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Research methods for heritage cotton fibres: case studies from archaeological and historical finds in a Finnish context

Jenni A. Suomela[1*], Mira Viljanen[2], Kirsi Svedström[2], Krista Wright[3] and Sanna Lipkin[4]

Abstract

Cotton (*Gossypium* species) was used as textile fibre already in the early Indus culture, and since then it has been cultivated in Tropical and Subtropical regions around the whole planet. The species *G. hirsutum* is nowadays the dominant cotton crop with more than 90% of the world market, while *G. barbadense*, *G. herbaceum* and *G. arboreum* combined, the other cultivated species of *Gossypium* genus total a minor part of world's cotton production. Even in places where cotton was not cultivated, it could be an important trade item and income source for local textile centres, with the imported raw cotton lint being spun, woven and for some part exported from such sites around the globe. This all occurred far away from Finland, until changes brought by the development of long-distance trade and the Industrial Revolution. Based on archaeological finds, cotton as a textile material reached Finland relatively late, in the early Middle Ages. The article focuses on the problematic nature of identifying these cotton finds: whereas modern cotton fibres are easy to identify, the archaeological finds can at first sight be confused with bast or undegummed silk fibres. This issue will be approached through reviewing recent Finnish cotton finds in heritage textiles. Additionally, the article examines whether the four cultivated cotton species could be differentiated using both classical and newly developed fibre identification methods, such as optical microscopy methods, a Scanning Electron Microscope (SEM), Fourier-transform infrared spectroscopy (FTIR) or Wide-Angle X-ray Scattering (WAXS).

Keywords Cotton, Fibre identification, Optical microscopy, SEM, WAXS

Introduction

In recent years, cotton has been identified from numerous archaeological and heritage textiles found in Finland. Cotton became a common textile material in Finland relatively late compared to most of the world, and research on it has mainly been a marginal topic at best, especially regarding archaeological finds. Yet, a more detailed study of heritage textiles and textile fragments has stimulated curiosity as to why the visual appearance of the cotton fibres often clearly differs from the fibres shown in textbooks. A brief literary review of cotton's global history will shed more light on the origins of these Finnish finds. Additionally, the peculiar appearance of the cotton fibres raised an important question: could they be assigned to the different cotton species? The aim of this study is to deepen understandings of these Finnish cotton finds and examine whether it is possible to identify cultivated cotton species like *Gossypium arboreum*, *barbadense*, *herbaceum* and *hirsutum* solely based on the fibre characteristics. The species assignment of archaeological cotton fibres is a central problem for a precise understanding

*Correspondence:
Jenni A. Suomela
jenni.suomela@helsinki.fi
[1] Department of Education/Craft Studies, University of Helsinki, Siltavuorenpenger 10, 8, 00014 Helsinki, Finland
[2] Department of Physics, University of Helsinki, Gustaf Hällströmin Katu 2, 64, 00014 Helsinki, Finland
[3] Nanomicroscopy Center, Aalto University, Puumiehenkuja 2, 15100, 00076 Espoo, Finland
[4] Faculty of Humanities/Archaeology, University of Oulu, FI-90014 Oulu, Finland

of trade routes and cultural links in the Antiquity or the more recent past [67].

Relevance of cotton in present-day textile production

In the year 2021, a total of 118.4 million tonnes of textile fibres were produced, of which cotton production accounted for 24.4 million tonnes [1], or one fifth (21%) of total production but 81% of all natural fibre production [1]. This makes cotton clearly the most produced natural textile fibre in the world and the most important non-food cash crop in the world [2]. Cotton is grown on 2.5% of the world's arable land [3].

Production on this scale also has its downsides. Irrigation for one kilogram of cotton lint requires between 7 000 and 29 000 L of freshwater [4]. Seventy-three per cent of cotton harvest comes from irrigated fields, and 53% of cotton farmland is irrigated [4]. Eleven per cent of pesticides and 25% of insecticides (which is a type of pesticide) in the world are used in cotton cultivation [2]. This also makes cotton production a huge environmental issue.

According to Clay [2], cotton cultivation was in the past more diverse. White cotton was preferred for its dyeing properties and plants with woody stems were eliminated because they presented difficulties for the machinery. Production on a rural scale in terms of yield and farming practices typically was more sustainable [2].

Early history of cotton around the globe

Cotton requires a subtropical climate, available between 32 and 35 degrees latitude in the Southern Hemisphere to 37 degrees latitude in the Northern Hemisphere [5]. The temperature cannot drop below 10 °C and should preferably stay above 15 °C. The maturing time for cotton is from 160 to 200 days [5]. With these parameters, and given the multiple varieties of cotton, it is understandable that cotton has been cultivated in numerous places around the world for thousands of years.

The global history of cotton originates rather simultaneously in South Asia, with the species *G. arboreum* and in South America with *G. barbadense*. The earliest cotton fibre finds are from Mohenjo Daro, in the Indus River Valley, and Merhgarh, west of the Indus River on the Kachi Plain. Cotton textile fragments were first discovered in Mohenjo Daro during an excavation led by Sir John Marshall and were dated back to 3000 BCE [6]. At Merhgarh, mineralised cotton fibres were found inside a copper bead dating as far back as 5000 BCE [7]. Viot [8] has suggested that already in the 3rd millennium BCE, the Indian peninsula was a cotton manufacturing and exporting centre especially to the Mediterranean region.

In ancient China, the species *G. arboreum* began to be widely cultivated from ca. 1300 CE in eastern China, and *G. herbaceum* appears somewhat earlier, ca. 500 CE, in Central Asia, including the region of Xinjiang [9]. Cotton production had arrived from the Indian subcontinent to China already before 200 BCE [5]. It took until the times of the Yuan dynasty (1271–1368 CE), though, for cotton production to boom in China and surpass ramie (*Boehmeria nivea*) as the principal fibre crop for textiles [5]. Cotton production arrived relatively late in Japan, in the sixteenth century CE, though the first literary mentions of cotton in Japan are from the ninth century CE [5, 10]. Many wild cotton species have originated in Australia, but cotton cultivation only began with the British colonisation in the mid-nineteenth century CE [11, 12].

Stephens and Moseley [13] have reported *G. barbadense* finds from coastal Peru as early as 2500 BCE. Splitstoser et al. [14] have pushed the dating much further back to 4900 BCE. A cotton ball dated to 5500 BCE was discovered in Coxcatlan Cave, in Mexico [15]. According to Beckert [5], cotton was being cultivated in Mesoamerica already by 3500 BCE, with the earliest archaeological cotton yarn find dating to between 1500 and 1200 BCE. The *G. hirsutum* species has its origin in Mesoamerica, but other varieties have also been used for textile production. Finds from Oaxaca, dating to 700–1300 CE, are probably the annual *G. laetifolium* or perennial, tree-like *G. punctatum* species [16]. Mexico and Peru were the cotton manufacturing centres of America, but the Navajo and Hopi peoples also started to produce cotton probably as early as 300 BCE [5].

Northeastern Africa is the cradle of the *G. herbaceum* species. Nubian cotton finds have been dated to between 500 BCE and 300 CE, with cotton production having arrived in West Africa by the eleventh century CE [5]. According to Bouchaud et al. [17], cotton remains have been discovered in Nubia dating from first century BCE to first century CE, in south Sudan from the first to fifth centuries CE, and in Egypt from the second to fifth centuries CE. The earliest West Asian (including Syria, Israel, Jordan, Saudi Arabia, Oman and Yemen) finds date to between the first and third centuries CE [17].

According to Beckert [5], archaeological cotton finds from the area of Iraq date back to 1100 BCE. Signs of cotton cultivation have been found in ancient Persia, Mesopotamia and Palestine, just before the Common Era, while the evidence from Anatolia is slightly younger. Liu et al. [18] claims that, based on microfibre finds from Tel Tsaf, in the Jordan Valley, the earliest cotton finds from West Asia can be dated to approximately between 5200 and 4700 BCE. According to Kirkinen et al. [19], these kinds of microfibre finds from soil samples should be approached with caution, though. The control samples in their study did not include any fibre material, which is strange, whereas airborne fibre particles in general are numerous and contamination during various stages of working at the excavation site or conducting analyses in the laboratory is possible and even probable.

Even though some cotton was imported to Europe in Greek and Roman times, it had little meaning before the turn of the tenth century CE, when its production spread to coastal Spain and Sicily during the Islamic invasion [5]. By the twelfth century, cotton production had become a major industry in northern Italy [5]. Production expanded to southern Germany after the mid-fourteenth century, and already then, trading routes for fustian (half cotton, half linen fabric) had been established all the way to Tallinn in northern Europe [20]. By the fifteenth century, cities in southern Germany were competing with the Italian markets, including in Eastern and Northern Europe. Both Italian and German production relied on Levantine cotton (present area of West Asia and Egypt), and the cotton species grown there was *G. herbaceum* [5].

