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Identifying Late Iron Age textile plant fibre materials with microscopy and X-ray methods — a study on finds from Ravattula Ristimäki (Kaarina, Finland)

Jenni A. Suomela¹ · Heikki Suhonen² · Riikka Räisänen¹ · Krista Wright³

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Abstract

In Finland, the earliest remains of a Christian church and cemetery date to the Late Iron Age (800–1150/1300 AD) and have been excavated in Ravattula Ristimäki, in Kaarina, southwestern Finland. In this study, seven assumingly plant fibre textile samples from individual inhumation graves were analysed to identify their materials. The aim of the study was to investigate the possibilities of identifying archaeological plant fibre samples using a three-stage procedure by observing the surface characteristics, microfibrillar orientation and cross section of the fibres via transmitted light microscopy (TLM). The identification process was based on such a TLM characterisation. Additionally, parts of the samples were studied with X-ray microtomography (micro-CT) and wide-angle X-ray scattering (WAXS) to test the possibilities of using the X-ray methods in research and to identify bast fibre textiles. Both flax and nettle were found in the samples, indicating a preference for these two fibre plants in Late Iron Age Finland.

Keywords Fibre identification · Optical microscopy · WAXS · Micro-CT · Bast fibre · Grave textile

Introduction

Archaeological background of Finnish finds

In Finland, the Late Iron Age dates from 800 to 1150/1300 AD, and Finnish prehistoric textile fragments are predominantly from this era. This is the case for at least three reasons. First, the soil is very acidic, affecting the rapid degradation of especially cellulosic material, such as textiles made of plant fibres (Tomanerä 1978b, 113). Second, during the Late Iron Age period Christian inhumation practises replaced cremation as a burial practice in Finland. Thus, textiles in most of the earlier graves became burned and charred and did not survive to the present day. Third, bronze burial goods and jewellery have played an important role in preservation by making the soil toxic for micro-organisms and thus preventing their attacks on textile materials (Tomanerä 1978a, 49; Geijer 1979, 266).

The plant fibre textile materials available in Northern Europe have been flax (*Linum usitatissimum*), hemp (*Cannabis sativa*) and stinging nettle (*Urtica dioica*) (Geijer 1979, 1). Additionally, tree bast fibres like *Tilia*, *Populus* and *Salix* have been used in ropes and nets — like in the oldest found fishing net, Antrea net from the early Stone Age

Geographical Coordinates
60°28'17.24903"
22°20'35.96304"
DMS coordinates for archaeological site of Ravattula Ristimäki were provided by Google Maps.

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which was made of *Salix* bast (Kujala 1948; Miettinen et al. 2008). Based on current pollen and macrofossil analysis, all flax, hemp and nettle were possible fibre plant choices for use as textiles during the Late Iron Age in Finland. Nettle is the only indigenous plant to Finland that grows in the wild, but both flax and hemp have travelled with humans and have been cultivated for their seeds and fibres. Pollen finds are not direct evidence of fibre use or even local cultivation, since especially hemp pollen can travel from far away (Tolonen 1978, 197). Even still, Alenius et al. (2017, 480) have obtained quite convincing proof from Lake Huhdasjärvi of human-influenced *Cannabis* pollen dating back to the Neolithic period, between 4400 and 3200 BC. The first flax pollen finds date to a much later period, discovered near Lake Ahvenainen and dating back to approximately 400 AD (Tolonen 1978, 196). The first macrofossil finds of both flax and hemp date back to the Late Roman Iron Age, with flax being discovered at Paimio Spurila (Seppä-Heikka 1985) and Salo Isokylä (Aalto 1982; Vanhanen 2020), where scholars dated the hemp seeds using accelerated mass spectrometry (AMS) to 258–425 AD (Vanhanen 2020).

Textile fragments have been found in several Finnish Late Iron Age cemeteries. The fragments are mostly of wool, since it tolerates a soil's acidity much better (Lehtosalo-Hilander 1984, 4; Good 2001, 211). However, some textile finds have been identified as plant fibres, but identifying them at the species level is demanding due to the challenges of recognising the characteristics of fibre. Even today, fibre identification methods give rise to debate, and they are constantly being refined and redeveloped. As McPartland and Hegman (2018) point out, the microscopic characterisation of flax and hemp (and nettle) has been an elusive goal for researchers for more than 150 years, including this paper's contribution to the ongoing discussion. Several researchers have helped advance the methodology for identifying bast fibres. An article by Bergfjord and Holst (2010) contributed greatly to the modern development of identification methods for bast fibres, and work has been further carried out by various researchers (e.g., Suomela et al. 2018, 2020; Haugan and Holst 2013; Lukešová et al. 2017; Lukešová and Holst 2020; Rast-Eicher 2016; Skoglund et al. 2019, 2020; Waudby 2017; Paterson et al. 2017). Reliable results demand the combining of different methods to best observe the various aspects of fibre morphology. Thus far, the best results have been obtained by combining techniques that make use of transmitted light microscopy (TLM). The methods are based on subjective visual analysis, though, with researchers studying blurred pictures in the hope of identifying fibre fragments (Lukešová and Holst 2020; Suomela et al. 2020). The other problem involves degraded samples, where the fibres have decayed either by oxidation or by microbial activity. A full methodological breakthrough still has yet to be realised,

with current methodology relying on combinations of various methods. This article introduces the application of two relatively new methods for assessing archaeological textile material: Wide-angle X-ray scattering (WAXS) and micro-computed tomography (micro-CT).

To map out previous studies, methods and identifications in the Finnish context, the impressive list compiled by Jaana Riikonen (2011) was studied thoroughly. In her article 'White Linen – Cloth of Luxury', she presents a table of 69 plant fibre textile finds, together with background information on them, from various Finnish archaeological sites. All these publicly available excavation reports, and several literature references (52 out of 69), were scrutinised to discover what in fact prior studies have said about bast fibre textile finds, including how they described the finds and the methods used for identification. The earliest of these studies was done in 1893 and the latest in 2010, so the list works as an historical survey of how archaeological textile finds have been discussed in the past.

Unfortunately, past studies rarely introduced or explained their methodology in the way current studies do, so we can only make general assumptions about how the identification systems employed by scholars related to their conclusions. Only a few of the 52 studies listed as references provided adequate details about how the researchers identified the fibres. Given the lack of adequate methods, these results have to be treated as educated guesses, even while allowing for the fact that they based their decisions on the best possible knowledge. Still, this leaves room for speculation and uncertainty.

Some general remarks can be made based on the 52 finds included in the list. Some excavation reports argue that plant fibre textiles were associated with the idea of cloth being used to cover the deceased — a covering cloth or shroud (e.g., Schvindt 1893, 154; Luoto and Fischer 1987). Birch-bark necklaces covered with linen cloth were also well represented in the finds (e.g., Schvindt 1893, 141; Heikel 1889, 192). Recent scholarship has interpreted some finds as deriving from the underdresses and wrappings of the grave goods (e.g., Tomanterä 2006, 337; Riikonen 2004, 12). In other cases, the plant fibre textile has been associated with knives or swords (e.g., Schvindt 1893, 52; Lehtosalo-Hilander 1982, 261–262). Plant fibre textiles may have had various applications in the past. Notably, all finds had a tabby (plain weave) weaving structure, all yarns were single ply, and all yarns but one (ÅM555:20 from Åland, Bender Jørgensen 1992) had a z-twist. More detailed descriptions and interpretations are available in Riikonen (2011).

