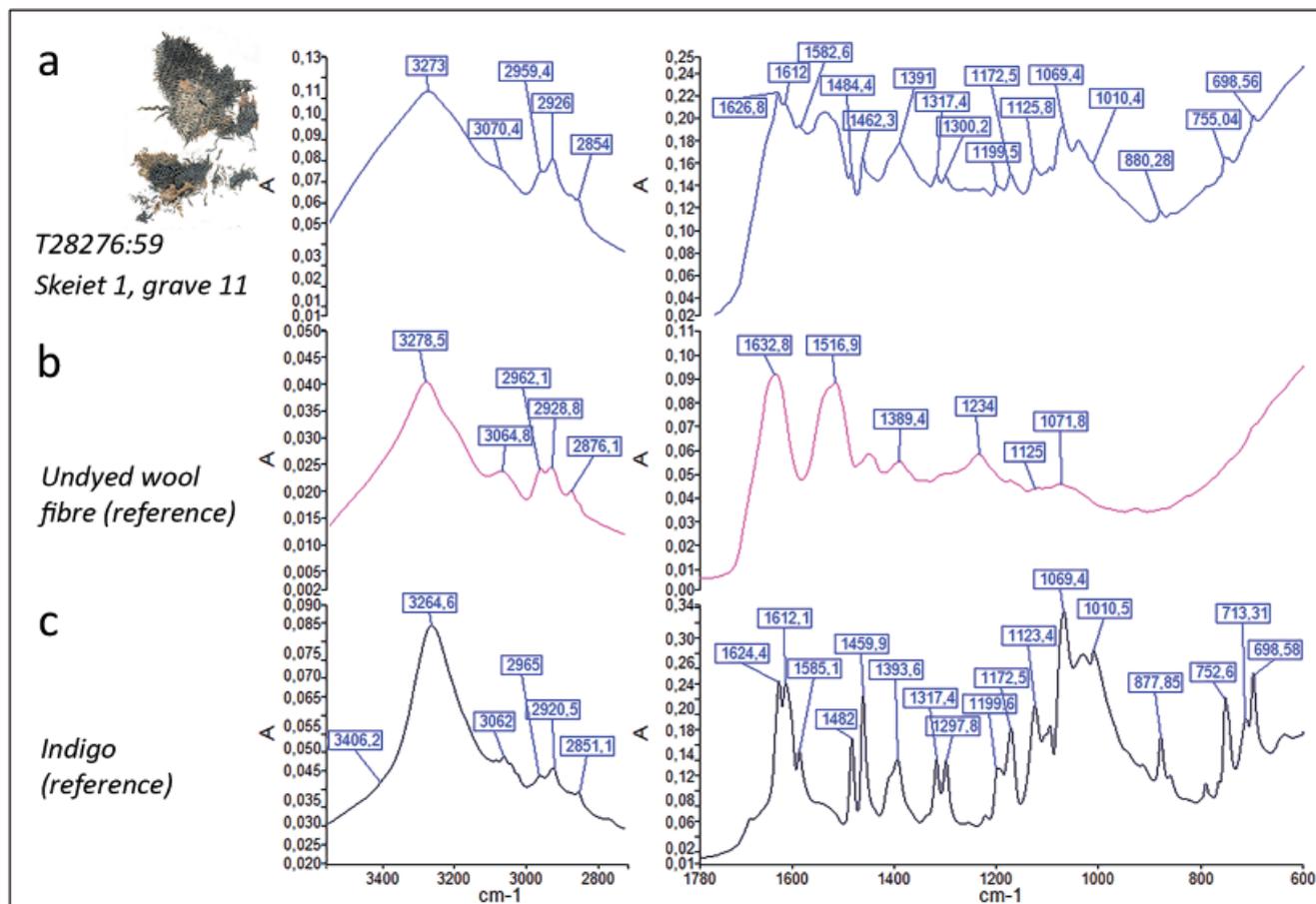


Fig. 7: Infrared spectra for the blue-colored textile fragment compared to reference spectra for modern wool and indigo: a) blue-coloured textile fragment from Skeiet 1, Grave 11, NTNU University Museum T28276:59, see fig. 4d; b) modern undyed wool yarn, reference sample; and c) modern indigo, reference sample (Image: Thea P. B. Christophersen)