According to Lemire [21], terrestrial trade routes in the thirteenth and fourteenth centuries extended all the way from India to Novgorod. This might have enabled Indian cotton goods, made of *G. arboreum*, to enter at least Eastern Europe. Finally, starting in the early seventeenth century, the trading routes of the East India Company made cotton an ordinary commodity in Europe [22].

Cotton in Finland

Cotton as a textile material arrived on the Finnish peninsula relatively late. The earliest finds thus far are from the relic assemblage at Turku Cathedral. Red cotton fabric, possibly used to line a relic casket, has been 14C-dated to 1290–1330 CE [23]. A small (67 × 29 mm) cotton fustian piece was found in the fillings of a relic bone package. It was 14C-dated to between 1290–1410 CE [24]. The Cap of St Birgitta of Sweden contains a cotton band 14C-dated to 1290–1410 CE [25]. The earliest piece of cotton fabric found in an archaeological context is contemporary with the previous item, discovered at Valmarinniemi (late 13th to fourteenth century CE), in the northern Bothnian Bay region of Keminmaa, discussed in more detail later in this paper.

Finland only gained its independence from Russia in 1917 and previously was, until the early nineteenth century (1809), under Swedish rule, which meant all formal documents had to be written in Swedish. We have done a cursory examination of a small sampling of randomly selected estate inventory deeds (n = 76) between the years 1632 and 1810. Probate inventories became increasingly common around the seventeenth century [26]. In the inventories we studied, cotton is mentioned for the first time in 1710, in the probate inventory of Gabriel Esping and his wife Maria from Piikkiö, near Turku. They had two *Natt Kjortells*, one of *tryckt cartun* with *bomull stoppat* (Swedish; calico nightgown with cotton filling) and another of *röd bomull cartun* (red cotton calico).

As is often the case, inconsistencies in the textile vocabulary hinder the trustworthiness of textual records [27]. Just as the term fustian has changed its meaning over time from flax warp and cotton weft to heavy cotton fabrics [28], so too the Swedish word *cartun/cattun* has different meanings. Usually it means printed cotton calico fabric, but the Russians manufactured a similar type of printed fabric made of flax, which was referred to with the same term [29]. Additionally, the often-used Swedish term *lärft*, which translates as linen, refers to both cotton and flax (and other bast fibres; see [30]), having more to do with plain woven whitish material than the actual origin of the material.

The probate inventory find is supported by Virrankoski's [31] hypothesis that cotton was rare luxury material in eighteenth century, though records also show that raw cotton was being imported to Oulu by the end of that century. Finally, in 1828 the first Finnish cotton factory, Finlayson, was established and production started on a large scale in the 1840s [31]. Due to climate conditions, the raw material had to be imported, as it had been the case in Italy and Germany before.

Four commercial cotton species

Before domestication, cotton plants had a shrub-like appearance and were perennials. Through domestication, though, the plants lost their photoperiodism and became annuals and compact in form [8]. According to Viot [8], the species cultivated in Africa and Asia in the Antiquity and until the Columbian Exchange were *G. arboreum* (*cv. indicum*, *burmanicum*, *bengalense*, *cernuum*, *soudanense* or *sinense*. according to epochs and regions) and *G. herbaceum* (cv. *acerifolium*, *persicum*, *kuljianum* or *wightianum*). The cotton fibre that can be found in European archaeological contexts before the Columbian Exchange and before the domination, beginning in the second half of the eighteenth century, of the American allopolyploid species in world global production could only come from the African and Asian diploids *G. arboreum* and *G. herbaceum*.

The *G. arboreum* species originated in South Asian, and it is around 1.8 m in height with yellow or purple flowers with short fibres [5]. It thrives in warm and humid conditions [8]. It is also called tree cotton, Ceylon cotton, Indian Cotton Tree, Nankeen cotton, Oriental cotton and red-flowered cotton tree [8]. Two thousand years ago, based on archaeological evidence, *G. arboreum* was grown in an area ranging from the Persian Gulf, on the South Asian peninsula, all the way to the area known today as northern Laos and Vietnam [8]. Until the nineteenth century, the South Asian peninsula was the leading centre of cotton manufacturing [5].

The South American *G. barbadense* species is a small bushy tree with yellow flowers and long fibres [5]. It is commonly referred to as Pima or Egyptian cotton—even though it originated mainly in the Peruvian Andes and Caribbean [32]. The name Sea island cotton has also been used, referring to cotton production in South Carolina, Georgia and Florida in the late nineteenth century [33]. It has an extra-long lint and a silky lustre [32]. However, due to its low yield, long growth period and susceptibility to various diseases, it is grown on less than 5% of the area cultivated for cotton production worldwide [34].

The *G. herbaceum* species is 'an African version' of the *G. arboreum* species [5]. It is also called Levant cotton, Syrian cotton, Arabian cotton, Maltese cotton or short-staple cotton [8]. Two thousand years ago, it was cultivated in an area ranging from present-day Egypt to north Saudi Arabia, Iraq and Iran [8]. In addition, an undomesticated wild variation, *G. herbaceum* subsp. *africanum*, is still found growing in southern Africa, around the Tropic of Capricorn [8]. The environmental requirements for growing it include rather dry conditions, but it tolerates low and high temperatures [8].

The *G. hirsutum* species originated in southern Mexico and Guatemala [35]. It dominates the markets nowadays and is also known as American Upland (as opposite to the Sea Island grown on coastal, low altitude enviroments); its heart-shaped leaves are hairy on the lower surface and its capsules are oval in shape and almost the size of an apple [5]. More than 90% of all cotton production uses this species [5]. The following numbers give context to when and how rapidly *G. hirsutum* invaded the markets—only 3% of all cotton grown in India in 1947 was *G. hirsutum*, whereas the percentage had increased to 53% by year 1970 and to 90% by the year 2007 [36].

Properties of cotton fibres

Some fundamental biological differences must be noted at this point, for instance that *G. arboreum* and *herbaceum* are diploid plants, whereas *G. hirsutum* and *barbadense* are allotetraploids [37]. According to Viot [8], it is difficult to distinguish between *G. arboreum* and *herbaceum*: both have a quite similar plant and seed morphology as well as fibre characteristics.

Since cotton fibres are the most cultivated natural textile material, their properties have been studied thoroughly for industrial purposes. Modern cotton fibres can be analysed in numerous ways, such as by their strength, fineness, elongation at break, thermal resistance, mean length, uniformity/circularity index, micronaire value, maturity ratio or short fibre content e.g. [38, 39].

The above-mentioned methods for analysing cotton fibres just attract attention to the sample size that is needed to observe properties of fibres found at archaeological sites. For the purposes of this article, yarn samples 2–5 mm in length were analysed; it was not possible to analyse longer samples from the textile material due to ethical reasons. Hence, any methods requiring a sampling of full fibres had to be excluded from the study. Taking deterioration into account, characterisations based on strength or elongation at break, should be excluded too.

Prior studies of cotton fibre characteristics have shown that the properties of cotton fibres differ depending on the maturity of the fibre. Immature fibres have a thin fibre cell, whereas more mature fibres have thicker cells and narrower lumen [7, 40]. In addition, the increasing number of convolutions is related to the fibre's maturity [41]. Hearle [42] states that malformations, such as crimps, kinks and waviness, on the surface of the fibres results from the drying in maturing process.

Usually, no distinction has been made between the species – according to Viot [8], reliably it has only been done thus far with respect to Nubian seed DNA [43] and based on fibre characteristics [9] noted in western China. In resent paper from Milon et al. [67], differences between species were studied from the seed morphology. Only in the study from Liu et al. [44] the tensile strength and elongation of single fibres from the four cultivated species were measured and compared.

Fibre characteristics affect the spinning properties and quality of the final product. For example, the famous Dhaka muslin widely used by the upper class in 18th-century Europe and already by the ancient Greeks and Romans and locally in the Mughal Empire disappeared from the markets long ago and it is no longer possible to reproduce it. The reason is twofold. First, the *G. arboreum var. neglecta* (locally *Phuti karpas*) species needed to weave this extremely fine fabric is no longer cultivated. Second, the skills of the weavers do not meet the standards needed to produce it. [45]

Research material

Modern reference fibres, 4 species

Reference material on the cultivated species was collected from two different sources. Two different *G. barbadense* samples (taxon numbers 1998–0267 and 2008–0392) and one *G. herbaceum* (1998–0268) sample are from the Kaisaniemi Botanic Garden (University of Helsinki). The other source is from small-scale seed distributors in the Etsy marketplace, who provided seeds that still containing some fibres. Seeds containing fibres from all four cultivated species are referred to as G. *arboreum, G. barbadense, G. herbaceum and G. hirsutum.*

The *G. barbadense* 1998–0267 and *G. herbaceum* 1998–0268 plants had grown for years next to each other, and it is possible that they have got mixed somehow, according to the gardener (personal communication), there are no visible plant morphological differences between them, and they cannot be considered as pure representatives of their species. Though, cross-pollination should not have been possible, because *G. barbadense* is genetically a tetraploid and *G. herbaceum* is a diploid. The trustworthiness of the seed distributors is based on the large variety of plants they regularly sell and their professionalism.