Textile terminology in Finnish and English presents challenges when referring to plant material. This was the case with the above-mentioned reports, too. Linen as a general term in a contemporary context refers to cloth made of flax. However, in the eastern parts of Finland, it

has referred to hemp. Throughout history, both in Finnish and English terminology, linen has been used as a sweeping term for all white tabby woven fabrics made of plant material. This is understandable since even for an experienced textile researcher, it is not easy to distinguish between flax, nettle, hemp, or even cotton by appearance alone (Suomela et al. 2020). In Finnish, the word '*palttina*' has been used as a general synonym for linen, a word that generally refers to a tabby weaving structure. It can be difficult to fully understand the general context and whether the author meant flax, hemp, any white textile or a weaving structure. Based on this confusion, this paper uses linen only as a general term; when exact material is known, the plant's name is provided. This practice is recommendable for all textile researchers to maintain accuracy in terminology.

Background to applied methods

Optical microscopy with transmitted light enables to study fibre characteristics both in longitudinal and cross-sectional directions. Longitudinally frequency of cross-markings and dislocations can be observed, as well as malformations and fractions. It also allows to notice possible existence of calcium oxalate crystals in surrounding plant tissues (Bergfjord and Holst 2010; Suomela et al. 2018, 2020). With the modified Herzog test, the orientation of microfibrillar structure in S2-layer of the bast fibres can be determined (Haugan and Holst 2013). Cross-sectional view reveals the shape and size of the fibre and furthermore the lumen. By combining all these fibre characteristics, conclusions according to the identification of plant species can be done.

X-ray micro-CT is a widely used imaging technique in numerous fields — materials sciences, archaeology, palaeontology, medical sciences and biology, to name just a few. This is because micro-CT provides a fully 3D image of the structure of the studied small sample with micrometre resolution, and the technique as such can be considered non-invasive. The technique provides a 3D image of the sample and visualises differences in the density of the material. The fact that micro-CT's resolution is at a scale of micrometres makes it possible to observe even fine fibre structures. When oxidation and microbial activity in soil have degraded the fibres beyond a certain degree, it becomes impossible to observe these fibre structures with TLM.

With micro-CT, the sample is X-ray imaged, in a slicing manner, every few micrometres. The imaging provides grayscale images depending on how much radiation is absorbed in the material (Toda et al. 2016). The x-ray images create a stack of digital pictures that can be operated in various manners. They can be studied as single pictures or used to create 3D images.

Micro-CT is one promising method for studying textile fibres and structures in a decayed state. In textile research, it makes it possible to study textile structures and create virtual models of them in a non-invasive manner (Toda et al. 2016). Micro-CT has already been used in various types of textile studies. For example, Barburksi et al. (2015) studied sheared textile composite structures, while Toda et al. (2017) used the method to study the unevenness of yarn structures and for structural analysis and to model ropes made of natural fibre (Toda et al. 2016). Then again, Smith et al. (2013) used it to conduct experiments as a means of identifying New Zealander plant leaf materials.

WAXS provides information about the nanostructures of the fibre. With WAXS, it is possible to study the sizes and lengths of the cellulose crystallites as well as their orientation (microfibrillar angle) and degree of crystallinity. Additionally, other crystalline components of the sample, such as calcium oxalates, can be explored. Scattering studies for archaeological textile materials were introduced by Müller et al. (2004, 2006, 2007). They used synchrotron radiation to measure archaeological plant materials for identification purposes. One previous study used home laboratory WAXS equipment instead of synchrotron to study the nanoscales structures of cellulose in modern, ethnographic and archaeological fibre samples and assess whether the method would be suitable for distinguishing between flax, nettle and hemp (Viljanen et al. 2022). The results of the study are discussed in more detail later in this article.

This article examines whether micro-CT scanning and WAXS make it easier to identify fibres material retrieved from archaeological sites. Hypothetically, surface characteristics microfibrillar orientation and cross sections should be possible to observe with 3D structural data of micro-CT scanning, and WAXS should make it easier to distinguish between fibres based on their varying cellulose crystallite sizes. Part of the samples included in this study were measured with both micro-CT scanning and WAXS, and they were also studied with TLM methods to compare the results. The major advances in micro-CT and WAXS for archaeological textile research have to do with their level of invasiveness. Even though modern-day sample sizes may consist of less than 5 mm of yarn, it is still a huge improvement if the find can be kept intact.

Ravattula Ristimäki site

The fibre samples discussed in this study are based on archaeological finds from Ravattula Ristimäki, on the southwestern coast of Finland (henceforth referred as Ristimäki; Fig. 1). It was excavated by the University of Turku's Department of Archaeology between the years

Fig. 1 Location of Ravattula Ristimäki, in Kaarina municipality, Finland



2010 and 2016. The Ristimäki (lit. ‘*Cross Hill*’) site consists of a Christian inhumation cemetery and the oldest known Christian church in Finland. The burial ground was used approximately from the 1150s to the 1220s. As an archaeological period, this falls at the end of the Crusade period (Ruohonen 2017, 2019). The site represents an interesting transition period when Christianity first arrived in Finland together with its cultural phenomena, which probably affected the material and structural choices of textile material, such as the weave and pattern of the garment. From a textile archaeological standpoint, it is interesting to see through future studies how this turning point from paganism to Christianity affected the sartorial culture and material choices.

The Ristimäki site has provided interesting new textile archaeological finds — for example, a hemmed piece of sleeve from an undergarment and woollen leg bands (Riikonen and Ruohonen 2016; Riikonen 2017), socks from woven cloth (Riikonen 2019a) and a complete project on reconstructing ancient dress based on finds in

grave 41 (Riikonen 2019b). Most recently, the fur and feather finds have been studied by Kirkinen et al. (2020).

This study is based on seven, assumingly plant fibre textile samples from Ristimäki, stored in the Department of Archaeology’s collections at the University of Turku (TYA). All seven of them were studied with optical microscopy, while two were also studied with micro-CT and three with WAXS. TYA 914:1607:8A was the only sample studied using all three methods. The research questions are as follows:

- 1) What information can be gathered from the Late Iron Age textile samples with the optical microscopy methods, and does the applied three-stage identification protocol work for archaeological samples?
- 2) What information can be obtained from the Late Iron Age textile samples with the micro-CT and WAXS methods?

