Fibres in Heritage Objects

Identification and Characterisation by Imaging Techniques

Hana Lukesova

Thesis for the degree of Philosophiae Doctor (PhD)
University of Bergen, Norway
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Abstract

The work presented in this thesis focuses on the identification and characterisation of plant fibres from cultural heritage objects. The main emphasis is on method development (Archaeometry) in the field of optical microscopy. This has been done in three ways: **i)** Investigating the validity of established plant fibre identification techniques applied to historical and archaeological samples; **ii)** The development of an identification method for a hitherto little-regarded textile plant fibre species and **iii)** Application of the identification methods on cultural heritage objects.

The thesis consists of five articles that are divided into the three categories listed above. **The first category** covers the overall methodology of how to adapt methods, that were developed for investigation on modern fibres, on archaeological materials. This is discussed in one separate article concerning questions regarding sampling, correct performing of tests as well as result evaluation of degraded sample materials that are in many ways different from the modern ones. The second article focuses on two features that have been used for the identification of fibres: the cross-section shape and the lumen shape. The application of these two features, in the investigation of cultural heritage materials, was re-evaluated. It was concluded that they cannot be used on their own as distinguishing features for plant fibres.

The material resources of ancient societies differ from the modern ones. Not only the species used for commercial fibres in modern times were used for textile production in past. The identification diagrams, derived mainly from industry and forensic science, are depending on relevant species. If species that were used in past are not included, the diagrams cannot be correct. The research area for future studies is therefore huge. **The second category** aims to diminish this discrepancy and focus on the development of an identification method for the (in a textile context) little-regarded species hops *Humulus lupulus*. This work is presented in a method article, where a new identification diagram, including hops, can be found. According to various written sources, hop fibres were used for textiles in Scandinavia. This was confirmed in an experimental study which is a part of **the third category**, concerning the application

of identification methods on cultural heritage objects. Here, in one article, the recently developed identification diagram for plant fibres, which includes the hops species was applied on historical textile samples, with results confirming that hops were used for textiles in past. The second article was about the modified Herzog test applied on degraded Viking Age and Merovingian Period objects from the Late Iron Age Collection of the University Museum of Bergen. The results showed that flax (*Linum usitatissimum*) was used for undergarments as well as small textile accessories at this time in western Norway.

List of Publications and Presentations

This thesis is based on 5 published, peer-reviewed articles listed below in chronological order. All articles, except number 5 are listed in the Web of Science and published in journals included in the Norwegian List of "Point-giving journals". The Norwegian list operates with level 1 and 2 journals. Level 2 journals are ranked as having higher scientific value. Articles numbers 1 and 3 are published in a level 2 journal, articles numbers 2 and 4 are published in level 1 journals. Article 5 is published in the proceedings for a highly respected conference in the textile archaeology community. The thesis defender is the first and corresponding author of articles No. 1, 3, 4 and 5. She is the last and corresponding author of article No. 2.

Published articles:

(1) **Lukesova, H.**& Holst, B. (2021): "Is Cross-Section Shape a Distinct Feature in Plant Fibre Identification?" Archaeometry. https://doi.org/10.1111/arcm.12604

(2) Skoglund, G., Holst, B., & **Lukesova, H.** (2020). "First experimental evidence of hop fibres in historical textiles." Archaeological and Anthropological Sciences, 12(9). https://doi.org/10.1007/s12520-020-01171-6

(3) **Lukesova, H.**, Andersen, H. L., Kolínová, M., & Holst, B. (2019). "Is It Hop? Identifying Hop Fibres in a European Historical Context." Archaeometry, 61(2), 494–505. https://doi.org/10.1111/arcm.12437

(4) **Lukesova, H.**, Palau, A.S., Holst B. (2017) "Identifying Plant Fibre Textiles from Norwegian Merovingian Period and Viking Age Graves: The Late Iron Age Collection of the University Museum of Bergen." Journal of Archaeological Science: Reports 13, 281-285.* https://doi.org/10.1016/j.jasrep.2017.03.051

(5) **Lukesova, H.** (2017): "Application of Herzog test on Archaeological Plant Fibre Textiles. Possibilities and limits of polarized light microscopy", In Bravermanová, M. – Březinová, H. – Malcolm-Davies, J. (Eds.) Archaeological Textiles – Links Between Past and Present. NESAT XIII. Liberec – Praha

* This article was highlighted in Nature Materials: Ball, P (2017): Seeking comfort in the Iron Age. *Nature Materials* 16, 789. https://doi.org/10.1038/nmat4950

Conference presentations:

(1) **Lukesova, H.**, Characterisation of hop fibres by optical microscopy. Conference: Fibres in Early Textiles from Prehistory to 1600 AD; June 2019; The University of Glasgow, Glasgow, GB. Oral presentation

(2) **Lukesova, H.**; Holst, B., Transmission electron microscopy - The nanoscale technology for tomorrow's archaeology? The use of TEM in Archaeology and its application on mineralized material. Conference: Westward bound, The University of Bergen, Bergen, 2018. Poster presentation

(3) **Lukesova, H.**, Application of Herzog Test on Archaeological Plant Fibre Textiles. Possibilities and limits of polarized light microscopy. Conference: The North European Symposium for Archaeological Textiles, Liberec/CZ, 2017. Oral presentation

Invited presentations:

(1) **Lukesova, H.**, Identification of historical textiles, Seminar at the Department of Archaeology, Conservation and History, The University of Oslo, 2019. Oral presentation and workshop on learning fibre identification techniques to an audience of PhD and master students

(2) **Lukesova, H.**, Using polarized light microscopy to identify plant fibres applied on textile artefacts from New Zealand, Guest Colleague presentation at the Swedish National Heritage Board, Visby, 2018. Oral presentation followed by an experimental workshop with Swedish cultural heritage professionals.

(3) **Lukesova, H.**, Laser scanning confocal microscopy and microtomography used for visualisation of plant material, Seminar at the Museum of Archaeology organized by the research group BEAM, Stavanger, 2017. Oral presentation

Broad Audience Presentations:

(1) **Lukesova, H.**, Seeking comfort in the Viking Age – Identification of Archaeological Plant Fibre Textiles, Popular Scientific Contribution, Seminar Series: Kunskapseplet, The University of Bergen, 2019. Oral presentation

Contents

Nomenclature

Abbreviations

aDNA ancient deoxyribonucleic acid

ATR-FTIR Fourier transform infrared spectroscopy in attenuated total reflectance mode

DNA deoxyribonucleic acid

FTIR Fourier transform infrared spectroscopy

ICOM International Council of Museums

ID identification

IR infrared light (electromagnetic radiation 0,7 – 1000 μm)

mtDNA mitochondrial deoxyribonucleic acid

PLM polarized light microscopy

SEM scanning electron microscopy

SEM-BE scanning electron microscopy with backscattered electrons detector

SEM-EDX scanning electron microscopy with energy dispersive X-ray detector

SEM-SE scanning electron microscopy with secondary electrons detector

TEM transmission electron microscopy

TLM transmitted light microscopy

UV ultraviolet light (the electromagnetic radiation 10 – 400 nm)

VIS visible light spectrum (the electromagnetic radiation 400 – 700 nm)

XRF X-ray fluorescence spectroscopy

μXRD X-ray micro-beam diffraction

Symbols

n_D refractive index

Δn difference of refractive indices

NaOH sodium hydroxide

I Overview and Summary

1. Introduction

1.1 Thesis structure

This thesis is divided into three parts. Part I presents an Overview and Summary in four chapters: 1. Introduction, 2. Plant Fibre Identification Methods, 3. New fibre identifications on cultural heritage objects and 4. Conclusion. Part II contains all articles that have been published during this thesis work. Part III contains appendices.

1.2 Motivation and background

Textiles have been enormously important for society throughout history. In many areas of the world, they are as critical for survival as food and water and they have always played an important role in the demonstration of gender, age, social-, political- and economic status as well as occupation, religion, and ethnicity [1, 2]. It has been suggested that textile crafts date back earlier than metallurgy and even pottery [3]. A very recent find of a Neanderthal tree bast string from Abri du Maras in France dated back to about 50 000 years ago suggests, that the beginnings of textile crafts are even much earlier than hitherto believed [4].

The importance of textiles is highlighted also by the fact that one of the most important events in modern history: the industrial revolution, was driven by the textile industry through innovations of mechanical spinning- and weaving machines [5]. It is interesting to reflect that a necessary prerequisite was the introduction of splicing, which describes a group of several techniques enabling the production of an infinite thread [6]. It is difficult to estimate the time in history when splicing was introduced. According to Gleba and Harris [6], a thread attached to a comb from Wadi Murabba'at dated in the 9th millennium BC [7, p.199] is one of the earliest confirmed examples of this technique.

The first major revolution in the human way of living: the transition from a hunting and gathering to an agricultural society [8, 9], was naturally not driven by textile

production alone, but the transition from the use of wild natural resources such as tree bast, nettle and fur to agricultural products such as flax, hemp and wool had a great impact on ancient societies [10-13].

Preserved textile objects constitute a rich source for archaeological, historical, and cultural heritage research. The areas of use can be split into three main categories: i) clothing (i.e. garments, headcover, shoes, accessories), ii) furnishing and art (i.e. upholstery, curtains, bedding, carpets, tapestries, wall hangings, textile wallpaper, canvas for paintings) and iii) functional textiles (i.e., sails, ropes, fishing nets, various packing). The importance of textiles for marine transport in form of sails and ropes is a largely unexplored research field, which deserves more attention as highlighted [14].