woven fabric (T28348:12, fig. 4b), lacks recognisable, clear characteristic signals from typical substances in organic textile fibres (fig. 7b). This makes it challenging to reliably assign the textile to a textile fibre group. The spectrum shows some distinct characteristics that can be attributed to proteinaceous fibre when compared to the wool reference spectrum (fig. 8a). This includes a broad shoulder at $\sim 3264\text{ cm}^{-1}$, two peaks around 2930 cm^{-1} and $\sim 2852\text{ cm}^{-1}$. The sample shows one broad shoulder in the region $\sim 1597\text{ cm}^{-1}$ where in well-preserved proteinaceous animal fibre, one would otherwise see two characteristic peaks (around $1625\text{--}1615\text{ cm}^{-1}$ and 1530 cm^{-1} , as in the blue textile from Grave 11, fig. 6). The peak at 1386 cm^{-1} can be attributed to C-N in amide III. A broad peak around $\sim 1035\text{ cm}^{-1}$ can be associated with sulphur-oxygen compounds in keratin. In combination with the preliminary visual examination, it is possible to assign a tentative identification of the textile's raw material as hair/wool.

The other textile from Hestnes, the seven small textile fragments (T28348:6, fig. 4a) has few characteristic signals that can be attributed to a specific fibre group (fig. 9).

Discussion

The results of the visual and macroscopic condition analysis show that the textile fibres have different progressive stages of mineralisation of the original organic fibre structure. This is because the contact area between the metal object and the textile fibres produced a microsystem in which the process of fibre degradation in the textiles and metal corrosion interacted both physically and chemically. Often the fibre has retained an organic character in the core in the form of intact fibres (Peacock 1994; Marian and Niculescu 2016) but has metallic salts on the surface that have played a major role in its preservation. Furthermore, the occurrence of both copper mineralisation and iron mineralisation together in