Table 1 Ristimäki fibre samples discussed in the study and the performed analyses

| Sample | Excavation data | Optical microscopy | WAXS | Micro-CT | Images |
|---------------------|---|--------------------|------|----------|-------------|
| TYA 912:523D | Found partly underneath the head of the deceased, on top of a bronze-coated knife sheath. The sheath was covered with a bronze plate | X | X | - | Figure 3a–d |
| TYA 914:1316:1E | The find was preserved via a tortoise brooch and found on top of it. The brooch was situated on the chest of the deceased | X | - | - | Figure 4a–d |
| TYA 914:1316:2A | The sample is from the same grave as the previous one. It was found in close connection on top of a penannular brooch situated in the middle of deceased's chest | X | - | - | Figure 5a–d |
| TYA 914:1607:8A | Found in close connection with a twisted bronze bracelet | X | X | X | Figure 6a–d |
| TYA 933:214:16A-16B | Sample is a fragment of plant fibre tabby among a cluster of skull pieces between 16A and 16B parts of the headgear. The headgear was situated on the face of the deceased | X | - | X | Figure 7a–d |
| TYA 933:214:9:50 | Sample is plant fibre tabby attached to a glass bead from a necklace. Next to the bead lay a silver coin and on top of the necklace a woolen cloak decorated with bronze spirals. These metal items probably explain the survival of the fragment | X | X | - | Figure 8a–d |
| TYA 933:864:8 | Sample is from the right shoulder of the deceased, close to the penannular brooch. It was not part of the woven structures, more likely from a braid or cord | X | - | - | Figure 9a–d |

Material

The Ristimäki textile fibre samples discussed in this study are presented in Table 1. The excavation data is provided by Jaana Riikonen.

Methods

Optical microscopy and sample preparation

When preparing the samples for microscopy, they were carefully studied and imaged with a Leica MZ6 stereomicroscope and Leica DFC420 camera with 5 megapixel resolution. At this stage, textile structures and yarn characteristics were observed. The longitudinal and cross-sectional fibre characteristics of the samples were observed using a TLM Leica DM4500P with rotating stage and polarised light features. The microscope was integrated with Leica application suite LAS Core 4.5.0 software using the same Leica DFC420 camera. Both transmitted and polarised light were used in the analysis. The identification protocol was conducted by following the procedure firstly introduced by Suomela et al. (2018).

Improvements were made to the cross-cutting technique during the identification protocol by embedding the fibres in LR White epoxy resin. The epoxy blocks were roughly cut using a Leica EM Trim into truncated pyramid shapes, approximately 500 µm by side, and then straightaway sliced with a Leica Ultra-cut microtome into 2 µm cuttings with a DiATOME

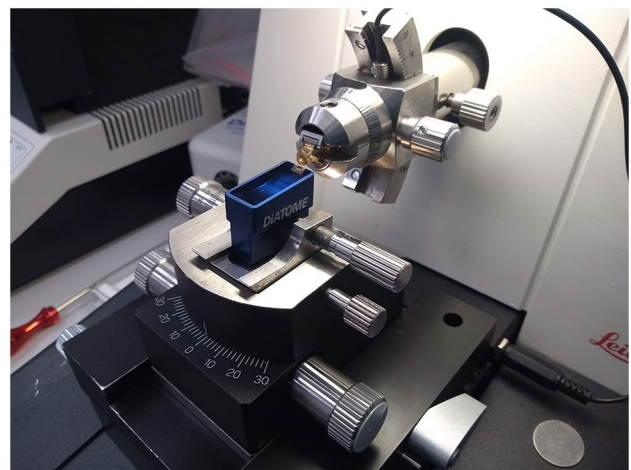


Fig. 2 Ultramicrotome Leica Ultra Cut with DiATOME Histo Diamond Knife

Histo Diamond Knife (45°, 4 mm) (Fig. 2). The procedure was the same one used for preparing cuttings for the transmitted electron microscope (TEM), but it was possible to bypass the trimming part to save time, and the slice thickness was far larger. The cuttings were transferred from the knife's water pool to glass slides using Perfect Loop equipment. Experiments were conducted with Entellan New, milli-Q water, paraffin oil and cover glass, but the best contrast was achieved when the cuttings were allowed to dry on the glass slide and observed as such without the cover slide.

Contrary to the suggested setting in standard SFS-EN ISO 20706–1:2019 (2019) for the modified Herzog test, the marking colours of blue and yellow for the microfibrillar twist are appearing the opposite. In the TLM Leica DM4500P microscope lambda plate is adjustable from 45° to 135°. Colours appear in 45° as in standard, and in 135° the opposite (Suomela et al. 2018). In this study, S-twisted fibres appear blue in 0° and yellow in 90° position.

Wide-angle X-ray scattering (WAXS)

WAXS is a technique used to determine the degree of crystallinity and the crystalline structures in polymers. In this study, three of the samples, TYA 912:523D, TYA 914:1607:8A and TYA 933:214:9:50, were studied with this method to compare if the average width of the cellulose crystals was in line with the identification made with optical microscopy and to determine what other results this method could provide. The samples were measured between two mylar foils attached to a metal washer with double-sided tape. The measurements took place in the X-ray laboratory of the Department of Physics at the University of Helsinki. More detailed information about the measurements, equipment and results have been provided by Viljanen et al. (2022).

Micro-computed tomography (micro-CT)

In this study, the samples TYA 933:214:16A-16B and TYA 914:1607:8A were imaged using a GE nanotom. The scanning was done in the Helsinki X-ray laboratory of the

Department of Physics at the University of Helsinki. With the TYA 933:214:16A-16B sample, a voltage of 60 kV was used with a current of 140 μ A. With these settings, an accuracy of 4 μ m pixel size was achieved. For the TYA 914:1607:8A sample, a voltage of 60 kV was used with a current of 240 μ A, resulting in a sample accuracy of 2 μ m. The number of projections for both of the samples was more than 1200, with a 360 degree rotation. The samples were imaged in Eppendorf tubes. The 3D models and other analyses based on the X-ray imaging were done using Fiji ImageJ software.

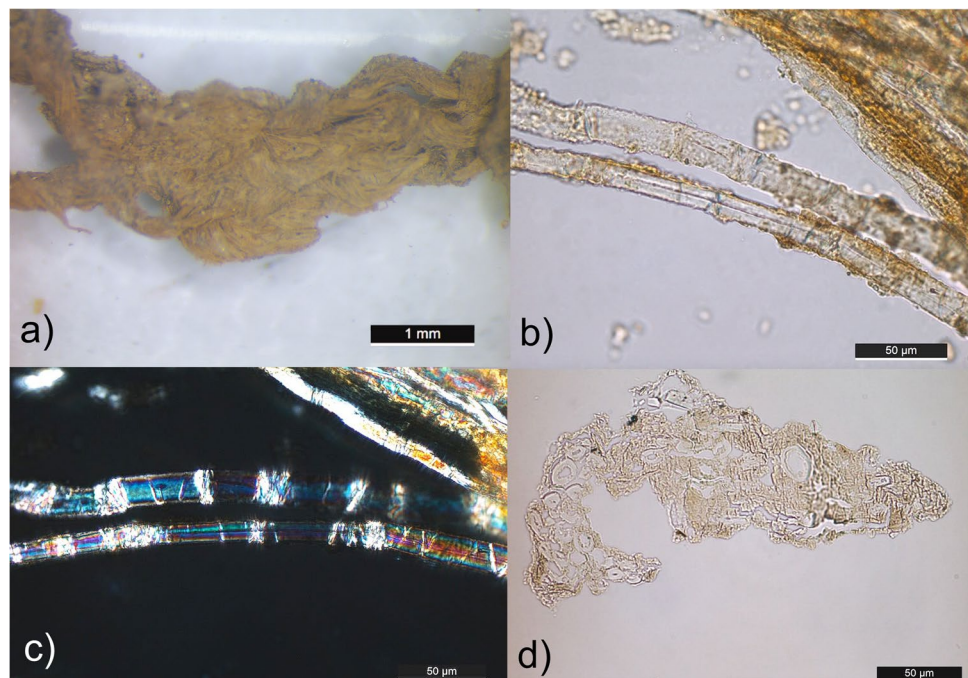
While fibre morphology is difficult to observe in degraded fibres, we wanted to discover just what fibre characteristics could be detected when using micro-CT. We wanted to know if it would be possible to observe longitudinal and cross-sectional characteristics up to the level of a microfibrillar orientation without destroying the archaeological find.