Recycled textile is another important area. Textiles were reused in many ways for example impregnated with tar as waterproof caulking in ships, and as a "raw material" for paper making until the 19th century, when an increased demand, which required a shift in material use, led to the application of pulpwood for paper production. The earliest preserved paper fragment known so far stems from the beginning of the Western Han Dynasty from the 2nd century BC [15, p.70]. Fibres of paper mulberry (Kozo) were used together with milled hemp rags for papermaking in China [16].

Information about what kind of materials have been used to produce different textile objects is very important because it provides knowledge about the infrastructure and resource management in the societies where the objects were made and used. Agnes Geijer was one of the first textile historians who pointed out the importance of distinguishing between species [17]. Despite importance, the investigation of textile materials has received little attention compared to metals, ceramics, lithics and glass, which have been the dominant topics of what may be phrased as "historical material investigations" up till now. In fact, the topic of textiles has frequently been ignored as is the case in the very recent book on Archaeological Science [18]. In this otherwise

excellent book, all the previously mentioned materials have their own chapters, only textiles, as a material group, are missing.

Modern times have seen the introduction of a large variety of semi-synthetic and synthetic fibres for textile production (viscose, nylon, polyester, polyamide, etc.), however, up to the 1880s, only natural fibres were available [19]. These are either of cellulose origin (i.e. flax and cotton), protein - (i.e. wool and silk) or even of inorganic origins such as metal threads or textiles made of minerals containing asbestos fibres [1, p.8, 20, p.3-11]. An overview of some selected fibres and fibrous materials used for textiles and cultural heritage objects in past can be found in Appendix A. While animal and plant fibres (not to mention metal and mineral fibres) are relatively easy to distinguish between each other, it can be very difficult to distinguish between different types of plant fibres and the identification is often done based on insufficient, sometimes even incorrect examinations [21, 22]. Up till now this lack of available identification methods has been a limitation for research. The work presented in this thesis addresses this challenge as specified in the thesis objectives presented below.

1.3 Thesis Objectives

This thesis has three related objectives:

i) An investigation of the validity of established plant fibre identification techniques applied to historical/archaeological samples. This is investigated in two ways: The specific behaviour of degraded material (article 5) and the use of cross-section shape as a distinguishing feature (article 1).

ii) The development of an identification method for a hitherto little-regarded textile plant fibre: hops, *Humulus lupulus* (article 3)

iii) Demonstration of the practical use of plant fibre identification methods through the application of identification methods, including the new

method for hops, on selected historical and archaeological textiles (article 2 and 4).

It is important to emphasize that, in line with the thesis defendant's training as a conservator, this thesis is rooted in the natural sciences (the main supervisor is a physicist, the co-supervisor is a botanist) and the overarching aim is restricted to Archaeometry: The development of correct and/or new methods for plant fibre identification and the application of these methods to archaeological and historical textile objects thus providing methods and information that can be used in the future by textile archaeologists and historians to draw fact-based conclusions.

This thesis contributes to the necessary task of reducing the gap between a humanistic approach to (textile) archaeology and cultural heritage studies and the application of tools from the natural sciences [23, p.124-165, 24].

2. Plant Fibre Identification Methods

2.1 Morphology of Natural Plant Fibres and Hairs

Plant fibres extracted from different species have been used for textile production since prehistory [3, 13, p.577]. Fibres may come from different parts of a plant. In this chapter, the morphological features of fibres will be discussed.

In the context of textile terminology, the term "fibre" has a much wider use than in botany. In textile publications, different types of cells or bigger structures are often applied to the term "fibre", for example, a fibre bundle with associated tissue [25, p.60]. Furthermore, what in botany is known as seed/fruit hairs (i.e. cotton and kapok) is referred to as fibres by people working with textiles. In this thesis, the word "fibre" will be used in its wider sense with exception of this chapter where the difference between the different types of "fibres" will be explained from the botanical point of view. It should also be noted that separation and textile fabrication processing have an impact on the fibre's quality and appearance. Thus, fibres in textile products do not necessarily display the same morphology as fibres in plants. This issue is addressed in chapter 2.3.2.

Furthermore, in this thesis, the term "plant fibre" is used consequently for materials extracted from plants and used to make textiles. Another expression, that can also be found in the literature, is "vegetable fibre".

Characteristic features of plant fibres have been a subject of numerous publications with elaborated overviews on how to distinguish between different species [26-32]. This chapter does not aim to explain the details of characteristic and distinguishing features, which is the topic of chapter 2.2.1. It aims to discuss the morphology of parts of the plants, to explain the proper terms used in later specialized chapters.

Natural plant fibres used for textile production can be divided into three main groups depending on what part of a plant they come from or which type of plant: monocotyledonous (monocots) or dicotyledonous (dicots). The seeds of monocots

contain typically only one embryonic leaf - called cotyledon, whereas the seeds of dicots contain two embryonic leaves.

A: Herbaceous and arboreal bast fibres (dicots)

B: Seed/fruit hairs (dicots)

C: Leave fibres (monocots)

All plant fibres as listed above are quite similar in appearance (which makes species identification so difficult). Plant fibres are built up of long and narrow cells, which consist of empty space (lumen) surrounded by a layered cell wall (see section 2.1.1) for a detailed discussion of the cell wall). Some species have remains of so-called protoplasm inside the lumen that can have a thin ribbon-like appearance, e.g., ramie and flax [26, p.124]. The fibre cells elongate during plant maturation. Thus, immature fibres are shorter than mature ones. The length of a fibre is closely related to the quality of a material – the longer the fibre – the finer thread can be spun.

2.1.1 Structure of a fibre

Cells of plant fibres and hairs have a similar structure as mentioned at the beginning of this chapter. The structure of a fibre (*Figure 1*) consists of a central empty space (lumen) surrounded by a cell wall which divides into a) primary- and b) secondary cell-wall, which again is divided into three sections (S_1, S_2 and S_3) as well as c) middle lamella or intercellular layer, which fills/divides the space between two neighbouring cells, see further explanation below. Some authors refer to an additional

tertiary cell wall that is the innermost part of a cell [33, 34].

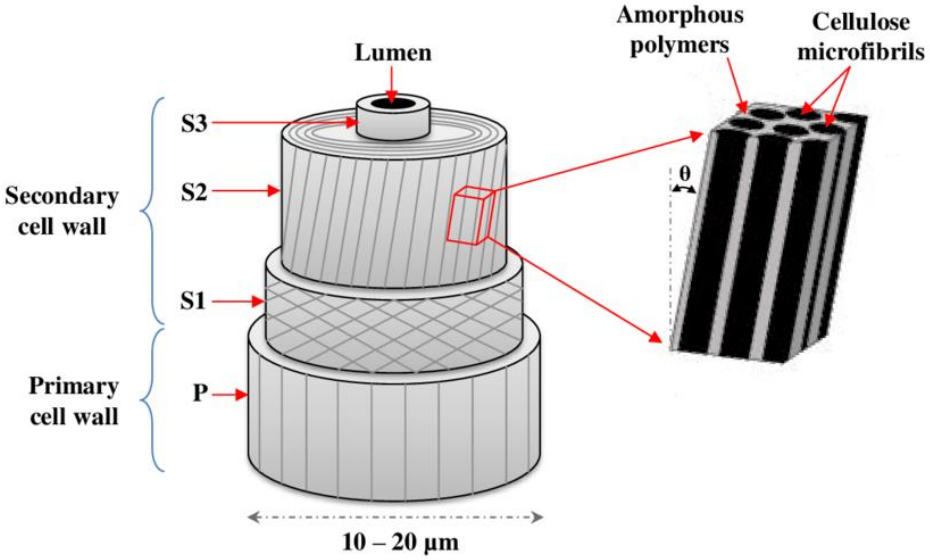

Figure 1: Structure of fibre cell, which consists of primary- and secondary cell-wall and central cavity – lumen. The secondary cell wall is divided into three sections S_1, S_2 and S_3, © Chegdani, F., El Mansori, M., Bukkapatnam, S., Reddy, J. N., open access: HAL Id: hal-02637097.

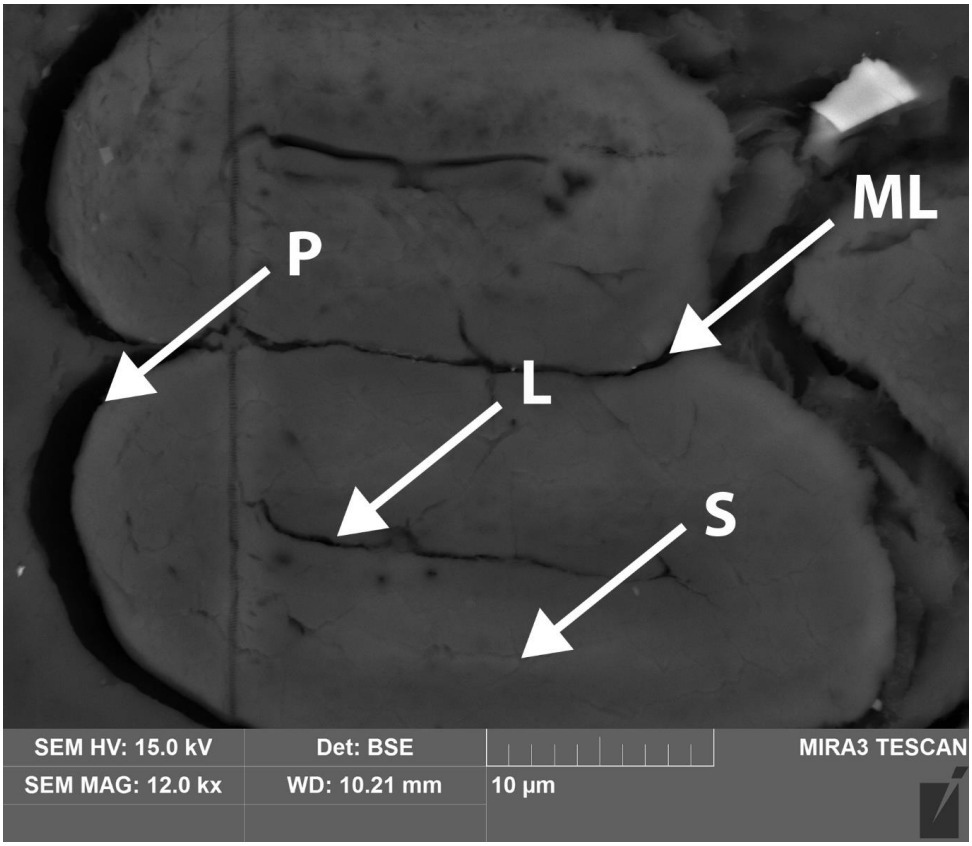

Figure 2: Fibre cell structure of hemp (Cannabis sativa) in cross-section: Two fibre cells above each other: primary wall (P), secondary cell wall (S), lumen (L) and middle lamella (ML), SEM-BSE image of hemp, © Lešniaková & Lukesova.