Recently identified Finnish cotton finds

The second part of our research material consists of the cotton finds recently identified from Finnish heritage textiles or archaeological finds (Fig. 1). Information on all of the material finds has already been published or is in the process of being published at the moment. The aim is not to reproduce the results, but to discuss the difficulties in the identification processes.

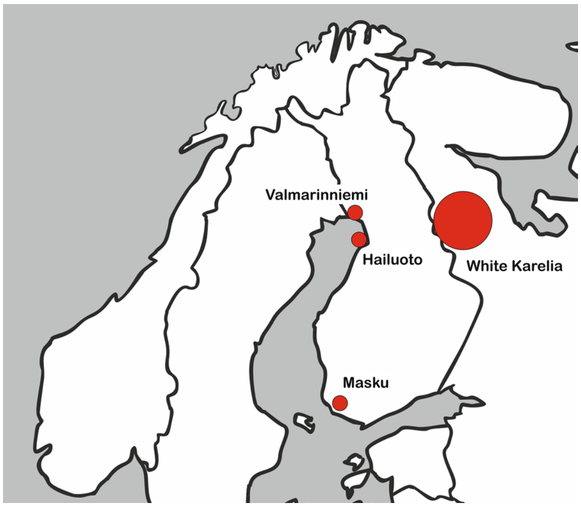

Fig. 1 Map of the heritage research material (image: Jenni A. Suomela)

Valmarinniemi, early fourteenth century

The Valmarinniemi site is located in the delta area of the Kemijoki River, in the northernmost Bothnian Bay region of Keminmaa. The site of the burnt Catholic church and cemetery was excavated by researchers from the University of Oulu in 1981. The church was destroyed by fire either in 1390 or in the early fifteenth century. The burial site (101) yielded a piece of tabby woven fabric, discovered on a metal ring of a belt worn around the waist of an approximately 20–30 year old woman (Fig. 2). Based on accelerator mass spectrometry (AMS) analysis and the grave finds, the burials at Valmarinniemi have been dated from the late thirteenth century to the early fourteenth century [46].

Antependium from Masku church, fifteenth century

An antependium was discovered in the bell tower of Masku Church (Southwestern Finland) in 1871 (Fig. 3). This large (276 × 207 cm), mediaeval Catholic fragment of ecclesiastical wool textile has been dated to approximately 1440 CE, and it was made by the Bridgettine Sisters in an abbey in Nådendal, nearby Masku. Currently, the antependium is listed as object H1223:1 in the Collections of the National Museum of Finland. In 2016, a reconstruction project was begun to produce a replica of the antependium [47]. At the same time, the textile was sampled to verify the materials to be used in the replica. As a result, cotton fabric was identified in the small white fabric pieces used to create white dots for the eyes in the tails of the peacocks (Fig. 4) [47]. Sample 12 was from the black peacock and sample 13 from the green peacock. Both peacocks were sampled from both yarn systems.

Fig. 2 The piece of cotton fabric (KM39304:1544, sized 2.8 × 2.4 cm) was found preserved on the belt's metal ring (images: Sanna Lipkin)

Fig. 3 Masku antependium, H1223:1 (image: Finnish Heritage Agency, National Museum of Finland, Collections of History. CC BY 4.0)

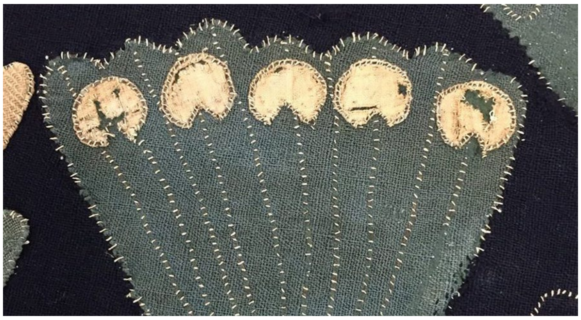

Fig. 4 Detail from the green peacock, with cotton eyes in the train feathers of the peacock's tail (samples 13) (image: Mervi Pasanen)

Hailuoto

Hailuoto is a small island close to the city Oulu. Its wooden church was built in the early seventeenth century and burnt down in 1968. The excavation done by the University of Oulu took place in the mid-1980s. Six textile samples dated between 1620 and 1756 were studied, and two of them included cotton (Suomela & Lipkin, submitted). The first sample (KM86088:255) was a piece of rag paper wrapped around a coin (öre of Queen Christina, 1667). The rag paper was made of bast and cotton fibres. The second sample (KM86088:251) was a fragment of tabby woven cotton cloth attached to a tin button.

White Karelia, nineteenth century

The White Karelian cotton finds from the eighteenth and nineteenth centuries, part of collection SU4522 in the Finno-Ugric Collections of the National museum of Finland, have already been discussed in two papers [48, 49]. Altogether, 40 of the 108 samples were identified as containing cotton. In this article, special attention is given to seven cotton samples studied by Viljanen et al. [48], based on WAXS results only referred to in supplementary material of the earlier study (Table 1; Fig. 5). In the current study, these seven samples were analysed further, noting especially the crystallinity index and comparative orientation parameter analyses based on the WAXS

Table 1 White Karelian samples studied using the WAXS technique [49]

Item number	Object	Sampling place
SU4522:1a	Woman's shirt, *rätsinä*	Hem, vertical
SU4522:1d		Sewing thread
SU4522:82c	Dress, *feresi*	Printed fabric in the lining
SU4522:85a	Dress, *kosto*	Nudge-coloured sewing yarn
SU4522:95b	Towel, *käspaikka*	Embroidery
SU4522:99c	Female under-headgear, *sampsuri*	Interlining

Fig. 5 Cotton textiles from collection SU4522 examined using the WAXS technique (images: Finnish Heritage Agency, National Museum of Finland, Finno-Ugric Collection, CC BY 4.0; except SU4522:95 Jenni. A. Suomela)

technique. Additionally, the samples were examined using optical microscopy methods.

Research methods

Variations of different analytical methods have been used in this study depending on the situation and available apparatus. Reference materials have been systematically examined with optical and scanning electron microscopy as well as with the WAXS technique.

Longitudinal and cross-sectional observation with optical microscopy

All of the samples, the reference material as well as the heritage textiles were studied with optical microscopy. Sample preparation was done under a stereomicroscope Leica S9D. For longitudinal observations, the fibres were mounted, using the permanent mounting media Entellan New™, on glass slides and coated with a cover glass.

For cross-sectional observations, most of the fibres were mounted using a LR White resin and cut with an ultramicrotome Leica ultracut into 2 µm thick slices. The cuttings were placed on a glass slide, observed, and imaged without any mounting or cover glass. A similar type of cross-section method for studying the maturity of cotton fibres has been described by Xu and Huang [50]. The White Karelian samples were sliced using the paper glue method and the Hailuoto samples with the cork sheet method [51].

The heritage textiles as well as the Hailuoto and White Karelian cross-cuttings were studied longitudinally using a transmitted light microscope (TLM), the Leica DM4500P, and imaged with a Leica DFC420 camera. The reference material was studied using a TML Leica DM4 P and the same camera. Occasionally, polarised light was utilised. Cross-sectional observations of the LR White-mounted fibres were conducted with a TLM Leica DM LM and imaged with a Leica MC190 HD camera.

Scanning electron microscope (SEM)

To image the archaeological and modern reference samples, the fibre materials were placed on a piece of double-sided carbon tape that was fixed to an aluminium stub and was coated with a 10 nm thick layer of platinum-palladium (Pt–Pd) to increase the electrical conductivity, create good imaging conditions and avoid any charging, beam damage to or drifting of the sample. The samples were then imaged with a Zeiss Sigma VP scanning electron microscope, using a secondary electron detector and an acceleration voltage of 5 kV. This method was used to examine the reference material and the Masku antependium.

Fourier-transform infrared spectroscopy (FTIR)

FTIR measurements were done with a Nicolet Spectrum 100 instrument by placing 1–2 mm long fibres at the ATR (Attenuated Total Reflectance) crystal. The cotton fibres' FTIR spectrum forms a sharp peak, with points at 1031 and 1057 cm^{-1}, and a wider blunt peak around 3300 cm^{-1} [52], while silk has signature peaks at around 1517, 1625 and 3296 cm^{-1} (see, e.g. [53]). This method was used to verify the fibre material identifications of the Valmarinniemi and Masku antependium textile finds.

Micro-computed tomography (Micro-CT)

With respect to the fabric sample from Valmarinniemi, a slight helical feature and a crescent shape of cotton fibre could be observed in the 3D nanoscale CT images acquired with a high resolution pixel size of 700 nm.