Optical microscopy results

Sample TYA 912:523D

Sample TYA 912:523D itself was quite mineralised, but a tabby weave structure and tight Z-twist in the single-ply yarn was recognisable (Fig. 3a). The microscopic analysis revealed that the sample was badly decomposed, with only a few pieces of single fibres having been preserved. The fibres were eroded, covered with residue and decomposed

Fig. 3 Optical microscopy images of TYA 912:523D



in a shrunken and dried manner (Fig. 3b). In polarised light, it was possible to observe dense cross markings and sections containing malformations (Suomela et al. 2020) (Fig. 3c). The Herzog test showed a clear S-twist in the microfibrillar orientation. A cross section of the sample revealed multiple shapes and lumen sizes, but also angular shapes with small lumen, which is typical only for flax (Fig. 3d). The WAXS results for this sample are explained in a separate section.

Sample TYA 914:1316:1E

Sample TYA 914:1316:1E had a dense tabby weave structure woven with Z-twisted, single-ply yarn (Fig. 4a). The microscopic analysis revealed mineralised residues, but surprisingly many fibres were in good condition for analysis with optical microscopy (Fig. 4b). Cross markings appeared in dense form, and strongly squeezed sections also appeared especially in polarised light (Fig. 4c). A clear S-twist was observed when using the modified Herzog test. Some of the fibres were relatively narrow, less than 15 µm. Cross sectioning only gave impressions of the fibres as holes, with most being kidney-shaped, which is recognisable for nettle (Fig. 4d).

Sample TYA 914:1316:2A

Sample TYA 914:1316:2A was only a bundle of fibres, and the spinning direction was not detectable (Fig. 5a). The fibres were in good condition, though numerous lens-shaped mould spores were visible (Fig. 5b). In polarised light, it

was possible to observe dislocations, cross markings and shrinkage caused by degradation (Fig. 5c). The results from the Herzog test demonstrated an S-twist. Cross sectioning showed only a few fibres, mostly flattened similarly to nettle fibres (Fig. 5d).

Sample TYA 914:1607:8A

When conducting a stereomicroscopic analysis of sample TYA 914:1607:8A, it was possible to detect a densely woven tabby structure and single fibres with elasticity still present. The sample was attached to some organic material. In general, the sample was dried in shape and the single strands of yarn were flattened (Fig. 6a). In addition to well-preserved fibres, presumably microbial residue was clearly visible (Fig. 6b). Cross markings were visible, and the modified Herzog's test yielded an S-twisted result (Fig. 6c). Cross-sectional observations clearly showed angular shapes with small lumen typical of flax (Fig. 6d). The WAXS and micro-CT results for this sample are explained in separate sections.

Sample TYA 933:214:16A-16B

Sample TYA 933:214:16A-16B was tightly attached to a lump of clay and had been mineralised into a state where only the structure of the weave was still visible on the surface (Fig. 7a). The fragment crumbled into dust, but it still contained a few fibres on the inside in