The middle lamella (see *Figure 2*) does not count as a proper wall, even though it shows up as a barrier. The middle lamella holds two individual cells together. It consists of pectic substances (a complex set of polysaccharides mainly) which can be dissolved by the enzyme pectinase. This process is used to obtain single cells. The middle lamella is isotropic [25, p.50-55, 35, p.26-28].

The cell walls consist mainly of cellulose molecule chains gathered in so-called microfibrils (*Figure 1* and *Figure 3*).

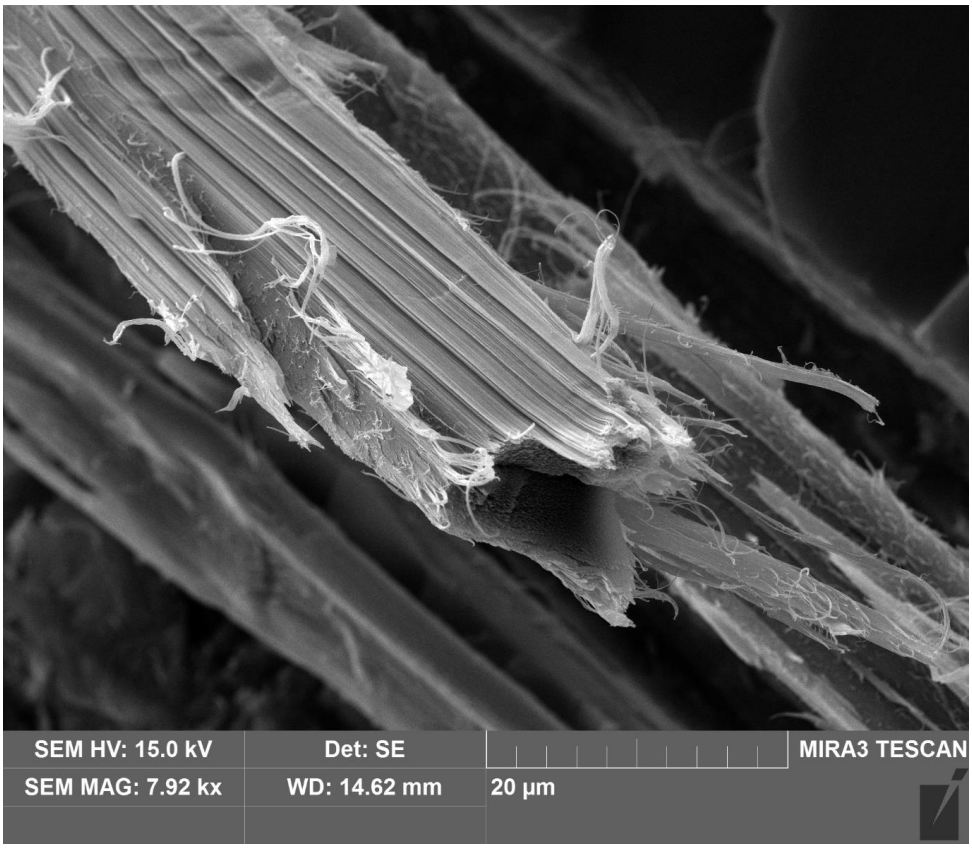

Figure 3: Microfibrils of nettle (Urtica dioica) – internal structure of a split fibre showing microfibrils in the secondary cell wall, SEM-SE image, © Lešniaková & Lukesova.

The primary wall (see *Figure 1*) is the first real wall of a cell. The primary wall is anisotropic (explained in chapter 2.2.2). It contains cellulose, hemicellulose and pectic substances. The microfibrils of the primary wall are often interwoven since the cell wall needs to expand significantly at the beginning when the cell grows. This affects the orientation of microfibrils that are distorted.

The secondary wall consists of three sublayers (S_1, S_2, S_3) (*Figure 1*), where the S_2 layer is usually significantly thicker than the other ones. This cell-wall structure is essential for the performance of the modified Herzog test (2.2.2). In the secondary wall, the cellulose fibrils are highly ordered with bundles of macro fibrils running around the fibre's longitudinal axis in spirals. Lignin may or may not be present.

Pectic compounds are usually lacking. The angels of inclination of the microfibrils differ in the sublayers S_1, S_2 and S_3. This is discussed further in section 2.2.2.

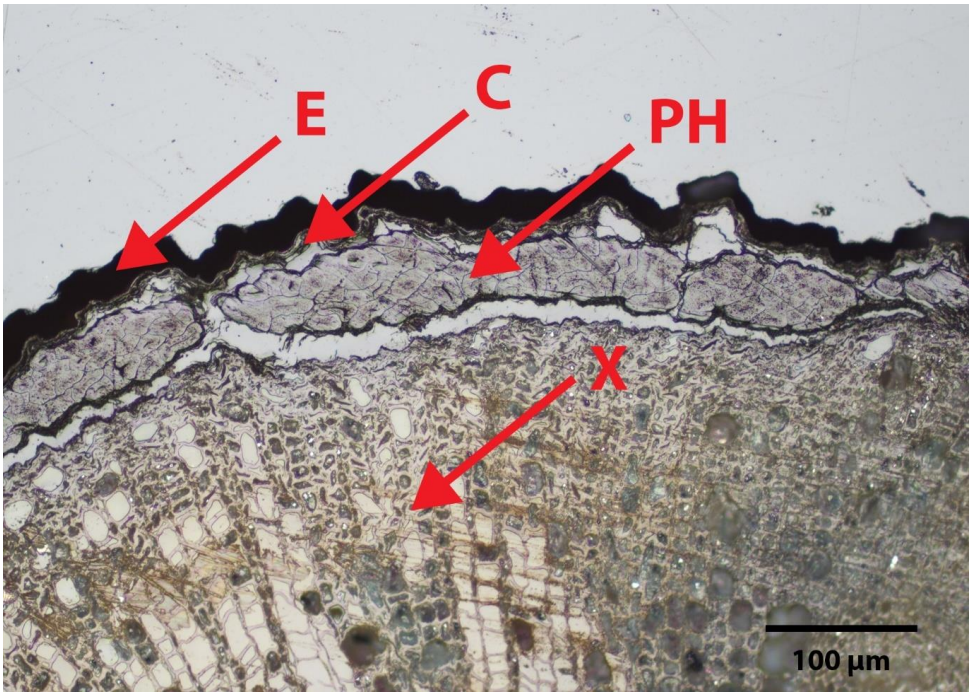

*Figure 4: Cross-section of an immature flax stem (*Linum usitatissimum*): E = epidermis, C = cortex, PH = phloem with sclerenchyma bast fibre bundles, X = xylem, all features are marked with red pillows. Optical microscopy image in reflected light modus, © Lukesova.*

2.1.2 Herbaceous and arboreal bast fibres

Bast fibres are part of the plant's vascular system, transporting water and nutrition through the stem/trunk. They are located in bundles in the phloem (inner bark) of certain dicotyledonous plants (*Figure 4*). Phloem is one of the two types of transport tissue in vascular plants- the other is xylem. The intercellular space is filled by pectin. They are called *extraxylary* fibres since they grow outside of the xylem. In contrast to *xylary* fibres such as *libriform fibres* and *fibre-tracheids* [35, p.86-88] that have not been used for textile production but are used in modern paper production [36, p.300-308]. Mature bast fibres often have lignified cell walls. The lignification varies between different species [37]. Two fibre cells are separated by a lamella. Flax lamellae can reach a thickness of 0,1-0,2 µm [35, p.86-88].

Bast fibres often contain so-called dislocations also referred to as nodes (*Figure 5*), which are disturbances along the longitudinal direction of a fibre [38]. The angle of the microfibrils relative to the fibre axis differs in these regions from the angle found in the surrounding cell wall [39, p.558]. The term cross markings, which has also been used in this context, describes narrower and less directional features that are often appearing in clusters forming an X-form [40, p.955], *Figure 6*. It has been suggested these features occur as a cell-wall distortion caused mechanically, i.e. by compression during the plant growth [28, p.2] or even during processing [26, p.121-122].