The fibres were segmented and thus could be observed individually, allowing an easy and quick way to observe the fibre properties both in 3D and in the cross sections. The sample, 1.5 × 1.5 mm in size, was imaged in a test tube using a nanoscale x-ray microscope device (SkyScan 2214, Bruker microCT, Kontich, Belgium) and computationally reconstructed into cross-sectional slices with NRecon software (vl.7.5.8., Bruker microCT). (see [54]).

Wide-angle X-ray scattering (WAXS)

Of all plant fibres, cotton fibres are the best source of pristine cellulose, making up around 90% of the fibre's composition [55]. Within the multi-layered cell walls of the fibres, the cellulose molecules have aggregated into partly crystalline microfibrils, which together with the hierarchical cell wall structure explain the mechanical properties of plant fibres. To study the properties of the cellulose microfibrils in the cotton fibres, X-ray diffraction and scattering experiments can be used to determine the crystallinity index (the amount of crystalline cellulose in the entire sample volume) and the average width and orientation of crystalline part of the basic cellulose microfibrils. All these parameters can potentially be used for identification purposes. However, the parameters can be estimated from the WAXS patterns in various ways. While all analytical methods are suitable for revealing differences inside a single sample series, but when the values are compared between different sample series/literature values, the method used must be the same in terms of both the computational and experimental factors.

X-ray scattering methods are non-destructive since they do not require any chemical treatment or sectioning of the samples, and often the measurements can be conducted using only a small amount of the sample material. After the measurements, samples can still be used for other future analyses. As a result, X-ray scattering methods are well suited for studying archaeological samples.

To study the properties of the cellulose microfibrils in different species of cotton, WAXS measurements were conducted at the University of Helsinki's X-ray Laboratory, located in the Physics Department. The WAXS setup has been described in detail by Viljanen et al. [48]: the voltage and the current in the X-ray tube as well as the wavelength ($\lambda = 1.541$ Å) used were the same as in the current study. Lanthanum hexaboride (LaB6) was used to calibrate the scattering angle (2θ) scale and to obtain the instrumental broadening of the setup (0.35°).

The single cotton fibres were aligned and collected into bundles. The samples were measured for 60 min to ensure good statistical data.

Data analysis: Crystallite width

To determine the average width of the cellulose crystallite, a 2D WAXS pattern was integrated using a 60° wide sector at around the 200 (cellulose $I\ \beta$) reflection. The average crystallite width (B) was obtained using the Scherrer equation based on a full width at half maximum (FWHM) peak of 200 peak, which was determined via a curve-fitting method and by taking into account the five main cellulose reflections, each modelled using a Gaussian function: 110, 1–10, 102, 200 and 1–21 peaks within a 2θ range of 13°…28° were considered. The Scherrer equation used was as follows: $\mathrm{B} = 0.9\lambda/(\cos\theta\ (FWHM^2—INST^2)^{½})$, where $FWHM$ was the width of the 200 reflection and $INST$ was the instrumental broadening.

Data analysis: relative crystallinity

For crystallinity analysis, the 2D WAXS pattern was integrated using a 180° wide sector, i.e. half the pattern, including all possible cellulose reflections. The relative crystallinity index was determined using the *amorphous fitting method*, explained in detail by Ahvenainen et al. [56]. The method was similarly used by Viljanen et al. [48]. In the present study, 19 cellulose reflections (the same as given by Viljanen et al. [48]) were selected for modelling the crystalline cellulose component.

Data analysis: crystallite orientation

For the orientation analysis, the cellulose 200 reflection was integrated azimuthally (at fully 180°). The orientation of the crystallites was estimated using three different orientation analysis methods.

For plant samples, the microfibril angle (MFA) refers to the helical angle in which the elementary cellulose microfibrils are wound around the cell axis. There are various analytical methods for estimating the mean MFA value (see [57, 58]). In this study, the mean MFA value was computed following the method described by Sarén et al. [59], where the azimuthal profile of the cellulose 200 reflection is modelled via pairs of Gaussian functions. It is assumed that each pair of Gaussian functions arises from the single cell wall layer (a symmetric pair models the fact that both the front and the rear cell walls contribute to the profile); additionally, one extra Gaussian function at the zero angle, a shape factor, is considered for the contributions from the side walls. In this study, two pairs of Gaussian functions were used to account for the contributions of the front and rear S2 cell wall (with the main contribution being at an azimuth angle of ~ 10°) and the S1 wall (with the secondary contribution being at larger azimuth angles of > 30°), as well as a relatively small Gaussian function at an azimuth angle of 0° (with the shape factor arising from the side walls).

The orientation parameter describing the universal level of anisotropy can be computed using the Herman's orientational parameter (OP) [60]. In this study, the contribution of the fully isotropic background was neglected, i.e. set at a background of zero, similarly as was done by Viljanen et al. [48] and also, e.g. Shenouda [61]. Especially for cotton, prior studies have determined an azimuth angle corresponding to 50% of the maximum intensity value of the cellulose 200 peak (see [9, 61]).

Results from the reference fibres

We studied the reference fibres using TLM, in a longitudinal and cross-sectional direction, as well as with an SEM and WAXS. We did not include the FTIR measurements due to inaccuracies in the device being used.

Optical microscopy

With respect to the longitudinal observations, we were able to detect differences between the species (Fig. 6). The *G. arboreum* sample had not flattened at all and contained very few convolutes (Fig. 6a). The surface was covered with similar-looking cross markings typical of bast fibres [27]. The *G. barbadense* sample had frequent convolutions, even though some fibres lacked a twisting pattern (Fig. 6b). The fibres were clearly flattened and thicker at the edges. The *G. herbaceum* sample did not contain any clear identical features (Fig. 6c). It had twists but also plenty of fibres without convolutions. The fibres in the sample were quite inconsistent. Some of the fibres were flattened, some not. Also, cross markings occurred on some. The *G. hirsutum* sample was clearly flat and ribbon-like (Fig. 6d). It had convolutions, but less so than the *G. barbadense* sample.

The *G. barbadense* 2008–0392 sample was similarly tightly twisted as the above-mentioned *G. barbadense* sample (Fig. 6g). Though again, some of the fibres did not have convolutions at all. Some squeezing did occur, which is more typical of bast fibres, especially nettle [62]. The *G. barbadense* 1998–0267 and *G. herbaceum* 1998–0268 samples (that had dubious mingled background) displayed mixed characteristics and some similarities (Fig. 6e and f).

We used cross-sections of the samples to measure the width of the fibre. Measurements were taken, using the Leica software analysing tool, from the thickest part of the fibre. If it had a C-shape, which is typical of cotton, then the measurement was taken from the transverse of the thickest point to the C-shape opening. With crescent-shaped cotton fibres, it is best to measure the width of the fibre from the cross-cut section. This makes it possible to standardise the measurement points equally. The results of mean and median values can be found in Table 2.

Table 2 Average and median widths of fibres in micrometres

Sample	Number of measurements	Mean µm	Median µm
G. arboreum	n = 32	23.82	22.44
G. barbadense	n = 29	18.81	18.80
G. herbaceum	n = 39	18.95	18.70
G. hirsutum	n = 40	18.85	18.90
G. barbadense 1998–0267	n = 14	24.17	22.43
G. herbaceum 1998–0268	n = 32	25.35	26.03
G. barbadense 2008–0392	n = 40	19.54	19.64

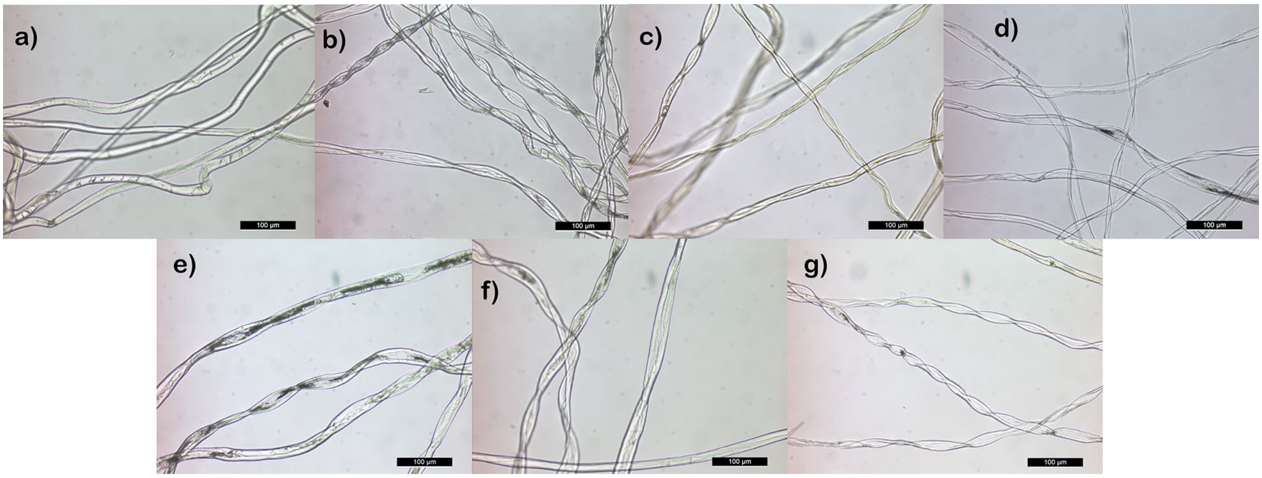

Fig. 6 Longitudinal characteristics when using TLM: **a** *G. arboreum*, **b** *G. barbadense*, **c** *G. herbaceum*, **d** *G. hirsutum*, **e** G. *barbadense* 1998–0267, **f** *G. herbaceum* 1998–0268 and **g** *G. barbadense* 2008–0392 samples (images: Jenni A. Suomela)

According to the measurements, the *G. arboreum* sample as well as the *G. barbadense* 1998–0267 and *G. herbaceum* 1998–0268 samples (which had intermingled background) had the largest diameters. All the other samples had fairly similar values of around 19 µm.