Fig. 4 Optical microscopy images of TYA 914:1316:1E

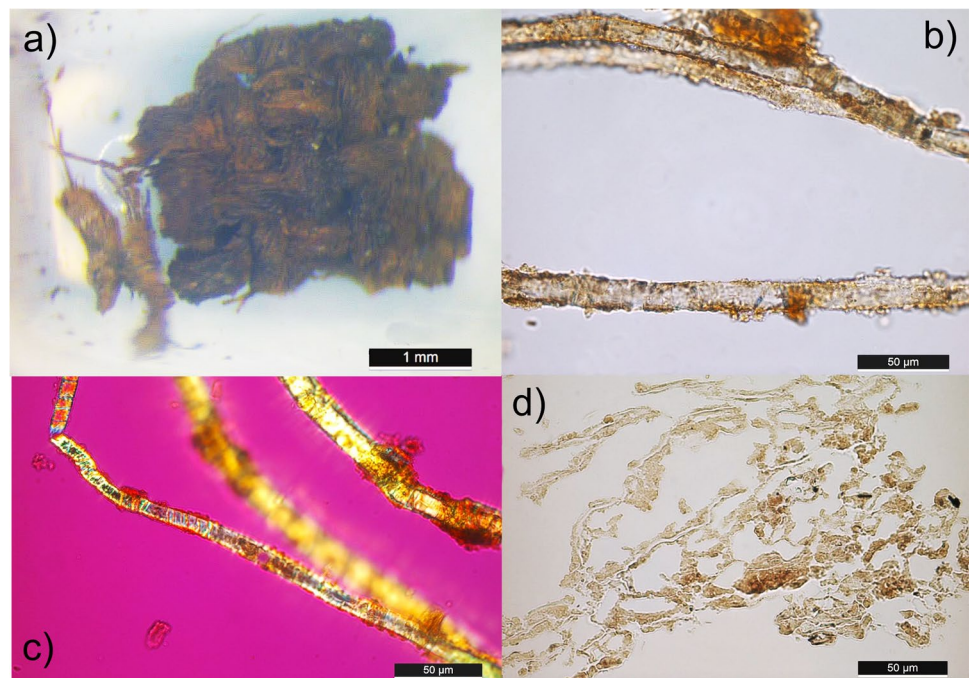


Fig. 5 Optical microscopy images of TYA 914:1316:2A

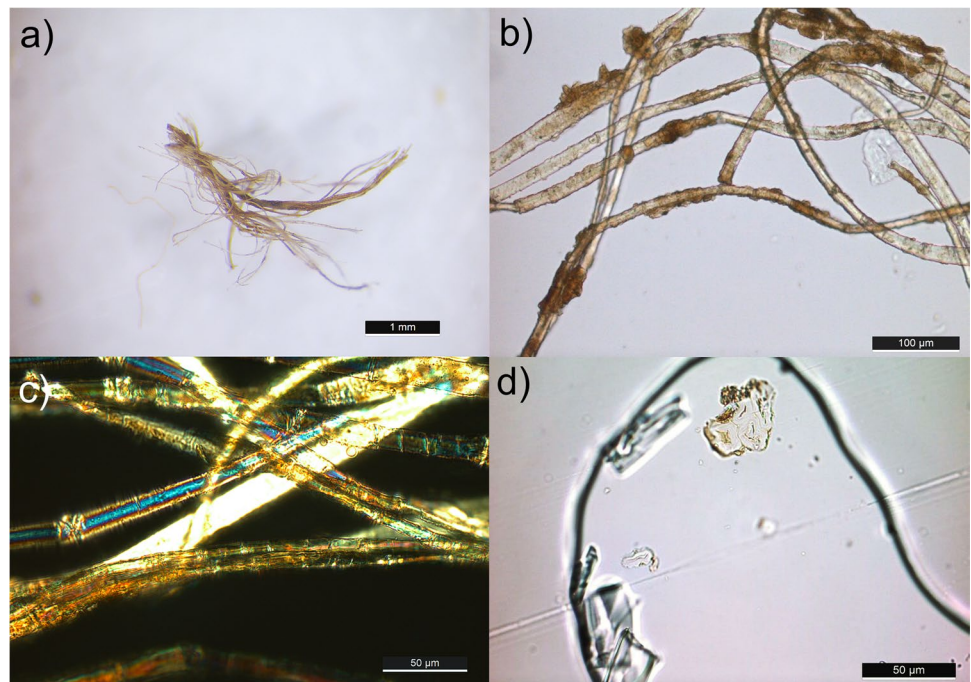
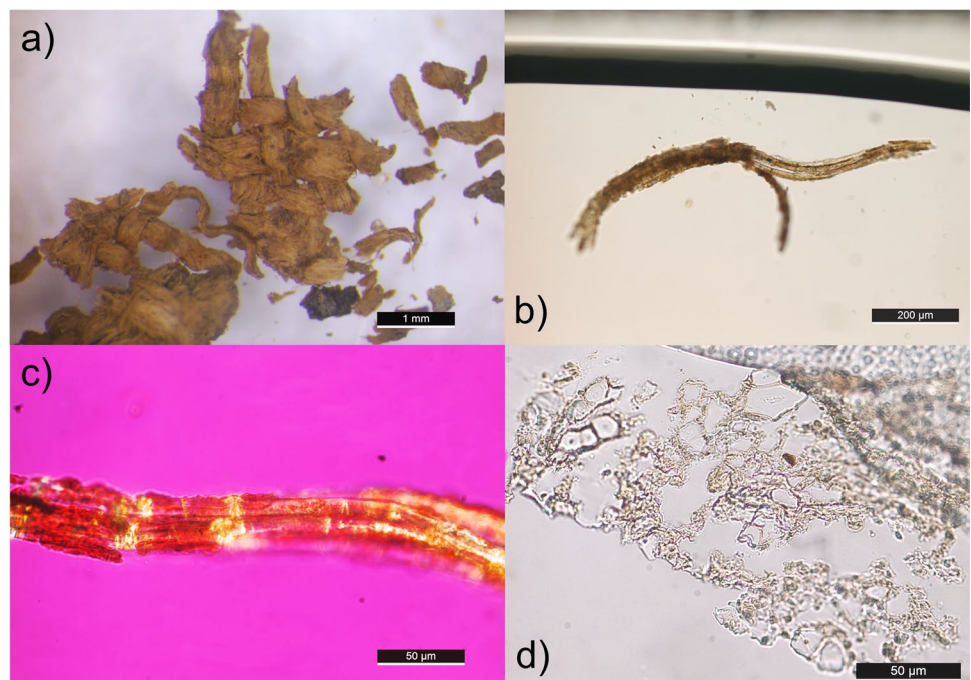


Fig. 6 Optical microscopy images of TYA 914:1607:8A



good enough condition to study with optical microscopy, even to conduct cross sectioning (Fig. 7b). The modified Herzog test yielded results for only one spot, so it cannot be taken as reliable (Fig. 7c). The result was an S-twist. In cross section, only the outlines of the fibres could be observed. They were clearly angular, like in flax (Fig. 7d). Attention should be paid to the exceptionally small fibre diameter, less than 10 μm .

The micro-CT results of this sample are explained in a separate section.

Sample TYA 933:214:9:50

Sample TYA 933:214:9:50 was in surprisingly good condition, with the fibres being elastic and moist and tabby structure contained several single-ply yarns with a Z-twist

Fig. 7 Optical microscopy images of TYA 933:214:16A-16B

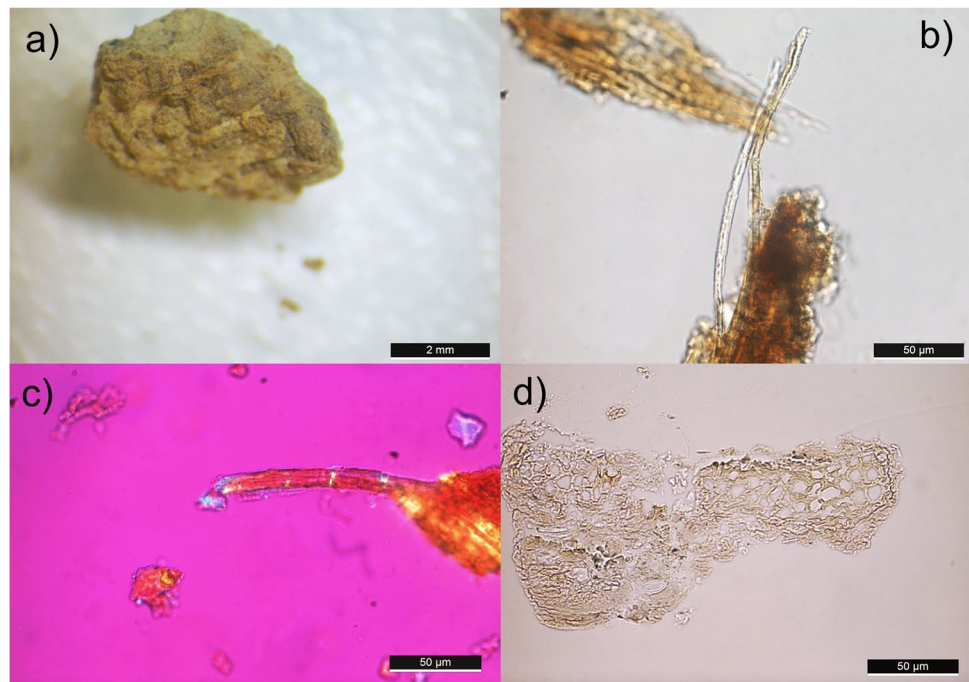
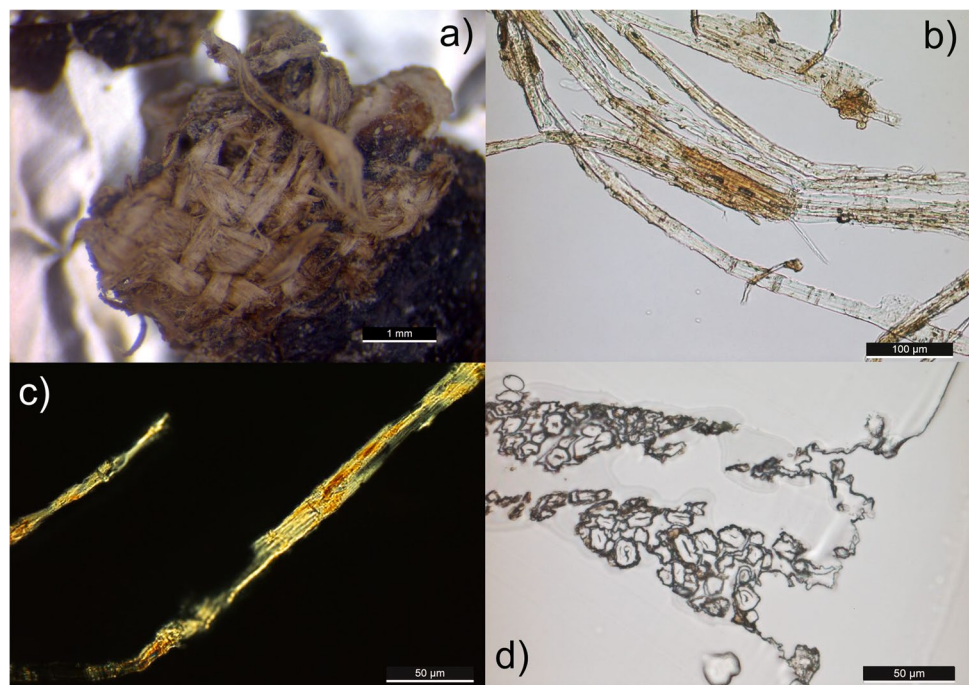