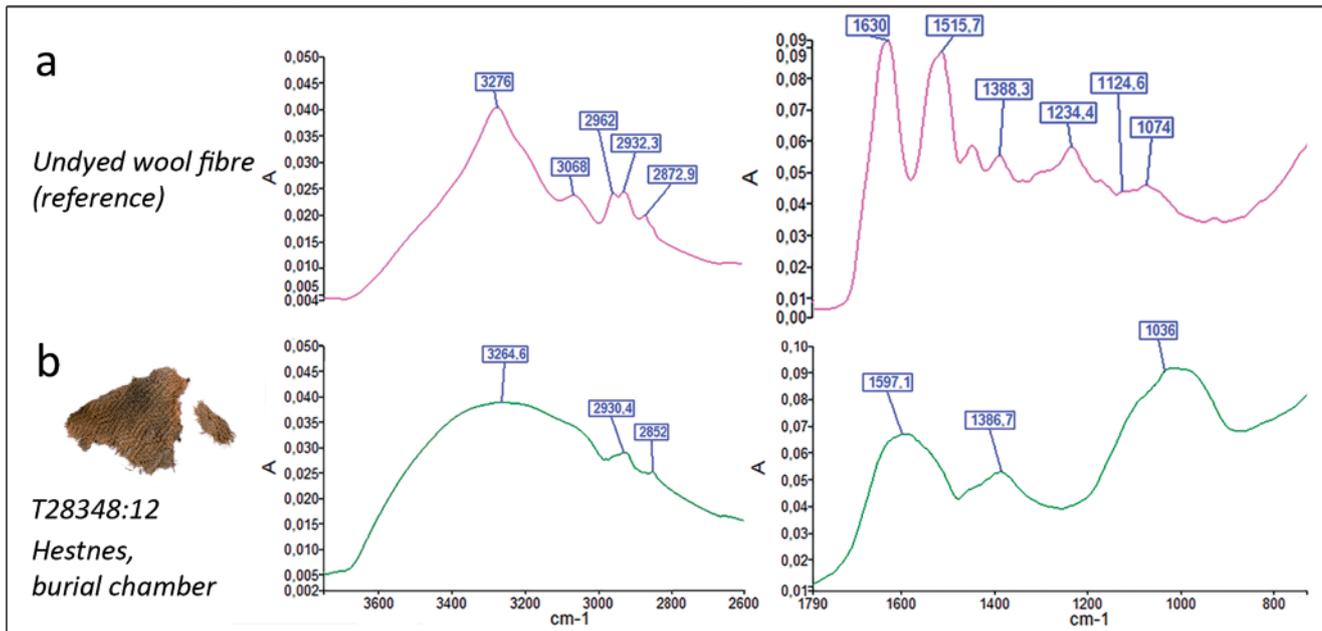


Fig. 8: Infrared spectra for textile fragment from the Hestnes burial chamber compared with a reference of modern wool yarn: a) undyed modern wool yarn, reference sample; and b) textile fragment (NTNU University Museum T28348:12, fig. 4b) found on top of trefoil brooch (NTNU University Museum T28348:3, fig. 3b) (Image: Thea P. B. Christophersen)

several of the textiles (fig. 4a and fig. 4d) illustrates that this is more common than often assumed (Mannering and Peacock 1998, 13).

The results from FTIR analysis show that the identified plant fibres in the two textile finds from Skeiet 1 (fig. 4c and fig. 4e) have clear, well-preserved cellulose/lignin signatures despite visually appearing highly mineralised. This suggests that mineralisation has not led to advanced or total replacement of the organic component of the textiles, which has also been observed in studies of other plant fibre mineralised archaeological textiles (Chen et al. 1996; Margariti et al. 2010; Reynaud et al. 2020).

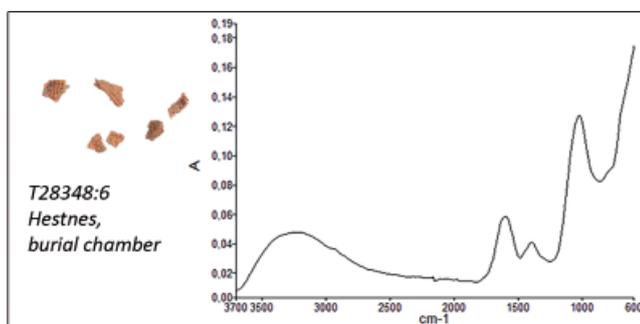
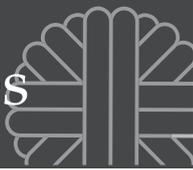


Fig. 9: Infrared spectrum for one of the seven small textile fragments (NTNU University Museum T28348:6, fig. 4a) found on the underside of an oval brooch (NTNU University Museum T28348:1, fig. 3a) from the Hestnes burial chamber (Image: Thea P. B. Christophersen)

The fact that it was possible to identify both wool/hair and indigo in the bluish-coloured textile fragment from Skeiet 1 (fig. 4d) demonstrates the additional potential of ATR-FTIR for the characterisation of dyestuffs in mineral-preserved archaeological textiles. Although both wool and silk are protein fibres, the distinctions in the spectra characteristics are significant enough to enable fibres to be identified as either wool/hair or silk (fig. 6). The results from the analysis of the two textile finds from Hestnes illustrate some of the limitations of ATR-FTIR. The unclear signals in the infrared spectra can be caused by a range of factors. The stage and nature of preservation of the textiles, their thickness, the way they have been preserved in the archaeological context, the use of dyeing mordants/assistants can all affect clarity of results obtainable with FTIR analysis (Margariti et al. 2010; Margariti 2023). Additionally, although not the case with the recently excavated E39 fragments, post-excavation storage and conservation treatment (e.g., consolidation) can interfere. The lack of clarity in the spectrum from the large textile fragment with well-preserved organic, flexible part (fig. 4b) that was identified as worsted wool in the preliminary survey, may be due to overlapping signals from other organic materials that have infiltrated the textile. Davis and Harris (2023, 16) note that mineral-preservation of textiles and the corrosion of the metals in proximity can be independent in forming the remains that survive due to the interaction between different materials,