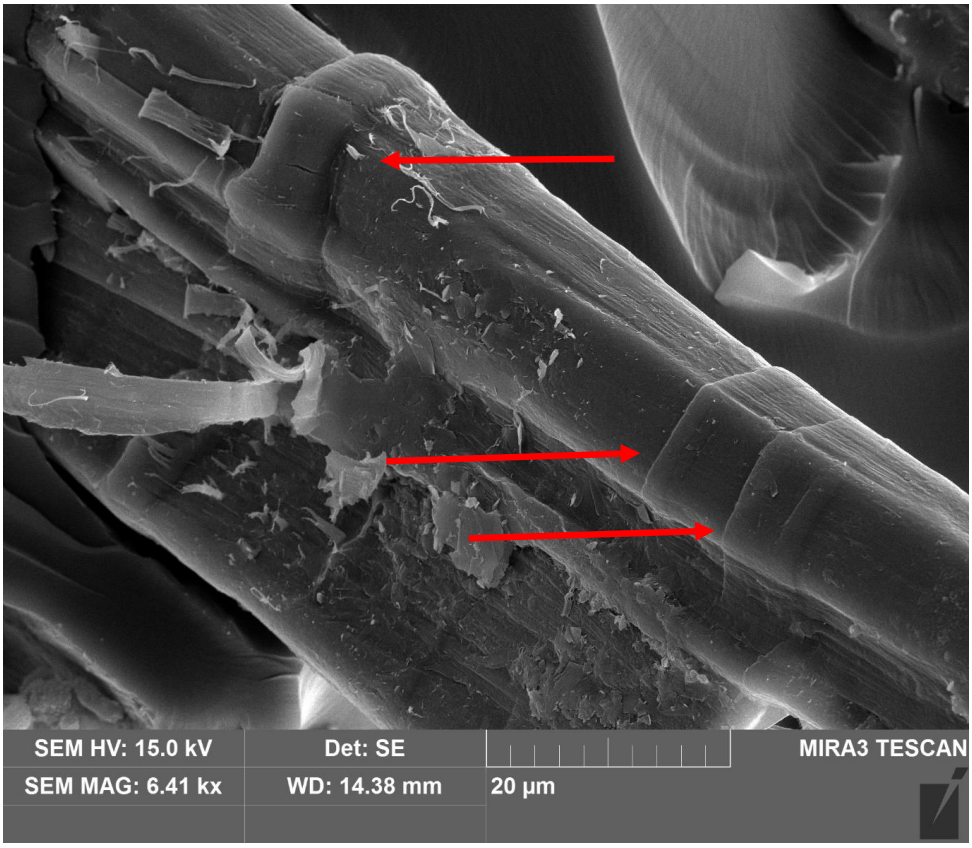

*Figure 5: Flax fibre (*Linum usitatissimum*); arrows point towards dislocations (nodes); SEM-SE micrograph, © Lešniaková & Lukesova.*

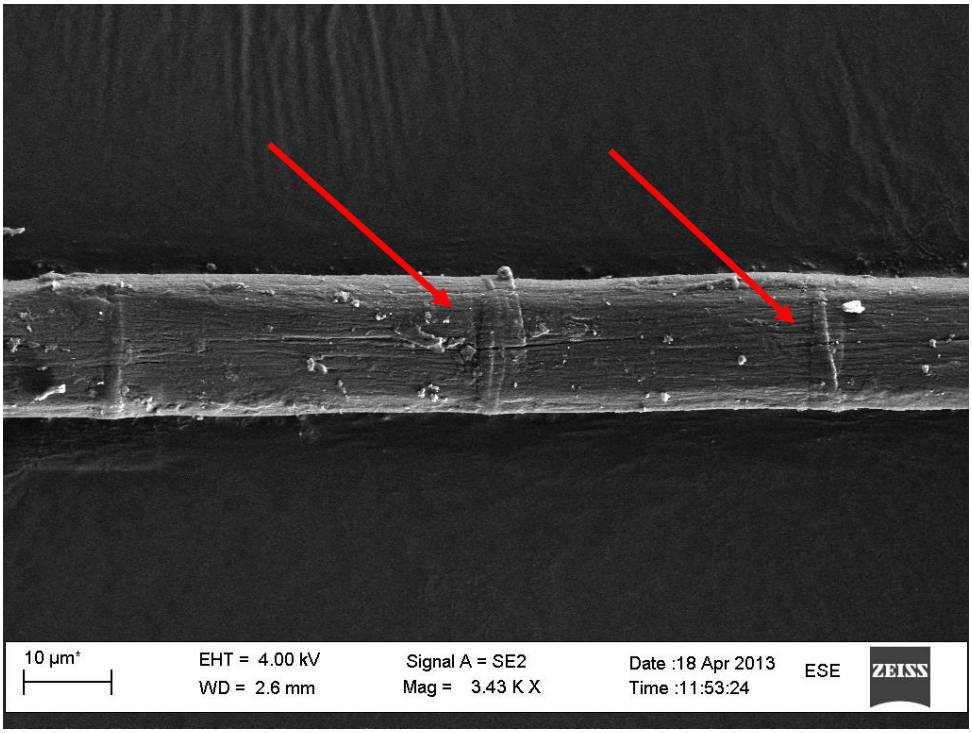

Figure 6: Flax fibre (Linum usitatissimum); arrows point towards cross-markings; SEM-SE micrograph, © Erichsen & Lukesova.

Bast fibres differ only very little in the content of cellulose: Flax 64,1%; Hemp 67%; Jute 64,4% and Ramie 68,6%. Whereas they differ in the content of lignin: Flax 2,0%; Hemp 3,3%; Jute 11,8% and Ramie 0,6% [41, p.31]. This is used for identification by the FTIR techniques, which is explained in chapter 2.2.6.

2.1.3 Seed and fruit hairs

Seed and fruit hairs are fine epidermal hairs also called trichomes. Many trichomes next to each other constitute a so-called indumentum, which is a covering having mainly a protection function. Cotton (*Gossypium sp.*) is nowadays the most important commercial seed hair. Growing, spinning, and weaving cotton was introduced in Europe by the Moors in Spain around the 10th century. However, cotton consumption in Europe was minor compared to the use of bast fibre products until the early 19th century [42]. Four species of cotton have been utilized for textile production. All four species were domesticated in antiquity (*Gossypium hirsutum, Gossypium barbadense,*

Gossypium arboretum, Gossypium herbaceum). One cotton hair consists of a single cell (is unicellular) and develops secondary walls at maturity [25, p.74]. S_1

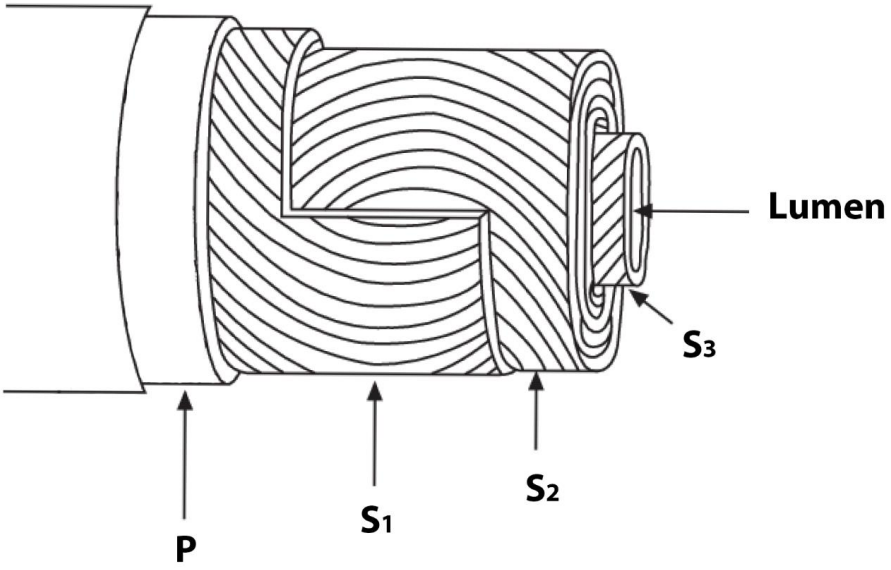

Figure 7: An illustration of cell-wall structure of the cotton fibre, after Morton and Hearle 1975 [43]

As for other plant fibre types, seed and fruit hairs consists of a primary- and secondary cell wall as well as a lumen (*Figure 7*). The primary wall of cotton consists of non-cellulosic materials such as pectin, hemicellulose, and amorphous cellulose, where the macro fibrils are oriented in a random criss-cross pattern. In the secondary wall, the microfibrils are highly organized, running parallel to each other and form a wavy structure. Cotton fibres do not contain dislocations and show convolutions (*Figure 8*). Convolutions are multiple fibre twists that can also have changing directions. The convolutions are denser by fully ripped fibres, they start to form first after the cotton ball opens. Immature fibres contain very little or almost no convolutions [32, p.2]. Cotton contains 82,7% cellulose and 0% lignin [41, p.31].