Fig. 8 Optical microscopy images of TYA 933:214:9:50



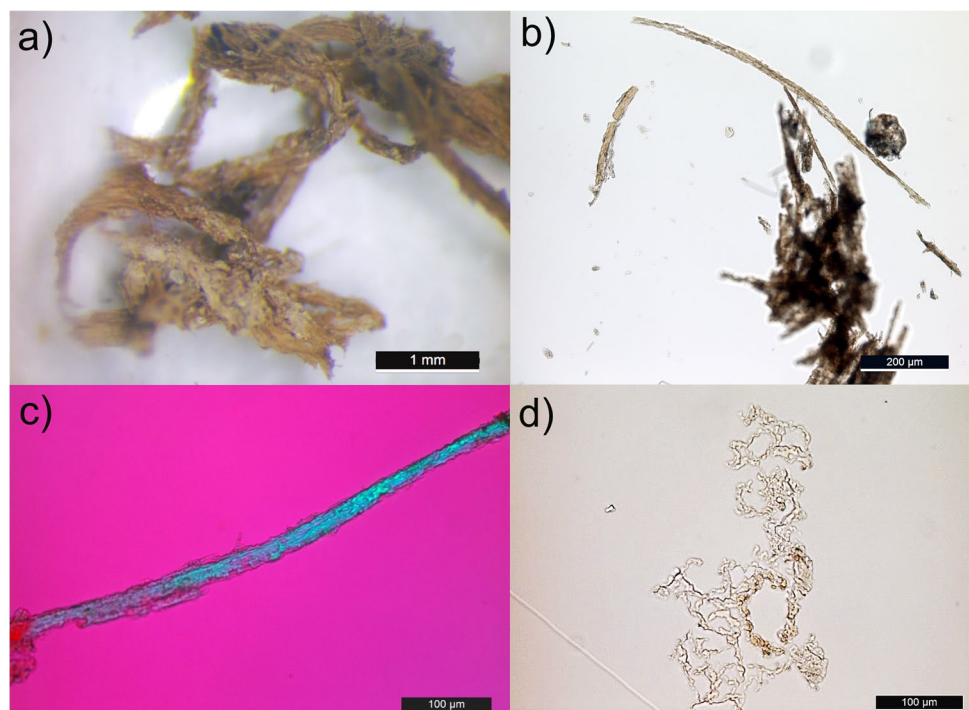
(Fig. 8a). The thread count of the tabby weave was 20–25 y/cm. Dislocations, cross markings and squeezed parts were all observed (Fig. 8b). Even though the sample appeared quite intact when viewed under the stereo microscope, it was clear that the cellulose structures had been badly degraded in some parts (Fig. 8c). It was still possible to conduct a reliable Herzog test, which yielded S-twist results. Cross sectioning revealed oval-shaped fibres with flattened lumen, clear indications that the sample was

nettle fibre (Fig. 8d). The WAXS results for this sample are explained in a separate section.

Sample TYA 933:864:8

When placed under a stereomicroscope, it became readily evident that sample TYA 933:864:8 was badly mineralised (Fig. 9a). The fibres were eroded, filled with holes from microbial activity (Fig. 9b and 9c). A certain scale pattern could be

Fig. 9 Optical microscopy images of TYA 933:864:8



faintly detected, and in polarised light at the spot where the lambda plate was attached, the colour changes behaved like in protein fibres (Fig. 9c). After cross sectioning, only the outlines were left, but it showed that the diameter of the fibres was closer to that of animal hairs and wool than to plant fibres (Fig. 9d).

WAXS results

WAXS results for the 2D and integrated scattering patterns, cellulose crystallite sizes and observations of possible Cu-compounds contained within fibres from the TYA912:523D, TYA914:1607:8A and TYA933:214:9:50 samples are all discussed in an article by Viljanen et al. (2022). Sample TYA 912:523D had a crystal width of 6.1, ± 0.5 nm, with optical microscopy revealing it to be flax. Sample TYA 914:1607:8A had a crystal width of 5.6, ± 0.2 nm, and it also was identified as flax using optical microscopy. The crystal width in sample TYA 933:214:9:50 was 7, ± 0.2 nm. As Viljanen et al. (2022) point out, in the case of bast fibres the sizes of the cellulose crystallites cannot be used as an identification tool, as they may be quite similar across all the studied fibres, while on the other hand, they can vary even within the sample plant. For some reason, crystal widths are much larger in cultural historic and archaeological samples than in modern reference samples. Based on the results presented by Viljanen et al. (2022), in the case of modern fibres, the observation of calcium and silica compounds could be

used for identification purposes. Thus, in this study, the WAXS patterns of the three TYA samples were carefully compared to the theoretical diffraction patterns of selected Ca/Si compounds (Fig. 10). The compounds were selected based on their abundance in plants (Franceschi and Horner 1980; Dietrich et al. 2003).

The scattering patterns of the TYA samples included numerous diffraction maxima besides the reflections of crystalline cellulose. Unfortunately, none could be indisputably connected to Ca/Si compounds, and thus, the reflections could not be used for identification purposes.

Micro-CT results

Sample TYA 933:214:16A-16B was in such a degraded state that optical microscopy could not give that reliable results when the sample was taken apart for preparation. Micro-CT scanning made it possible to study the weave and yarn structure without destroying the sample, which had totally, at least at a visual level, degraded to a pseudomorph state and merged with the clay fragment to which it was attached. Average diameter for bast fibres like flax, nettle or hemp varied based on who had conducted the research (Bergfjord and Holst 2010), but it can be estimated as approximately 15 μm . The voxel size during scanning was 4 μm , which meant that nanostructures such as microfibrils, or even cross sections, could not be observed (Fig. 11). The scanning made it possible to produce a rotating 3D simulation (Online Resource 1) and structural sectional view (Online Resource 2) of the sample.