both organic and inorganic, during degradation of the assemblage. The lack of distinct signals from the second, highly mineralised textile fragment (fig. 4a) can be due to its thickness and/or much reduced organic matrix in the fragment, for example. The specific degradation or mineralisation products that may appear in the spectra from the archaeological samples was not investigated. SEM-EDS analysis of the samples might provide some clarity and insight into what degree these affect the spectra, if at all.

FTIR spectroscopy is becoming more accessible to conservators either in laboratory acquisition or interlaboratory cooperation, especially with the introduction of compact, portable handheld spectrometers. FTIR can operate in transmission, reflectance or attenuated reflectance (ATR) modes. Peets et al. (2019) and Margariti (2019) have evaluated their application to modern and mineralised textiles, respectively. Both investigated FTIR spectroscopy in reflectance and ATR modes and concluded that FTIR applied in the contactless reflectance mode was less damaging. The pressure of the ATR arm and its potential to deform or crush a sample with the potential consequence of reducing the spectrum quality, is a drawback of consideration for ATR-FTIR. The analytical study of archaeological textiles is challenging. Multi-analytical and combined techniques are recommended. Advanced analytical techniques require specific expertise and often are invasive and/or destructive, both of which are to be avoided if possible. Bertrand et al. (2025, 124–125) review necessary considerations when evaluating non-invasive versus invasive analysis and non-destructive versus destructive analysis of archaeological textiles. The necessary handling during post-excavation study or conservation treatment unavoidably disassociates small samples from artefacts. In the present study, these were collected and stored for future analysis, which was the case for the Insular brooch (T28276:3, fig. 3e) and thread fragments (T28276:75, fig. 4e). In this study, small fibre samples (in the form of thread) were taken from three intact fragments, loose shedding fibres from the reverse of one brooch, and in the case of the fifth, a whole hard, brittle fragment was used. The first three were invasive, whereas the remaining two were non-invasive in that no sample was taken. None of the samples required preparation such as mounting for analysis, and all could be used in future chemical analysis. The analysis itself was quick to carry out but requires reference spectra (libraries) to aid interpretation.

It was possible to identify indigo in one of the textile samples. The concentrations of dyestuffs such as

indigo in textiles is low, due to indigo's high tinting strength (Kramell et al. 2015, 1039). More conclusive identification of indigo dye in ancient and historic textiles requires instrumental analysis of high sensitivity. There is a wide selection of methods to choose from with the most sensitive and widely used Gas Chromatography (GC) or GC coupled with pyrolysis (Py-GC-MS), or High-Performance Liquid Chromatography (HPLC). These, however, completely consume the sample through derivatisation or thermal decomposition to identify dye compounds (Kramell et al. 2015, 1039; Shahid et al. 2019, 63).

Conversely, spectroscopic techniques, including Raman, fluorescence and IR spectroscopy, can be employed as either partially or completely non-destructive surface techniques, making them preferable in assessment of dyestuffs in heritage textiles of limited availability (Kramell et al. 2015, 1039; Shahid et al. 2019, 63–64, 66). Fiber Optics Reflectance Spectroscopy (FORS) is another non-invasive technique, which has been investigated by Gulmini et al. (2013) and Shahid et al. (2019, 64). As for ATR-FTIR, the common disadvantage of these less-invasive techniques is that they require interpretation and comparison with known reference materials (Shahid et al. 2019, 63). The spectra may be challenging to interpret due to signal overlap from molecular bonds with similar absorbance bands from different components in the sample. Dyestuff identification is further complicated by the minute concentration of dye relative to the fiber matrix (Kramell et al. 2015, 1040). As outlined above, archaeological samples will also contain impurities from use and burial context, as well as products from mineralisation and chemical degradation of the organic textile (Bertrand et al. 2025, 128–129; Kramell et al. 2015, 1040). It is therefore necessary to base spectral interpretation on the known absorbance bands that are characteristic of the chemical compounds under study. In the study reported herein, this colour assessment challenge was overcome by employing literary references (summarised in table 5) as well as samples of known references of modern indigo dye (table 2, fig. 7).