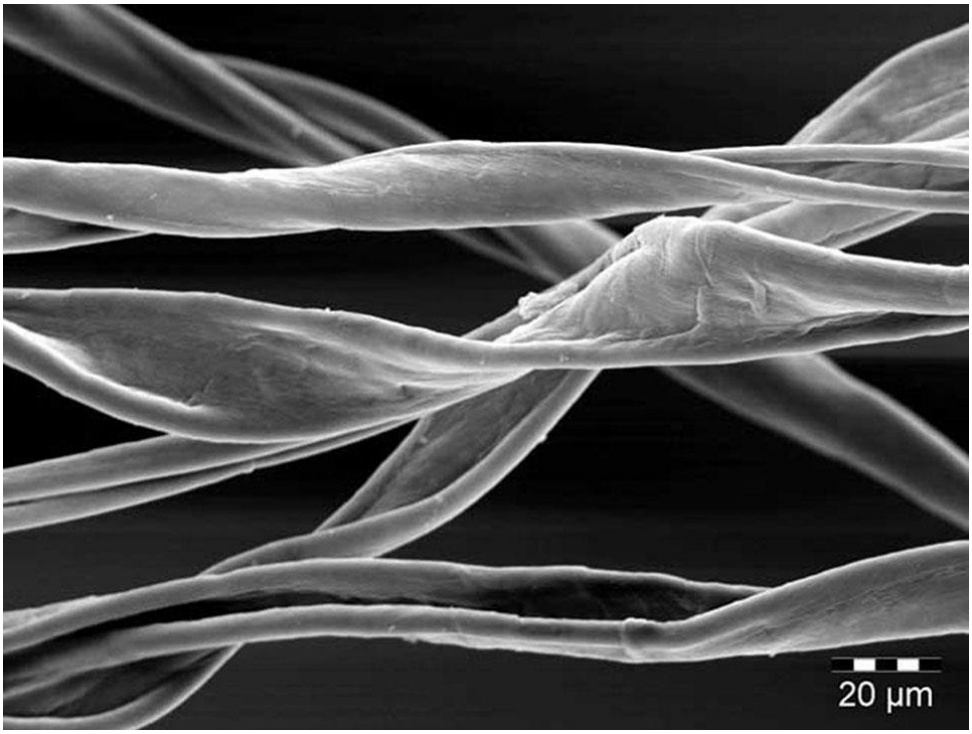

Figure 8: Convoluted cotton fibres, SEM-SE; © Dochia, Sirghie [44]

Other materials belonging to this group are kapok and milkweed (*Asclepias syriaca*). Kapok comes from the inner surface of a fruit capsule *Ceiba pentandra*. Unlike cotton, kapok hair does not have any convolutions and contains a large lumen. Kapok contains 43,2% cellulose, 32,4% hemicellulose and an extremely high content of lignin 15,1% compared to other hairs [41, p.31]. As for milkweed, the fibres are relatively brittle and rather difficult to spin, they are typically used as filling material in pillows and similar. Other examples are for instance hairs of poplar and willow [32, p.8-10].

2.1.4 Leaf fibres

Leaf fibres are extracted from the leaves of monocotyledonous plants. They are sometimes called hard fibres because many of them contain more lignin in the cell walls than bast fibres and therefore they often have a stiff texture [25, p.60]. Sisal extracted from agave (*Agave sisalana*) contains up to 14% lignin and 72 % of cellulose [45, p.148]. The fibre cells are very long (up to 8 mm). The primary wall is

porose, a lumen is present. Other examples of leaf fibres are New Zealand Flax (*Phormium tenax*) and Cordyline/Cabbage-tree (*Cordyline australis*).

2.2 A Review of Identification Techniques Applied so Far

This chapter presents an overview of fibre identification techniques that have been applied so far for the identification of plant fibres in archaeological and historical objects. It should be noted that the main purpose of the chapter is to evaluate publications that describe fibre identification methodology. While some application papers are also cited, the chapter does not aim to evaluate articles dedicated to the application of fibre testing method on archaeological and historical objects, such an evaluation would be a very valid contribution since in the cause of this work several papers were found that either applied wrong methods or applied valid methods wrongly. However, it is beyond the scope of this thesis objectives.

2.2.1 Classical Optical Microscopy

The classical fibre identification using transmitted light microscopy has been a scientific subject matter for more than 100 years. Authors such as von Höhnel, Luniak, Herzog, Isenberg, McCrone, Catling and Grayson, Wülfert, Petraco and Kubic, Houck, Holst, Nayak and Padhyde and Marková [26-28, 30, 32, 36, 40, 46-52] and many others contributed to the topic of fibre analysis and developed a wide range of technical and methodological procedures. Investigation of cultural heritage material is in many ways specific. Working with cultural heritage samples needs to adapt methods because the studied material was changed through numerous processing steps different from the modern ones, degradation, and sample amount usually needs to be very small due to ethical issues [36, 53-55].

Microscopic examination in transmitted light provides information about the size, shape, and surface- as well as internal morphology of fibres. Characteristic features of different species have been summarized in various atlases and other publications as mentioned above. Note that there is a difference between characteristic features and distinguishing features that can be used for identification. Different fibre species may

share characteristic features whereas distinguishing features distinguish species from each other.

Fibres have been studied in a longitudinal direction as well as in cross-section [32, 51, 56]. The use of fibre cross-sections for distinguishing species is discussed in detail in a separate paper published as part of this thesis work (2.3.2).

The following features have been used as distinguishing for identification: i) dislocations/nodes and cross-markings, ii) fibre length, cross-section diameter, lumen diameter, cross-section shape and lumen shape and fibre cell ends, cell structure (such as convolutions and flexions), crystals, and adhering tissue.

i) Dislocations/nodes and cross-markings

The use of dislocations/nodes and cross-markings for differentiation between specific herbaceous fibres was refuted already in the 50ties [26, 28, 40, 57]. When textiles from the mid of the19th century onwards are examined, it should be remembered that these features can eliminate or disappear after maceration. Maceration is an alkali treatment in the textile industry, which is used instead of water- and dew retting when extracting bast fibres from plants and for achieving a better quality of cotton. Maceration was invented by John Mercer in 1844.

While dislocations/nodes and cross-markings cannot be used for differentiation between specific herbaceous fibres, they can be used to sort fibres into categories: herbaceous bast fibres that contain these features, whereas plant hairs (such as cotton) do not [32, p.12]. Nodes can even help to distinguish between the herbaceous bast fibres and arboreal bast fibres of lime tree (*tilila cordata*), which do not contain nodes [58, p.412]. Caution must be taken in case of modern bast fibres that can be heavily macerated causing mitigation of these features as mentioned above.

ii) Fibre Length and Diameter

Luniak highlights that there is a big variation in fibre length- and diameter dimensions. He does not exclude these features completely but calls for caution when using them for analytical purposes [26, p.121]. This is misunderstood by Carr et al.

[59, p.79-83] that refer to Luniak's fibre length and diameter measurements as if they were distinguishing features. Fibre diameter was refuted as a distinguishing feature based on a comparison of measurements of various microscopists [50], see Appendix B.

On the other hand, Luniak finds the cross-section shape and lumen shape as valuable distinguishing feature. The lumen size is for him a useful indicator, even though the diameter is not constant to any marked degree [26, p.121-122].

Herzog points out that the features of fibre cell length and fibre diameter vary even more within different parts of one plant than between different species. He compares the middle part of flax and hemp. Besides, he mentions that the growing conditions, as well as the density of sow (scattering the seeds), play an important role in the quality of a plant and thus a form and a shape of fibre [27, p.250-253].

Unfortunately, as the recent research and the article (1), which is part of this thesis work (chapter 2.3.2) show, the features mentioned above (cross-section shape and lumen shape) cannot be employed on their own for differentiation of species [50, 60]. Especially not, when dealing with small sample amounts as is the case in historical/archaeological plant fibre identification.

However, features like convolutions that were explained above (chapter 2.1.3) and twists that are sporadic fibre twists/flexions (*Figure 9*), can help with identification. For instance, cotton can easily be distinguished from kapok due to convolutions that change direction; nettle can be distinguished from flax due to twists/flexions. Caution must be taken with modern macerated samples as mentioned above. Maceration of cotton was introduced since it improves an affinity to dyestuffs, hygroscopicity and tensile strength. Maceration under tension causes a significant change in fibre's cross-section resulting in a cylindric shape and it gets thus a silk-like lustre on the surface. Cotton may deconvolute completely, which makes the identification intrigue [41, p.31, 61].

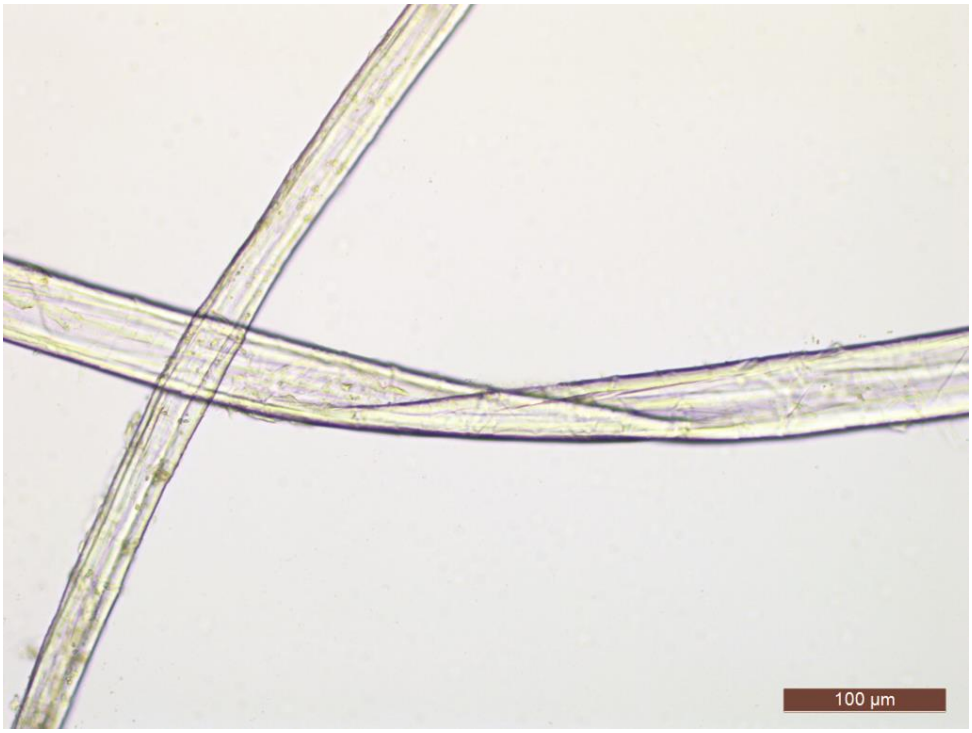

Figure 9: Twisted fibre, which is a sporadic flexion compared to convolutions that are multiple flexions, often of a three-dimensional character. This figure shows nettle, Urtica dioica (compare with convolutions of cotton Figure 8), transmitted light microscopy micrograph, © Lukesova.