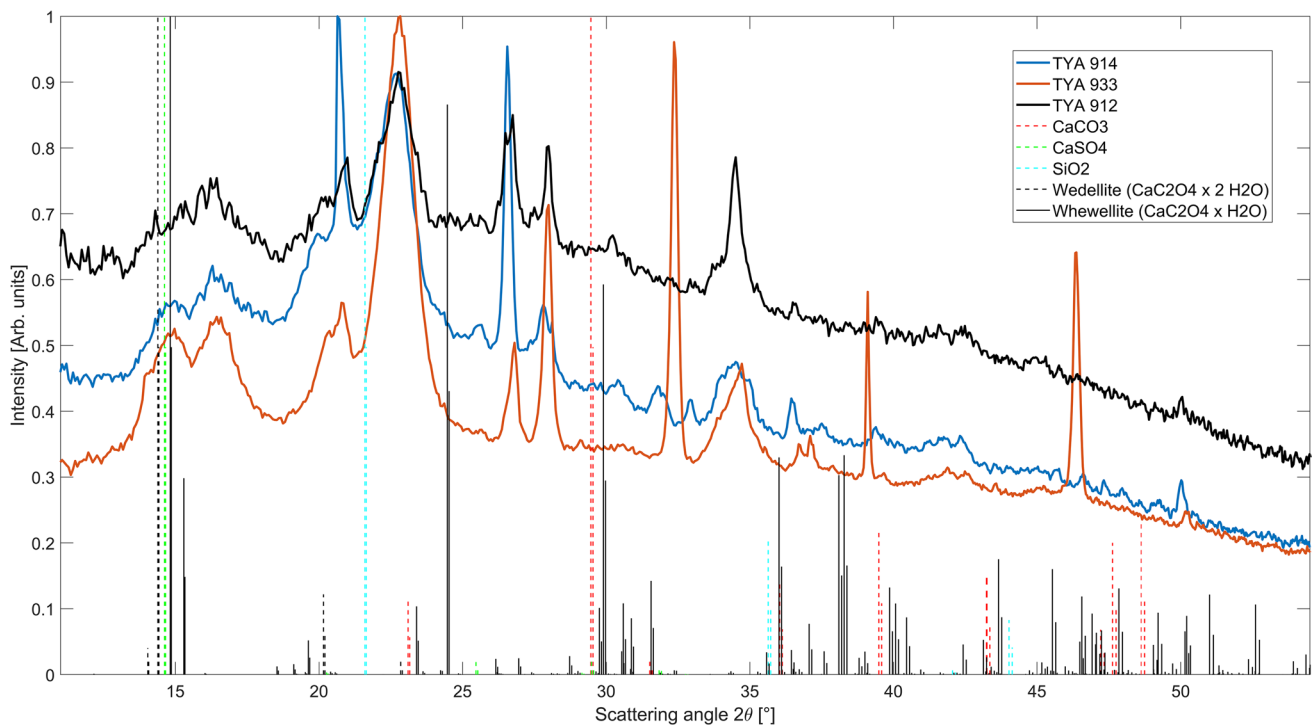
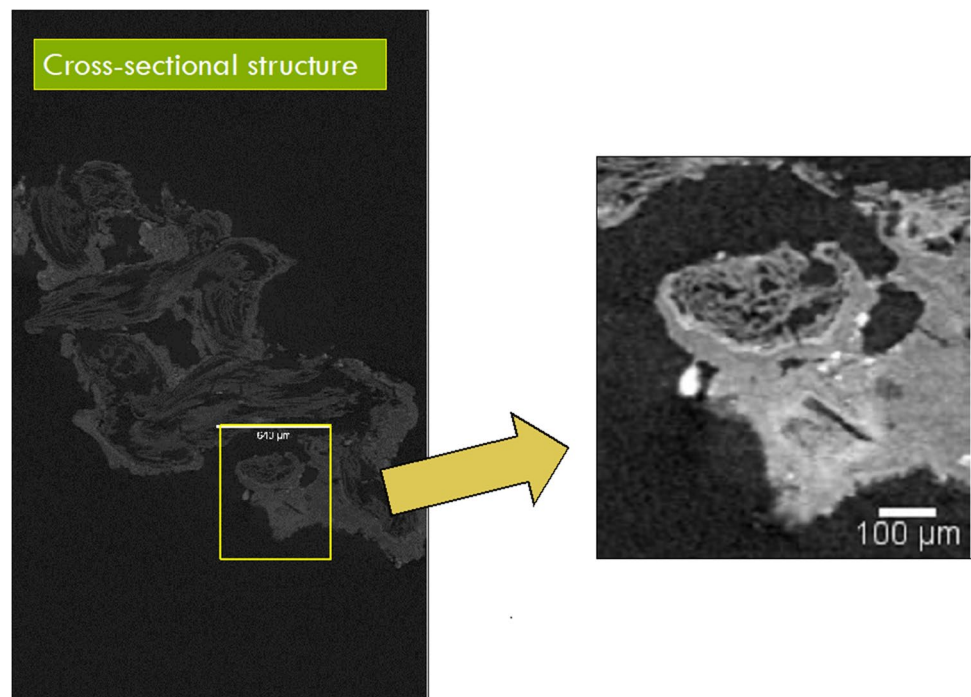


Fig. 10 Scattering patterns of samples TYA 912:523D, TYA 914:1607:8A and TYA 933:214:9:50 with the theoretical diffraction peaks of the calcium and silica compounds

Fig. 11 Cross-sectional structure of TYA 933:214:16A-16B with micro-CT scanning



With sample TYA 914:1607:8A, the voxel size was reduced to 2 µm. The fibre's characteristics could not be detected with longitudinal projection (Fig. 12), but the cross-sectional images revealed angular-shaped fibres with

small and round lumen — typical of flax fibres (Fig. 13). Unlike sample TYA 933:214:16A-16B, this sample was not attached to any external surface, such as a piece of clay, and it was more difficult to create a clear, rotating 3D projection

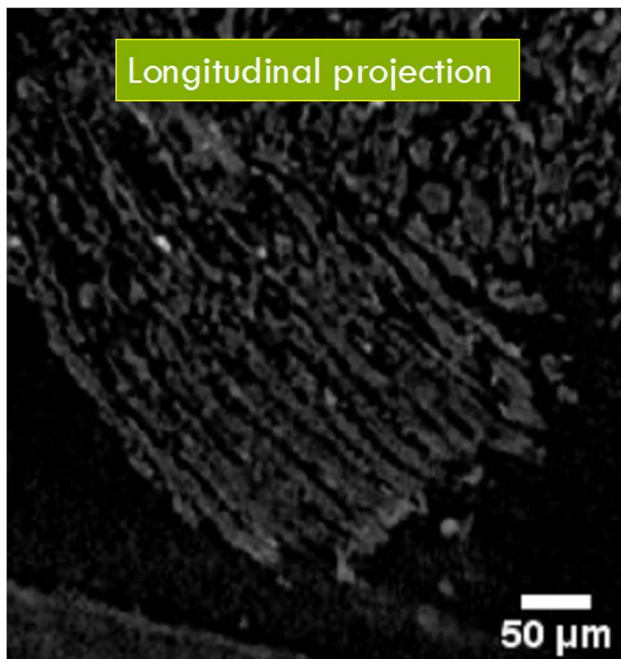


Fig. 12 Longitudinal projection of TYA 914:1607:8A

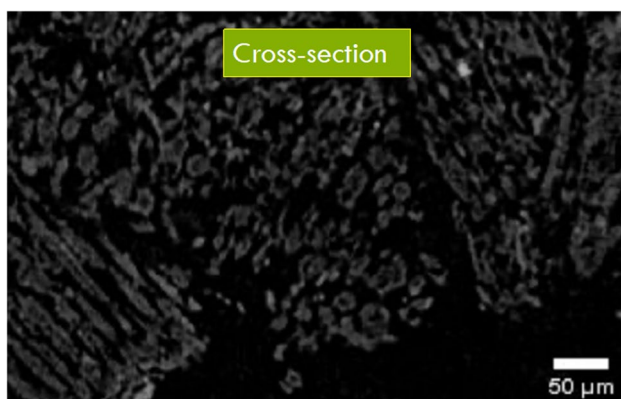


Fig. 13 Cross-sectional structure of TYA 914:1607:8A with micro-CT scanning

(Online Resource 3). The rotating projection and cross-sectional view (Online Resource 4) both showed a structure of cloth layers, produced by sample pieces being located one after the other in the Eppendorf tube.