When observing the cross-sections to determine the level of maturity of the samples, *G. arboreum* (Fig. 7a) and *G. herbaceum* 1998–0268 (Fig. 7f) contained the most of fibres in a mature or overmatured state. The *G. barbadense* sample (Fig. 7b) had mostly mature fibres, while the *G. herbaceum* (Fig. 7c) and *G. barbadense* 1998–0267 (Fig. 7e) and 2008–0392 samples (Fig. 7g) had both immature and mature fibres. Cross-sections of the *G. hirsutum* sample (Fig. 7d) are interpreted to be mostly immature fibres.

SEM results

The differences in fibre characteristics were not as distinct using TLM (Fig. 8). All of the fibres had convolutes to varying degrees. The waxes and fats were clearly visible on the surface of all the cotton samples as amorphous wavy patterns (Fig. 8h; see [55]). The *G. barbadense* 1998–0267 sample clearly showed the cross-markings in a look-a-like structure on the fibre surface (Fig. 8e).

WAXS results

We used WAXS to determine the structural parameters (crystallite widths, crystallinities and orientation factors) of seven reference cotton fibre samples. The results are summarised in Table 3.

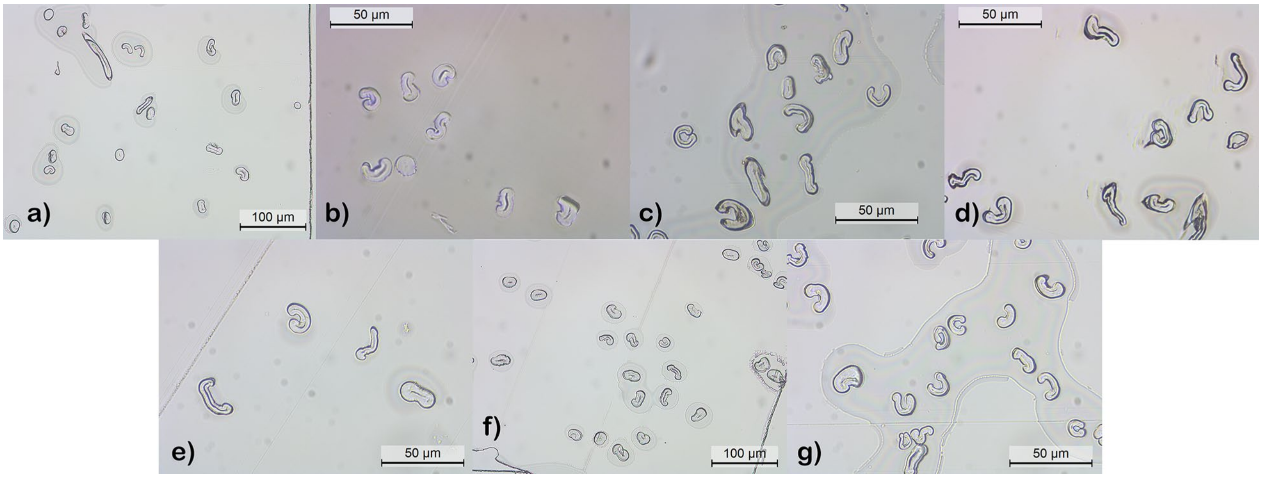

Fig. 7 Cross-sectional characteristics: **a** *G. arboreum*, **b** *G. herbaceum*, **c** *G. hirsutum*, **d** *G. herbaceum*, **e** *G. barbadense* 1998–0267, **f** *G. herbaceum* 1998–0268 and **g** *G. barbadense* 2008–0392 samples (images: Jenni A. Suomela)

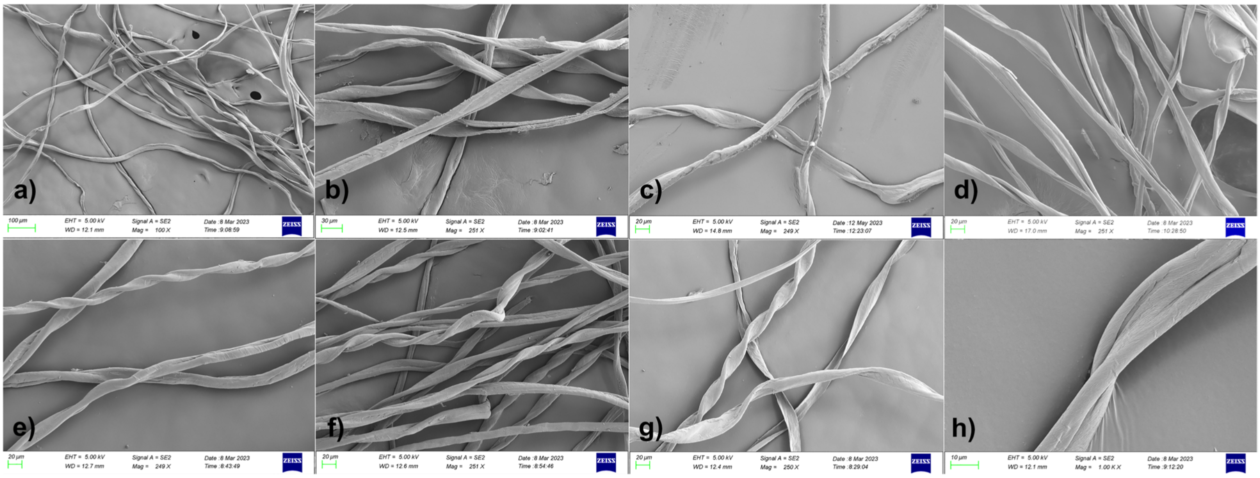

Fig. 8 SEM images of the cotton fibres: **a** *G. arboreum*, **b** *G. herbaceum*, **c** *G. hirsutum*, **d** *G. herbaceum*, **e** *G. barbadense* 1998–0267, **f** *G. herbaceum* 1998–0268, **g** *G. barbadense* 2008–0392 and **h** close-up of *G. arboreum* samples (images: Krista Wright)

Table 3 Structural parameters determined using WAXS: cellulose crystallite widths, crystallinity indexes (CR), Herman's orientation parameter (OP), mean microfibril angles (mMFA) and the azimuth angle values for the 50% I_{max}

sample	Width (nm) ±0.2 nm	CR ± 0.03	OP [±0.07]	mMFA (°) [±2°]	50% I_{max} (°) [±2°]
White Karelian[1] [48]					
1a	7.5[1]	0.50	0.49[1]	24	33
1d	7.8[1]	0.45	0.43[1]	27	40
82c (blue)	7.5[1]	0.44	0.30[1]	28	37
82c (orange)	6.8[1]	0.50	0.47[1]	24	32
85a	6.6[1]	0.48	0.54[1]	22	31
95b	6.4[1]	0.47	0.54[1]	22	32
99c	6.9[1]	0.45	0.48[1]	24	35
Reference fibres					
G. arboreum	6.0	0.54	0.56	20	27
G. barbadense	6.6	0.57	0.53	24	35
G. herbaceum	5.9	0.53	0.43	24	41
G. hirsutum	6.3	0.54	0.57	24	32
G. barbadense 1998–0267	5.9	0.57	0.44	27	38
G. barbadense 2008–0392	6.3	0.54	0.57	23	32
G. herbaceum 1998–0268	5.8	0.54	0.49	24	34

Crystallinity index and crystallite width results

The crystallinity values for the reference cotton fibres were between 53 and 57%. Considering the margin of error for the values (3%), the crystallinities of the various native cotton fibres being studied were the same. Shenouda et al. [61] determined crystallinities for native and treated Egyptian cotton fibres using the Ruland method, which is comparable to our amorphous fitting method. For native cotton fibres, they obtained values ranging between 56 and 65% [61], which are quite close to our crystallinity values.