The presence of crystals and associated tissue (adhering to fibres caused by insufficient fibre extraction) as special features of epidermal- and/or parenchyma cells can be used as an identifying feature in combination with other features [26, p.124, 27, p.253-259, 50, 62].

Petraco and Kubic state that features such as cell size, cross markings, cell shape, lumen shape and crystal shapes and cell structure can be used to determine classification between different fibre categories [49, p.89]. They combine these features with other optical properties in polarized light to determine various plant fibre species. This will be discussed closer in chapter 2.2.2.

An overview of the evaluation of different morphological features commented in literature is presented in the table included in Appendix B.

Characteristic features do not distinguish between species on their own. They can sometimes be used as an indication in combination with other tests e.g., microchemical tests and/or polarized light microscopy (chapter 2.2.2).

Characteristic features should not be confused with distinguishing features. Studying the earlier microscopists, one can notice there is a clear shift in the timeline: the first authors [27, 46, 63] performed many measurements and came with rather modest claims. The second-generation elaborated it and drew conclusions [26, 64]. The generations coming after often reused, what has been written in a rather simplified way, and claimed characteristic features to be distinguishing features which can be very misleading [58, p.412, 59, p.79-83, 65].

2.2.2 Polarized Light Microscopy

The use of polarized light microscopy on fibres

Normal white light consists of electromagnetic waves that are oscillating perpendicular to the direction of propagation in all directions. In a transmitted polarized light microscope, two crossed polarizing filters are placed in a light path. The first polarizing filter (the polarizer) is located below the specimen and only light waves oscillating in one specific direction are passing through it. The light passes through a specimen to the second polarizing filter (the analyzer). Polarized Light Microscopy (PLM) is suitable for the investigation of so-called birefringent materials where the refraction of light depends on the polarisation. Bast fibres are birefringent due to the highly oriented crystalline cellulose chains running around the fibre's central axis in a helix [41, p.11].

The technique has proven to be very applicable and reliable not only because many characteristic features such as dislocations, crystals, convolutions, and adhering tissue are enhanced in polarized light, but also because it provides valuable analytical data [26, 49, 63, 66]. PLM was recently evaluated for the identification of different plant fibre materials native to New Zealand commonly used to produce Māori textiles [67]. Three different Phormium subspecies (New Zealand flax (Harakeke) *Phormium tenax*, coastal flax (Whaririki) *Phormium cookianum - subspecies hookeri*, mountain

flax (Whararikī) *Phormium cookianum - subspecies cookianum*), three different Cordyline species (cabbage tree (Tī kōuka) *Cordyline australis*, forest cabbage tree (Tī ngahere) *Cordyline banksii*, mountain cabbage tree (Tī tōī) *Cordyline indivisa* and Kiekie *Freycinetia banksia* were studied. It was demonstrated that morphological and birefringent features observed when using PLM have a potential to distinguish between- and within-plant genera.

Another study is a comparison of inner bark fibre cells from New Zealand genera (Hoheria and Plagianthus) and Pacific genera (Artocarpus, Broussonetia and Ficus) used for making of bark cloth (tapa) was presented [68]. Smith et al. confirm the ability of PLM to use morphological features as well as optical properties of fibres to distinguish the New Zealand and Pacific genera from each other. However, the six species from New Zealand - Hoheria and Plagianthus genera cannot be distinguished from each other. Fourier-transform infrared spectroscopy in attenuated total reflectance modus (ATR-FTIR) was used to distinguish between different groups of bark cloth materials [69]. The discussion of the method is in chapter 2.2.6.

The Modified Herzog Test on plant fibres

The (modified) Herzog test known since 1920's [63, 66] has been reported in literature [26, 27, 36, 50] and re-examined by a mathematical model recently [40]. It was concluded that it is one of the easiest and most reliable methods for distinguishing different plant fibre groups from each other [40]. The test has been demonstrated as an educational video (https://www.youtube.com/watch?v=sC9GlUKjBDE).

A proper microscope setup, including Köhler's Illumination, which uses the potential of the numerical aperture of the lens-system completely and spreads the light over the image evenly with no over-or underexposed areas, is necessary for getting a clear image for the test. It is also important to pick a proper section of a fibre to test. Generally, thicker parts of single fibres are most suitable for the test. The ideal fibre section does not have any nodes or cross marks that disturb the crystalline structure. A focus at the top of the fibre is required for a reliable result [70], see chapter 2.3.1,

article 5). Such a place should show dark grey (black) extinction when the polarization filters are crossed, and the fibre is placed in an orthogonal position relative to one of the polarisation filters (East-West or North-South).

The secondary cell wall of plant fibres is built from several sublayers (S_1, S_2, S_3) as explained in chapter 2.1.1. As discussed here the microfibrils rotate across the central axis of the fibre. The rotation can be right-handed or left-handed, referred to as twist. Flax and hemp have opposite twist directions of microfibrils in the S_1 sublayer of the secondary layer and this distinguishes the birefringent materials. In the Herzog test a so-called red-plate compensator is introduced in the light path, which converts the phase difference induced by the refractive interference difference into a colour difference and the two different twist directions can be distinguished from each other, which makes that S-direction appears blue (Indigo II) and Z-direction appears orange (Orange I) when oriented in the 0° position and exactly opposite (S-direction orange and Z-direction blue) when oriented in the 90° position. In Article 4, the modified Herzog test has been used to investigate the textiles from Norwegian Late Iron Age graves.

The Herzog test sometimes does not yield any clear result. This may have various reasons, but one point is that the secondary cell wall thickness can vary considerably, and this may influence the test result as pointed out by [40]. This is the reason why thicker fibres with a well-developed cell wall are preferred.

If more species than flax and hemp come into question, additional characteristic features such as fibres' morphology, associated tissue, presence, of crystals (as well as their shape and chemical composition) and/or swelling behaviour must be used for fibre identification.

The modified Herzog test can distinguish between S- or Z-twist of the S_1 sublayer of the secondary layer. This layer is hidden under the surface and its direction can therefore not be distinguished by SEM analysis of epidermis as wrongly reported [71, p.90]. Further discussion on the confusion of the use of different microscopic techniques applied on historical samples can be found in chapter 4.1.

2.2.3 Scanning electron microscopy

In Scanning Electron Microscopy (SEM) an electron beam is produced, focused, and scanned to raster an image or another type of information as e.g., element spectra. The signals are produced from the electron-beam – specimen interaction. Scanning electron microscopes reach significantly higher resolution (r) than light microscopes because the wavelength of the electrons is much smaller than the wavelength of visible light used in optical microscopes. The smaller wavelength also leads to a much higher depth of field than in conventional optical microscopes [72].

In SEM, the detected signals come from the outermost part of the sample, the penetration depth is typically around 1 micron, thus, in contrast to transmitted light microscopy, it does not yield any information about the inner structure of the fibre [72, p.197-198].

The main signals produced are secondary electrons (SE), Back-scattered electrons (BSE), X-rays (EDS) and Auger electrons. Secondary electrons are by far the most used imaging signal in SEM for studying fibres [72, p.51-54].

The SEM techniques can be useful for fibre identification regarding features that are of an external character, such as the identification of animal hairs through the presence of scales (*Figure 10*) or nodes and dislocations (*Figure 5* and *Figure 6*), which can be used to separate between herbaceous and arboreal fibres (such as hemp and lime tree bast). Phytoliths (silica crystals) can be identified through elemental analysis [50] ideally combined with microdiffraction. SEM is particularly useful for the investigation of carbonized samples that cannot be investigated with transmitted light microscopy.

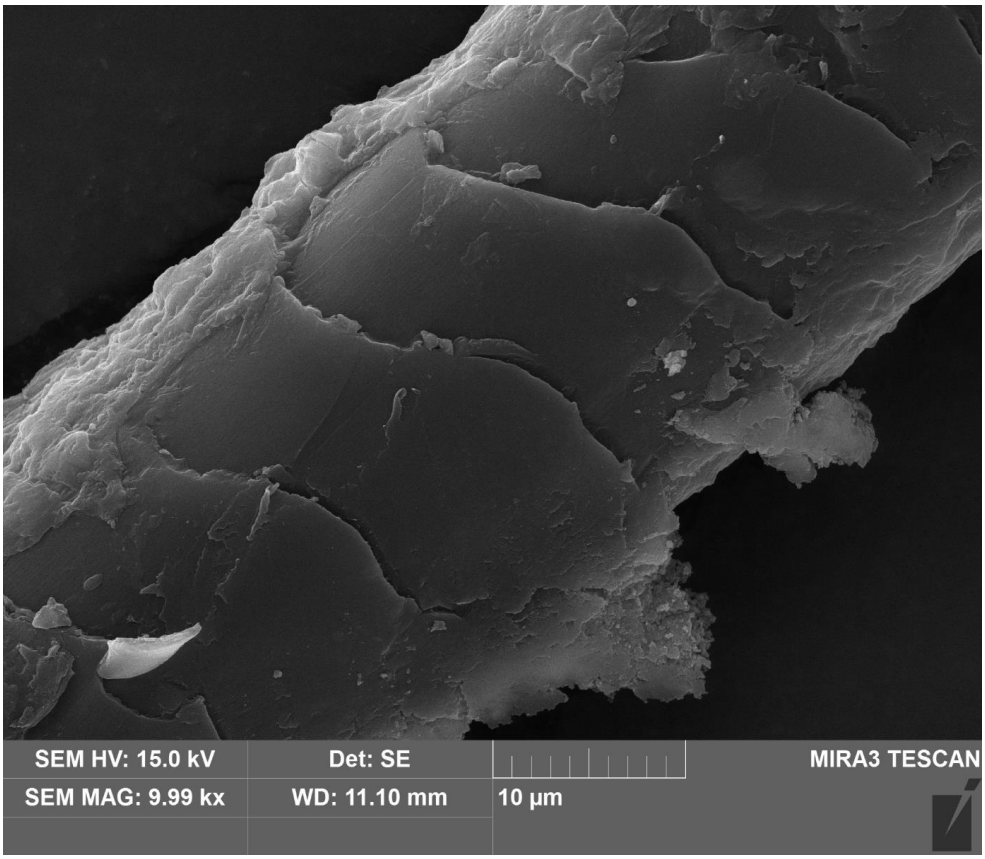

Figure 10: Scales on an archaeological animal hair with a regular distance of about 10 µm, SEM-SE micrograph, © Lešniaková & Lukesova.