Discussion

At the Ristimäki site, the plant fibre textile artefacts had survived surprisingly well given the circumstances. The fragments were small and seemingly in a degraded state, but every sample contained at least a few fibres with the morphological characteristics still present, as observed

with optical microscopy. Most of the samples (TYA 912:523D, TYA 914:1316:1E, TYA 914:1607:8A, TYA 933:214:16A-16B and TYA 933:214:9:50) had a recognisable weaving structure of tabby/plain weave. All the strands of yarn in the samples were single-ply strands and spun in the Z-direction. Sample TYA 933:214:16A-16B was so mineralised that the spinning direction was clearly visible only with the aid of micro-CT scanning. The conclusions regarding the structure of both textiles (plain weave and Z-twist) are in line with previous Finnish finds (Riikonen 2011) and with earlier Scandinavian finds (Lukešová et al. 2017; Bender Jørgensen 1992).

We can only speculate on how the textiles were used due to small fragmentary finds. Based on the interpretations of earlier finds listed by Riikonen (2011) and the accumulated knowledge about the finds, some suppositions can be made. More reliable conclusions require holistic analysis of the graves, though, which is not possible based only on fragmentary textile samples with limited background information. The location of sample TYA 912:523D has resulted in various possible interpretations. The fragment was found on top of a knife sheath that was partly under the head of the deceased. Since the actual placement of the fragment is unknown, it may have been part of the sheath or wrapping of the knife if it had been placed there as grave goods. Or, it may have been from the headgear of the deceased or from a covering cloth. Sample TYA 914:1316:1E was found on top of a tortoise brooch, and based on earlier finds, it most likely was part of a covering cloth. Sample TYA 914:1316:2A was found in the same grave as the previous one, but on top of a penannular brooch. Both textile finds have been identified as nettle, so it is possible that they are from the same covering cloth. Sample TYA 914:1607:8A was connected with a spiral bracelet, suggesting that it was part of a long-sleeved underdress or a shirt. The position of sample TYA 933:214:16A-16B gives rise to two possible interpretations. The fragment was found among skull pieces from the facial area, where the headgear had been placed. It can either be from the headgear or from a covering cloth. The actual location of sample TYA 933:214:9:50 in relation to the body of the deceased is unknown, but the site contains numerous related factors. A glass bead, a silver coin and a woollen cloak with bronze spiral decorations may give hints about its origin, but interpretation in this case would only be mere guesswork. The last sample, TYA 933:864:8, situated close to a penannular brooch on the shoulder, proved to be of animal origin and thus beyond the scope of this paper.

The identification protocol, created and tested using modern reference samples and museum textiles (Suomela et al. 2018, 2020) for flax, nettle and hemp, is also suitable for archaeological textile fibre samples so long as the sample consists of at least a few fibres that have not been degraded.

The modified Herzog test yielded results of an S-twist for all the samples, meaning that they are either flax or nettle fibres. This is consistent with current knowledge about the arrival of fibre plants to Finland based on pollen and macrofossil records. The records, however, do not provide information on whether the plants were used for textiles, even if Tolonen (1978, 196) has suggested that *Linum* pollen grains in lake sediments are direct evidence of flax retting. Based on our finds, we can state that in the Late Iron Age, at least flax and nettle were used as textile fibre materials. Samples TYA 912:523D, TYA 914:1607:8A and TYA 933:214:16A-16B were shown to be flax and samples TYA 914:1316:1E, TYA 914:1316:2A and TYA 933:214:9:50 were nettle based on the three-stage protocol (Suomela et al. 2018).

Using cross-sectional characteristics as an identification tool has recently been criticised by Lukešová and Holst (2020). They, with good cause, point out that due to natural variations, different lumen sizes and cross-sectional shapes occur within species. This natural variation is a well-acknowledged fact in bast fibre studies. If sample images of flax, nettle and hemp presented by Lukešová and Holst are observed, it is actually simple to recognise typical features for each species. Flax has polygonal shape with narrow lumen, hemp is also polygonal with wider lumen and nettle has kidney shape with long and flatten lumen. In some cases, Lukešová and Holst had pinpointed space between fibres as a lumen or clearly malformed fibres as representatives of certain shape. Therefore, bast fibres have to be studied holistically and the identification cannot be based on only one method. When fibre diameter is close to 50 µm, which is the case in many of their examples, it should be clear that the fibre is not typical representative of its species. Hence, this point should be stressed especially with archaeological samples that are small in quantity, since cross-sectional interpretations should be based on average characteristics of the fibres in the cross cutting, not on a single fibre.

The micro-CT method appeared to be more suitable for studying textile structures than the characteristics of single fibres. The achieved resolution (2 and 4 µm) is not yet at a sufficient level of accuracy for detailed bast fibre morphology. With nano-CT, the resolution can be cut down to 100 nm, and this methodology is already available (Kuan et al. 2020), but the equipment is still not commonly used. The method would be advisable for future studies of archaeological textile samples because of its non-invasive and non-destructive qualities. It would help researchers understand complex textile structures existing in a delicate state due to degradation or, for example, covered with soil and threatening to break apart if cleaned further.

The WAXS measurements did not yield direct results for bast fibre identification in the case of archaeological fibres. However, the notion that calcium oxalate crystals or a lack of them, which some researchers consider an identification

marker, can be detected from modern fibres with this method without destroying the sample by plasma ashing (Bergfjord and Holst 2010). However, as demonstrated in this study, the calcium oxalate crystals can interfere with other mineral crystals from the find site when archaeological finds are in question. When this interference from the soil minerals can be omitted, the dimensions and orientations of the plant mineral crystallites reveal added information to the identification question. This was demonstrated in the case of the modern nettle samples in the study by Viljanen et al. (2022). This observation requires further research in the case of archaeological samples. Other WAXS applications for studying archaeological fibres may include, for example, assessing the degradation processes in bast fibres by measuring their relative crystallinity.

Conclusions

This paper is the first to study flax and especially nettle with an open methodology, demonstrating that the fibre plants were used in a Finnish context in the Late Iron Age period. The two X-ray-based methodologies presented in this paper provided useful new information about the textile finds, but unfortunately, they did not solve the identification problem with the bast fibres in question — so the search must go on. Though, there were clear advantages in using these new identification tools for conducting archaeological textile research. The extent and quality of plant fibre material usage in textiles in Late Iron Age Finland require further study and hopefully better-preserved finds, meaning larger fragments with indicative textile structures. This paper has revisited previous plant fibre textile finds, studied new ones and provided tools for future studies.

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Declarations

Competing interests The authors declare no competing interests.

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