Orientation of cellulose crystalllites

We estimated the orientation of the cellulose crystallites quantitatively using three different parameters: the Herman's orientation factor, the azimuth angle at 50% maximum intensity and the mean MFA. The second approach was chosen purely for comparison purposes, because prior studies of cotton fibres have typically tabulated this value. The third approach was chosen as a way of comparing our results with more recent analyses of the MFAs of plant fibres. We detected no clear differences between the orientational order of the reference cotton fibres, as shown in Fig. 9.

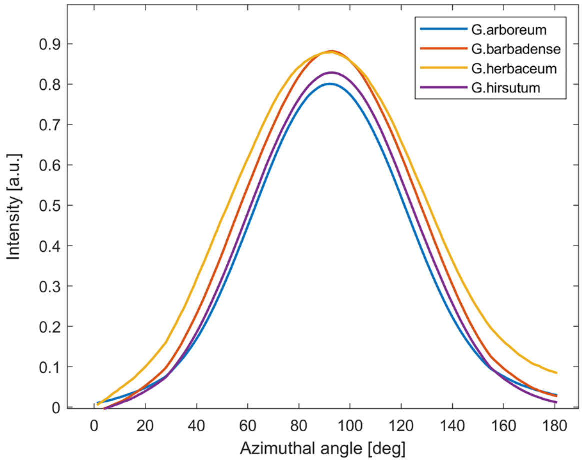

Fig. 9 The azimuthal intensity of the 200 cellulose reflection for four native cotton samples (*G. arboreum, G. barbadense, G. herbaceum* and *G. hirsutum*); data for the *G. arboreum* sample corresponded with the highest orientational order (i.e. the smallest mean MFA and the highest OP value), but the differences between the samples are close to the experimental accuracy of the results

The typical cross-sectional shapes of the cotton fibres are relatively flat (e.g. compared to the fully circular or square cell shapes of many wood cells). If the cotton fibres are compressed together so that the long axis of their cross sections are parallel with each other, the contribution of the side cell walls to the azimuthal profile of the cellulose reflection will presumably be small compared to the contribution of the front and rear walls. Thus, the relative height of the Gaussian function at the zero azimuth angle will presumably be small in the MFA analysis used in this study. In this case, the mean MFA values obtained using Gaussian fitting would be comparable to the orientation of the crystallite angle when using DeLuca and Orr's [63] method, as determined by, e.g. Cao et al. [9] and Shenouda et al. [61].

It must be noted that in addition to the cell shape, any orientation parameter determined by X-ray scattering contains contributions from many other experimental factors as well. These factors, which affect the azimuthal profile of cellulose reflection 200, include the number and width of the different cell wall layers, each of which makes its own contribution to the distribution of the orientation factors, the spiral angle, which can vary between individual fibres, and most importantly, the macroscopic alignment of the fibres and fibre bundles relative to the X-ray beam. Thus, any comparison of macroscopically different cotton fibre bundles (and/or historical fibres) is difficult without the possibility of studying a single fibre isolated from the rest of the sample. However, to study single fibre samples, a synchrotron X-ray source is needed to achieve enough intensity, and the synchrotron measurements need the approval of the beamtime application, making access to the synchrotron methods extremely limited. A recent example of an orientation study of a single nettle, sisal and cotton fibre using a synchrotron source, including an important discussion of the different factors affecting the MFA value, was conducted by Richely et al. [64].

Heritage material results

This section concerns unpublished analysis results from recent Finnish cotton finds that are not dealt with in the original publications, except for Valmarinniemi, for which all the results are previously unpublished.

Valmarinniemi

Microscopy analysis revealed that Valmarinniemi sample KM39304:1544 is made of cotton. The sample was covered with microbial residue (Fig. 10a, b), and due to its exceptionally early dating, we wanted to be sure of its identification and so conducted an additional analysis. Cross-sectional observations confirmed the identification, with the fibres clearly exhibiting the C-shape characteristic of cotton (Fig. 10c). FTIR provided additional confirmation when we compared the sample to a

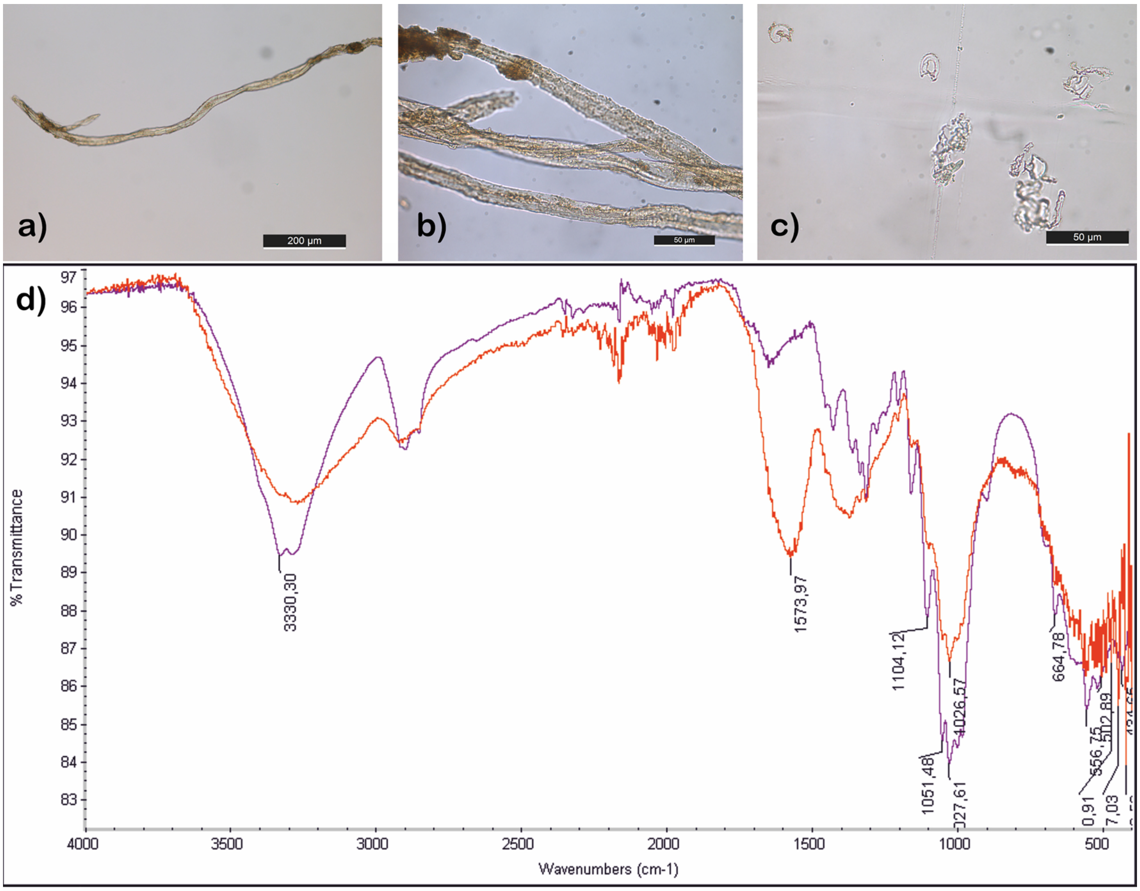

Fig. 10 Valmarinniemi sample: **a**, **b** longitudinal image, **c** cross-sectional image and **d** FTIR spectrum of Valmarinniemi cotton sample (images **a**, **b** and **c** by Jenni A. Suomela; image d by Krista Wright)

modern-day cotton shopping bag. Figure 10d shows that the peeks have similar wavelengths, which excludes the possibility of silk fibres. The differences in peeks can be explained by degradation processes in the actual samples or by dying or finishing treatments used with reference samples. We received additional confirmation for the cotton sample by using nanoscale CT imaging. A slight helical feature and a crescent shape of cotton fibre could be observed when using 3D, high-resolution images with a pixel size of 700 nm [54].

Masku antependium textile

Optical microscopy results showed that, most likely, the samples obtained from both the black (samples 12a and 12b) and green (samples 13a and 13b) peacock were from the same fibre source. The fibres had small convolutions, many cross-markings and included relatively many dead fibres (Fig. 11a; [7]). The dead fibres appear as flat and transparent with no birefringent properties (Fig. 11b and d).

FTIR was used with these samples to confirm the identification (Fig. 11e). This method proved useful since in some cases the historical cotton and un-degummed silk fibres can have quite identical morphological features when observed microscopically [19].

Hailuoto

Hailuoto cotton samples have been thoroughly evaluated in a study by Suomela and Lipkin [65]. The fibres found in the rag paper (KM86088:255) were in poor condition, probably due to the paper manufacturing process. The other cotton sample, which had been preserved in connection with a tin button (KM86088:251), was in much better condition. The fibres had few convolutions and, based on the low-quality cross-cutting image, they were in a mature state (Fig. 12).