Conclusion

The E39 Betna-Stormyra project is the most extensive archaeological excavation of Late Iron Age cemeteries and settlement features conducted in Mid-Norway. Investigations carried out in the Vinjefjord area have provided insights into past lives and societies that thrived in the region in the Iron Age and medieval period. The detailed preliminary technical survey carried out by Øien and Heen-Pettersen (2024, 200–206,

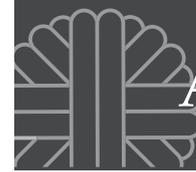


table 1) formed the basis for an evaluation of dressing practices related to mortuary rituals during the Viking Age. The investigation reported herein shows that ATR-FTIR can be successfully applied in the identification of raw materials in mineral-preserved archaeological textile fibres. The organic matrix in three of the five selected mineral-preserved textile fragments was well enough preserved in the archaeological context for the fibre type to be identified. The infrared spectra showed characteristic features that gave clear indications that the textile finds from Skeiet 1 found in association with the copper-alloy oval brooch recovered from Grave 2 (fig. 3c) and the light-coloured fibres from the copper-alloy Insular brooch in Grave 11 (fig. 3e) undoubtedly consist of cellulose-containing plant fibres.

Spectra from the visually bluish-coloured textile from Skeiet 1 Grave 11 (fig. 4d) show that the textile was produced with a protein-containing animal fibre, probably wool/hair and dyed with indigo. For the two textile finds from Hestnes, it was not possible to inconclusively determine the appropriate fibre group with ATR-FTIR. Although, the spectrum of the sample from the larger, woven fragment (fig. 4b) can be tentatively classified as belonging to a proteinaceous animal fibre group.

The study demonstrated the potential of ATR-FTIR for identifying fibre type and dyestuff in mineral-preserved archaeological textile fragments – a distinct category of archaeological textile. It is a quick, minimally to non-invasive, micro to non-destructive and low-cost analytical technique that requires minimal sample preparation. Although it may be considered only partially informative, it is useful to guide selection and sampling for further analysis that may be destructive in nature (Bertrand et al. 2025, 128). The dye results reported herein complement other studies of the textile remains from the three female inhumation burials including traditional visual technological analysis of fragments and down feather remains to conclude that the woman in Skeiet Grave 11 (T28276) was partially covered by a blue-dyed, down-filled wool quilt. To Øien and Heen-Pettersen's (2024, 209) knowledge, this is the only example of blue-dyed bedding known from Viking Age Scandinavia.

The application of FTIR analysis to archaeological textiles to identify both fibre type and dyestuffs, is not uncommon. However, its application to Viking Age period textile remains recovered from excavations in Norway has not been previously reported. Lukešová (2015; 2017) reactivated the University Museum of Bergen's (Norway) archive of textile fragments from the Viking Age that were excavated at the end of the 19th century or in the first half of the 20th century and

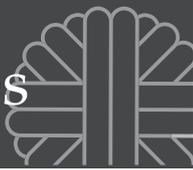
is one of the oldest archaeological textile collections in Norway. Most fragments were recovered in association with metal artefacts and many are mineral-preserved. Recent studies focused on optical and scanning electron microscopy and staining techniques (Lukešová et al. 2017, 282–283), the latter excluded mineral-preserved and carbonised fragments from investigation. ATR-FTIR spectroscopy analysis of mineral-preserved fragments in this collection could form the basis for a combined approach to complement fibre type and dyestuff identification by other analytical methods.

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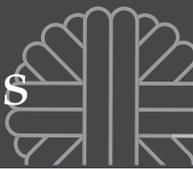
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