An extensive collection of SEM images of animal- and plant archaeological fibres, with some inclusion of modern references, was recently published [71]. Unfortunately, this otherwise beautiful piece of work contains a range of unsubstantiated claims as to what can be inferred from the SEM images.

Another recent application of the SEM in fibre identification is the FIBRANET project [73] which also contains optical microscopy images. The very attractive idea behind this project is to provide an online database that presents micrographs of various fibre species in longitudinal and cross-section view that were aged artificially by laboratory carbonization and soil burial. Micrographs of untreated reference samples complete the database for identification purposes. A long list of

identification criteria is provided, and the idea is that by clicking on features it should be possible to arrive at the right fibre. Unfortunately, at the moment the database is not supported by proper documentation on how and on what scientific basis the selection criteria were chosen. Many of the selection criteria are not established in the textile community and it is thus difficult to infer what they actually refer to. Furthermore, a list of fibre types included in the database is lacking. A search suggests that only the most common plant fibres used in history are included. Hops is not included.

2.2.4 Ancient DNA analysis

Deoxyribonucleic acid (DNA) is a molecular hereditary material in all eukaryotic organisms, which is stored in cell nuclei and mitochondria. A DNA molecule constitutes two chains forming a double helix carrying all genetic information, which can be coded employing DNA analysis.

Studies exploiting DNA analysis of modern samples are used for phylogenetics - a systematic discipline dealing with relationships among species and consequences of their evolutionary history. On the contrary, ancient DNA analysis (aDNA) has been used in current archaeometry more and more; often for the reconstruction of population histories as well as for various studies that need distinguishing between plant and animal species [74-76]. Ancient materials used for DNA extraction traditionally are bones, teeth, and seeds. However, hair, skins and feather have also been proven as a possible material source [77], especially after breakthroughs in sequencing technologies, in particular, the “second generation” sequencers [78]. Advances in laboratory techniques made it possible to gain genetic information from many other archaeological materials that are often degraded [79]. The oldest genetic data are from the Pleistocene and has been gained from permafrost, where conditions are stable, dry, and cold [80, 81].

However, DNA barcoding of archaeological plant fibres is highly problematic due to the degradation of DNA material, which often means a lack of recoverable DNA. It can be induced by many reasons such as changes in temperature and/or pH,

hydrolysis, oxidation, photodegradation, actions caused by enzymes and microorganisms and background radiation. Generally, it is challenging to gain well-preserved DNA from hot and humid conditions. Many factors contribute to different degradation grade of preservation of DNA, that can vary within a single site or even within different samples coming from the same material from an object significantly [78]. DNA degradation in an archaeological context is a complex matter that still contains many question marks. Besides, contamination in form of exogenous DNA can also cause challenges, since it is often difficult to differentiate which bands belong to an original sample (endogenous DNA) and which ones to a contaminant [74].

DNA extracted from modern hair offer both mitochondrial DNA (mtDNA) and nuclear DNA (nuDNA) that is well amplifiable. Generally, a hair root contains high-quality DNA and the non-root part, poor quality. But even the hair root analysis of aDNA is challenging. However, it has been proven by PCR-based studies over the past two decades, that it is especially the ancient mtDNA that can give reliable results of archaeological samples if the mtDNA is well preserved [82]. Occasionally, very short fragments of nuDNA of ancient material can be recovered [83].

Plant fibres are formed of single-cell units called sclereids that are dead when a plant is still living. Even freshly extracted fibres from modern plants contain very little DNA. Some species (e.g., flax) show a very thin ribbon-like structure inside of lumen called plasma when observing microscopically under normal transmitted light. The question, if a plant textile material can give DNA data, was investigated earlier [84]. Coarse textiles (such as ropes) made of modern fibres, that were not processed thoroughly may sometimes contain remains of epidermis that have mtDNA useful for testing. If the studied textile material contains not only textile fibres but also parenchymal cells, there is a higher chance to perform mtDNA analysis [84, p.109]. Another study [85] investigated aDNA from rope and fabric preserved in the Christmas Cave in Israel. In this case, it was possible to extract amplifiable DNA. However, numerous challenges have been shown in this study. The reason for the

exceptional preservation state of the material was most probably the very dry and stable climate in the Christmas Cave.

The retting process, when fibres are extracted from plants, causes degradation of the little amount of nuDNA so that even modern fibres are difficult to identify with DNA analysis [22, 75, 84, 86]. Most of the excavation sites are exposed to changes in relative humidity and the possible damage of DNA by hydrolytic processes is extremely high. The use of DNA analysis of plant fibres is therefore very limited.

2.2.5 X-Ray Diffraction

Materials having highly organized structures on the atomic level can be investigated with X-rays. X-rays are electromagnetic radiation (light) with a very short wavelength that makes it possible to do diffraction at the atomic scale.

As mentioned earlier bast fibres contain crystalline and amorphous regions. Therefore, measurements of crystalline structures can help with the characterization of fibres using X-ray diffraction. The different rotations of the microfibrils will lead to different diffraction patterns.

Because fibres are so small, the preferred method is X-ray micro-beam diffraction (μXRD) [87]. This technique requires synchrotron radiation. Such instruments are unique, and it is therefore not easy to get an instrument booked. The analysis is costly, and it is hard to expect it could be used for massive investigations of historical textiles. So, it is a possibility, but only in very special cases. The method is primarily of interest in the case of archaeological fibres which cannot be investigated with the modified Herzog test. Müller et al. showed that it is possible to obtain diffraction patterns good enough to identify the twist from archaeological fibres [88, 89].

2.2.6 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier-transform infrared spectroscopy (FTIR) relies on the absorption or emission of infrared light obtained from a specimen. An FTIR spectrometer collects data over a wide spectral range (400–4000 cm^{-1}). Recently, portable instruments allowing in situ measurements have become available.

It is the bonds of the chemical compounds of a studied substance – that give a characteristic spectrum on absorption or emission of electromagnetic radiation. Attenuated total reflectance FTIR (ATR-FTIR) is a special mode of this technique that collects data from a specimen's surface. The chemical composition of plant fibres is very similar; however, it has been reported that ATR-FTIR is suitable for distinguishing between groups of plant fibre species – the following species were tested: flax, hemp, jute, ramie, cotton, sisal [37]. The procedure is based on measurements of lignin content, namely the comparison of two ratios: lignin-to-cellulose ratio (R_1) and lignin to the organic material ratio (R_2). Native and processed fibres were compared with the conclusion, that the processed ones contain less lignin.

Polarized ATR-FTIR was used to distinguish between flax and hemp [90]. The study shows the presence of various di-choric and non-dichoric bands in both species, that can be used for identification purposes. The difference between ratios of specific band intensities (crystallinity indices) should indicate that a specimen is either hemp or flax. Negative values indicate flax, whereas positive values indicate hemp.

However, a very recent study on the degradation processes of bast fibres (flax and hemp) based on examination of modern-, historical- and accelerated aged fibres shows that the degradation process influences the IR spectra of the fibres to the point of making them spectrally indistinguishable [91].

2.2.7 Raman Spectroscopy

Raman spectroscopy excites molecular vibrations through monochromatic irradiation in the visible (VIS) light region, ultraviolet- (UV) or infrared (IR) region. The last frequency range has been reported as the most useful in the field of Archaeometry [92, 93].

Raman spectroscopy is suitable for delicate specimens due to the use of low power lasers. The technique has been applied for the identification of modern and archaeological plant fibres [94, 95].

Raman spectrometers have recently become available as portable instruments that can be used in situ as shown in several studies [96, 97]. This is a great benefit for studies on cultural heritage since no sampling is needed if an instrument can be moved to a studied object.

Edwards et al. studied the use of FT Raman Spectroscopy on ancient flax, modern flax, jute, kapok, sisal and coconut fibres with the conclusion that the technique can be used for distinguishing of species and indicated future possibilities for the application of this technique to archaeological textiles [95].

However, Raman spectroscopy has the same difficulties with the identification of degraded organic material as reported for FTIR [98].

2.3 This thesis work: Plant fibre identification methods

Even though plant fibre identification has been a matter of research interest in many decades, there are still areas to discover especially with regards to the application of the methods on historical fibres and/or less known species used in history.