White Karelia

White Karelian cotton samples were studied only in a longitudinal direction with TLM, because in the original study the emphasis was on material identification only, thus making it unnecessary to conduct a cross-sectioning analysis.

Based on their morphological characteristics, all six cotton samples seem fairly similar—very few convolutions, flattened fibres, but swollen from the sides (Fig. 13). Sample SU4522:99c, taken from the *sampsuri*, under the headgear, was in poor shape with lots of additional residue on the surface (Fig. 13f). When comparing the visual properties with the reference samples, we found that it most closely resembles *G. herbaceum* (Fig. 6c). In Fig. 14, some of the White Karelian samples and *G. herbaceum* are compared based on their azimuthal WAXS intensity, revealing that the orientational profiles of especially samples 85a, 95b and 99c corresponded quite well with the orientational profile of *G. herbaceum*.

The nano-structural properties of the samples, as determined by WAXS, describe the crystallinity index of the fibres and the size and orientation of the elementary cellulose crystallites. The results are summarised in Table 3. The crystallite widths and Herman's orientation parameters for the White Karelian fibres are from a study by Viljanen et al. [48]. Regarding the crystallinity values of any cellulose samples, it must be noted that they reflect effects from multiple different factors, especially when considering the historical or treated samples: these competing factors include, e.g. hydrolysis due to exposure to acidic compounds and/or sunlight, which might have degraded the amorphous components (leading to a higher crystallinity index), and/or degradation of the crystalline cellulose chains (leading to a lower crystallinity index).

Analysis revealed slight nano-structural differences between the White Karelian cotton samples: the crystallinity values of the samples were 44–50%, the mean MFA values were between 22° and 28°, and the Herman's orientation parameters were between 0.30 and 0.54. Thus, we observed the largest difference with sample 82c, which had the lowest OP value, while the crystallinity index was also lowest for this sample. The low levels detected for both the orientation parameters and crystallinity might be connected; sample 82c (or the exact area of it sampled using WAXS) could have been slightly more deteriorated than the other White Karelian cotton fibres included in the study.

Based on the OP values, the mean MFA values and the angles of 50% I_{max}, the White Karelian cotton fibres had a quite similar orientational degree compared to the reference cotton fibres evaluated for this study (Fig. 14).

Cao et al. [9] determined the width of the cellulose crystallite to be larger in their archaeological cotton sample (6.0 nm based on the 200 reflection) compared to the modern reference cotton sample (around 5 nm). We observed a similar kind of trend between our reference cotton fibre samples and the White Karelian cotton samples. In our previous research as well, we observed the same effect with bast fibre samples (nettle, flax, hemp): the crystallite sizes were clearly larger in all the historical samples compared to the corresponding modern reference fibre samples [48].

Discussion

In the pre-Columbian era, before any trans-Atlantic trading activity, it is safe to assume that Northern European cotton dating to before the sixteenth century are from either the *G. arboreum* or *G. herbaceum* species.

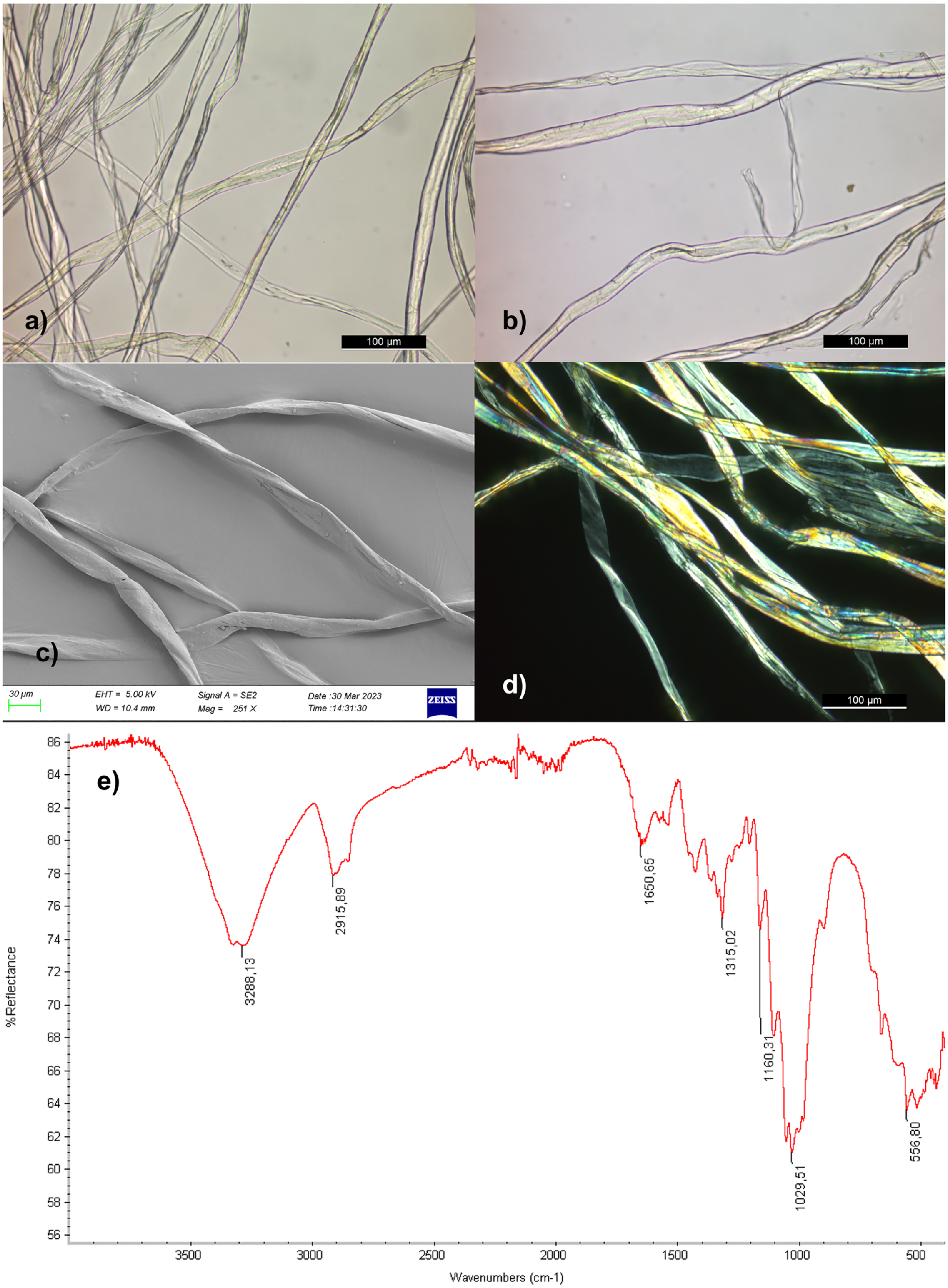

Fig. 11 Cotton samples 12 and 13 from the Masku antependium textile: **a** TLM image of sample 12a, **b** TLM image of sample 13b, **c** SEM image of sample 12, **d** POL image of sample 13b and e FTIR spectrum of sample 12 (images **a**, **b** and **d** by Jenni A. Suomela; images **c** and **e** by Krista Wright)

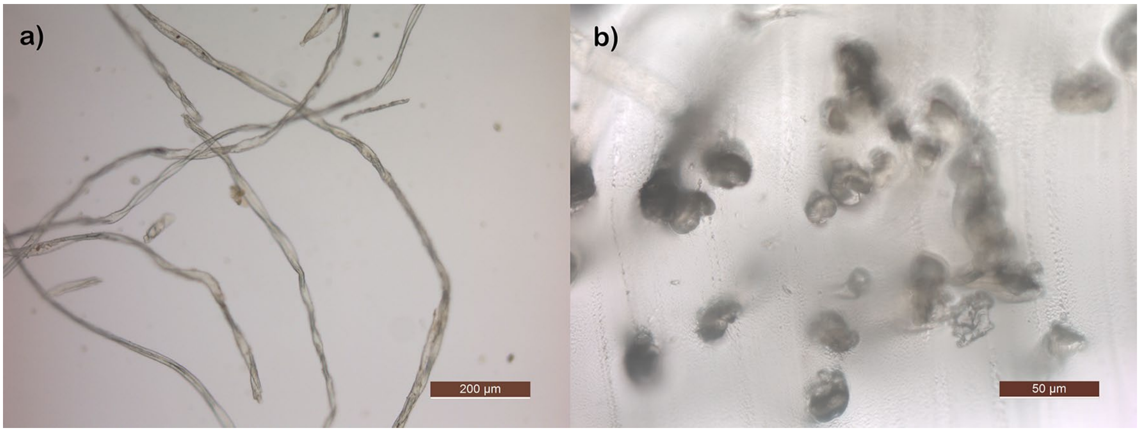

Fig. 12 Hailuoto KM86088:251 images with TLM: **a** longitudinal and **b** cross-section images (images: Jenni A. Suomela)

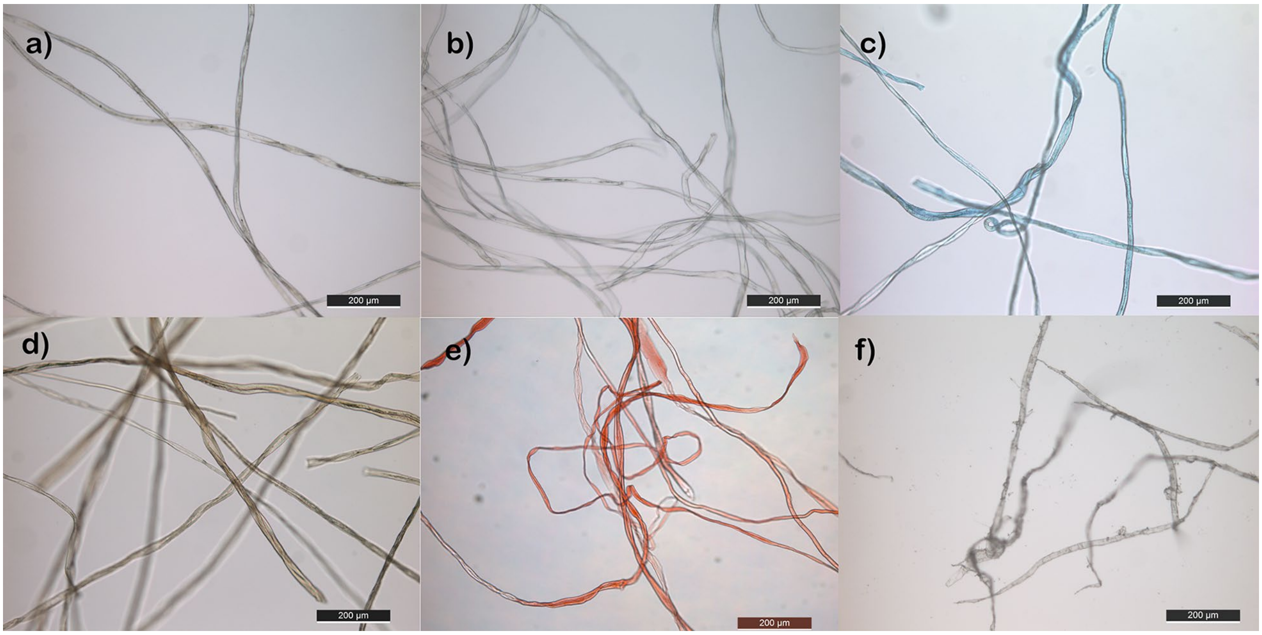

Fig. 13 Longitudinal TLM images of White Karelian samples: **a** SU4522:1a, **b** SU4522:1d, **c** SU4522:82c, **d** SU4522:85a, **e** SU4522:95 and **f** SU4522:99c (images: Jenni A. Suomela)

The global prevalence of *G. hirsutum* only dates to the twentieth century [36], so the *G. arboreum* and *G. herbaceum* species are the safest bets for the heritage materials studied in this paper, meaning the timespan is from the turn of the fourteenth century to the late nineteenth century. Based on Beckert's [5] description of mediaeval cotton markets in Europe, *G. herbaceum* is the most probable candidate.

Viot [8] has asserted that only the DNA analysis of seeds done by Palmer et al. [43] and the analysis of fibre parameters by Cao et al. [9] are the only reliable studies to date on a species level. This assertion inspired us to experiment further, to determine if we could detect any differences at the species level when using microscopy and various diffraction methods.

Palmer et al. [43] has shown how significantly the genomic composition differs in modern and archaeological *G. herbaceum* species. According to Stephens [66], modern versions of different species do not represent the characteristics of archaeological fibres, and thus, the comparison with modern reference fibres is useless. Most likely this genomic change has also influenced the parameters of the fibres.

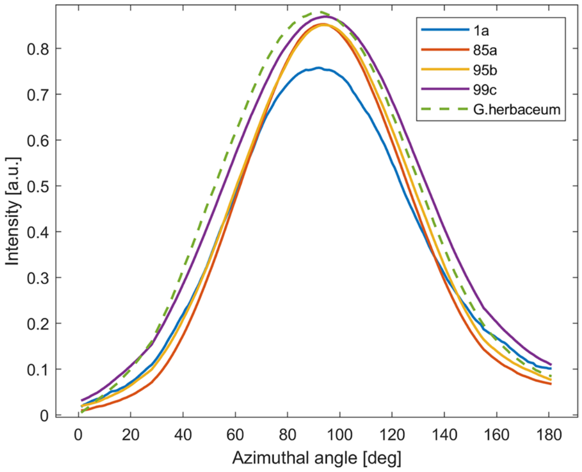

Fig. 14 The azimuthal intensity of the 200 cellulose reflection for the four White Karelian cotton samples compared to that of the *G. herbaceum*

Evaluation of the DNA analysis is beyond the scope of the study, but some of the fibre characterisation methods used by Cao et al. [9] require more discussion. Measurements taken of common textile fibre parameters, such as their elongation, fibre length or fineness [9], are inapplicable in the case of heritage textiles due to the small number and sample sizes characteristic of Finnish finds. Besides, promoting the smallest possible sampling and non-invasive research methods is in our interest. The effort to take samples in either a non-destructive or least-destructive manner does not allow for an analysis of fibre length, elongation or strength measurements in heritage textiles—the sample size is too small. Additionally, calculating the number of convolutions per centimetre seems irrelevant due to the huge variation within samples, as is clearly visible in the TLM and SEM images (Figs. 6 and 8).

To provide more reliable results concerning the different reference cotton species, the research material should have been much larger, with several representatives from each species. In cultivated species, fibres can be found around the seed in two layers—convoluted lint fibres and fuzz fibres more tightly attached to the seed, which lack any convolutes [66]. Since our reference material consists of seeds that had only some fibres left on them, it must be noted that some of the material could be just fuzz fibres.

However, this study shows the difficulties in applying already studied fibre properties to our material. The fibre maturity and number of convolutions are not in line with the reference literature. Our *G. arboreum* sample, which, based on an evaluation of its ripeness, was mature/overmature and had barely any convolutions at all, while both *G. barbadense* samples clearly had more convolutions than the other species. Based on the cross-sections, *G. barbadense* 2008–0392 was immature/mature and *G. barbadense* was mature. The samples collected from the Botanical Gardens were from plants that have been grown for exhibitional purposes and based on visual evaluations; the cotton balls had been there already for some time, which does not support the finding that the *G. barbadense* 1998–0267 sample contains immature fibres in the cross-section (Fig. 7e).

Clear differences in the reference material could be detected when using optical microscopy, but it is possible that the reasons might have to do with the maturity state of the fibres. If this maturity state is set aside for a moment, and only the visual properties of the fibres are observed, while bearing in mind the historical factors supporting *G. arboreum* and *G. herbaceum* as choices, our results do make sense. All the heritage samples have few convolutions and are fairly swollen, whereas all the *G. barbadense* samples clearly have many more convolutions and the *G. hirsutum* sample was flat and ribbon-like—all features associated with modern cotton fibres in the existing literature.

Most of all, our main contribution is that the study highlights the varying visual properties of the heritage samples. Often it is difficult to make an identification if only longitudinal characteristics are observed. The cross-markings and squeezes typical of bast fibres and the appearance of two round fibres attached to each other, which is typical of un-degummed silk, hinder the interpretation process. Neither cross-sections alone should be used as an identification method. Nettle and mature cotton have similar cross-sectional properties. Overmature cotton fibre with small lumen and round shape, can easily be confused with flax.

Then again, it is possible to observe convolutions in cotton fibres already with a stereo microscope. In suitable cases, this makes the identification process easy. FTIR is a suitable and easy analytical tool for revealing the differences between silk and cotton. PLM and a lack of dislocations make it easier to separate cotton fibres from bast fibres. WAXS makes it possible to identify cotton, but not at the species level, and micro-CT allows for fibre visualisations both in 3D format and in cross-sections.

Conclusions

In this article, we have reviewed the historical background of cotton fibre around the world as well as fibre characteristics and reflected on such a historical trajectory in relation to recent Finnish cotton finds. Our attempt at identifying cotton fibres at the species level provided hints for understanding why the heritage samples differ from modern ones in their characteristics. For the first time, an attempt was made to compare all four cultured species based on their visual and nano-structural properties. However, not all the questions found answers and further research is required with more extensive comparative material.

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Author contributions
JAS has conducted and written the literary review in the introduction, optical microscopy analysis and interpretation, interpreted FTIR and SEM material and wrote the discussion and conclusion sections. MV and KS are responsible for all the WAXS measurements, the analysis and the text. KW has done SEM imaging and FTIR measurements. SL has contributed the micro-CT sections.

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Availability of data and materials
The datasets used and analysed for the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests
The authors declare that they have no competing interests.

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