2.3.1 Sample Preparation and specific behaviour of historical material (Article 5)

This article examines issues related to sample preparation, manipulation, and investigation of degraded fibres. Fibre analysis of historical/archaeological material is in many ways different from the study of modern material. This is often overseen, and researchers tend to use procedures developed for the textile industry. Such procedures are not always appropriate for research on cultural heritage. Article 5 can also be understood as a supplement to articles 2 and 4, describing in detail the sampling of fibre material from historical objects, including ethical considerations.

The way to a reliable result starts already before sampling a studied object. Good knowledge on a macroscopic level is a must together with a clear strategy regarding the research aim. This may sound obvious, but careful planning of sampling and sample preparation is crucial for a successful result since any sampling inevitably narrows down the focus from a whole object to a specific object area. A sample must

be representative for an object and for a research question. The choice of such a place is crucial for later investigation.

Cultural heritage objects are unreplaceable. It is necessary to consider the need for the research and to consider possible harm to a studied object. Many museums follow ICOM's ethical guidelines regarding treating cultural heritage (see chapter 3). Sampling of a cultural heritage object must be performed with the highest caution, documentation and use of appropriate tools like fine tweezers and surgical scissors.

Sample preparation requires concentration, time to breathe slowly, stereo- or digital microscope, ultra-fine tweezers, and tungsten needle [48, 70]. For transmitted light microscopy investigations, the choice of mounting media is essential because the difference in refractive indices of a mounting medium and a studied object ($\Delta n = n_{D1} - n_{D2}$) influences the object's visibility as phase contrast. This is illustrated with a small experiment, documented in *Figure 11*.

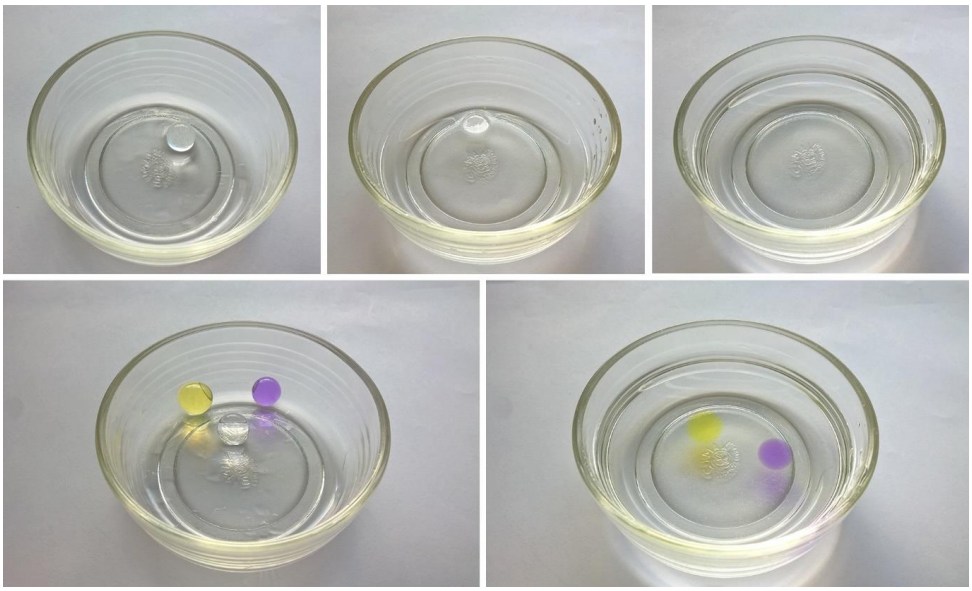

Figure 11: The difference between refractive indices of a transparent object and its mounting medium is crucial for the object's visibility: Left above: A gel bead ($n_D \approx 1{,}33$) is surrounded by air ($n_D \approx 1{,}00$); middle above: the same bead is half sunk in water ($n_D = 1{,}33$) – only its upper part, which is surrounded by air is visible; right above: the same bead is completely sunk in the water and is not visible, because refractive

indices of the bead and water are too similar. Bottom left: Two coloured beads and one transparent bead are surrounded by air. Bottom right: The three beads are completely sunk in water – only the two coloured ones are visible with blurred edges. The edges are blurred because there is only colour contrast and not phase contrast, © Lukesova.

This demonstrates that staining of transparent samples helps to enhance the contrast, but it cannot substitute the proper choice of mounting medium. Refracted indices of some selected fibres and refracted indices of mounting media are in Attachment II.

2.3.2 The use of fibre cross-sections for identification of species (Article 1)

Before this article, it had been shown that the features fibre diameter, lumen diameter, dislocations (nodes), and cross markings cannot be used on their own to distinguish between the typical bast fibres used for textiles in ancient Europe: flax, hemp, and nettle [50].

Cross-section shape and lumen shape of fibres have been used as characteristic features for a long time. The result in article 1 shows clearly that a cross-section shape and a lumen shape cannot be used as distinguishing features of plant fibres. Especially not, when only small sample amounts are available for an examination so that statistical analysis is not possible. Identifying small amounts of sample material is a situation one often faces. It needs to be taken almost as a prerequisite in the case of archaeological material identifications falling under the ethical guidelines of cultural heritage.

Excluding the two features clearly shows the need for reproving old methodologies that were developed for use in industry. Here, it is not a problem to procure a big amount of sample material (many fibres) that allow statistical analysis.

Another issue is the fact that some authors refer to characteristic features of cross-section shape and lumen shape of extracted fibres [26, 36, 65, 99], whereas others refer to fibre cross-section features observed in complete stems that contain unretted fibres [27, 28, 100]. This is a source of potential confusion because fibres may change morphology during processing: the size and shape of fibre's cross-section and presence of dislocations. There are also mentions in historical documents of different

harvesting time for flax. Usually, flax plants are harvested for fibres when the seed capsules start to get ripe and stems become yellow [101, p.7]. There are also mentions about the use of immature flax for fibres to procure particularly fine yarn, but for stronger cloth the stems were left until they turned yellow [58, p.152]. Therefore, in the article, tests on extracted fibres as well as fibres in plant stalks were performed and investigations were done on both mature and immature flax (*Figure 12*).

Figure 12: Left: Immature flax (Linum usitatissimum); right: mature flax (Linum usitatissimum); the state when harvested for fibres, © Lukesova.

2.4 Characterisation of less known species

The material resources of ancient societies differ from the modern ones. Not only the species that have been used as commercial fibres were used for textile production in history [13, p.578, 71, 102, p.122, 103, p.13, 104-106], see also Attachment I. This means that the subject of fibre identification of cultural heritage objects goes far beyond the application of results derived from fibre identification of modern materials. Even though there has been a raised interest in archaeobotanical studies on textile materials that can be seen in bigger projects like THEFBO and FIBRANET

[73, 107], as well as on smaller-scale individual research activities [67-69, 108], the need for knowledge that would cover all geographical areas and all epochs is enormous and obviously, there is a lack of systematic research on this fascinating topic.

However, as the conference Fibres in Early Textiles from Prehistory to AD 1600 showed, there is rising interest in the topic of fibre identification. A rich overview of contributions covering various species across the world was presented. The conference was held as the 16th conference of the Early Textiles Study Group in Glasgow in 2019.

This thesis contributes to the characterization of hop fibres (Article 3) that were used for textiles in Scandinavia in past (Article 2).

2.4.1 Hop fibres (Article 3)

Hops (*Humulus lupulus L.*) is an ancient perennial climbing liana (*Figure 13*), native to the Northern hemisphere. Hops belongs to the Cannabis family (Cannabaceae), with several species of plants, i.e. hemp (*Cannabis sativa L.*) containing bast fibres within the phloem in the stems that have been used for textiles in past. An overview of some selected fibres and fibrous materials used for textiles and cultural heritage objects in past can be found in Appendix A.

The main use of hops is as a flavour for beer brewing. Archaeological finds suggest that this usage dates to at least the 6th century in Europe, however a clear evidence that the findings are *Humulus lupulus* date back to the ninth century AD [109, p.129]. Hops have also been used as a sleeping draught and for antibacterial purposes [110, p.263-273]. What is less known is that the fibres from hops have also been used for textile, see [110, p.255-256, 111, p.84-87, 112, p.130] for discussion of historical references to textile production of hops. The use of hops in historical textiles has been confirmed experimentally for the first time as part of this thesis work (Article 2, chapter 3.1). The main topic of the Article 3 was the development of suitable and reliable fibre identification method - the first necessary step for testing of historical objects.

Figure 13: Hop plant is a climbing liana that can reach up to 10 m. Only cone-shaped flowers – strobili – are used for beer brewing. The plant waste is enormous during beer production since the plant is cut close to roots yearly, © Lukesova.

Article (3) presents a combination of features of hop fibres that can distinguish them from the other main herbaceous bast fibres used for textiles in Europe in the past: hemp, flax, and nettle. The modified Herzog test gives a similar result for hemp- and hop fibres, which is Z-twist of microfibrillar orientation (as one would expect since they belong to the same family) but differs from flax and nettle that show an S-twist. A microchemical test of a swelling behaviour in an alkali solution cuoxam differs hops from hemp: hops show irregular undulation and remains of protoplasm sticking out of fibre's end, whereas hemp shows clear harmonica-like folding and strangulations. Besides, there are other characteristic morphological features of hop fibres as very long fibres, thick flat regions, frequent flexions, and undulated fibres. Crystals can be present.

In summary, the article presents a relatively easy and low-cost method to distinguish hops from other herbaceous bast fibres common in past. The method is micro-invasive and is suitable especially on relatively well-preserved materials. It requires transmitted light microscopy and can therefore not be applied to carbonized and mineralized fibres.

3. New fibre identifications carried out as part of this thesis work

Application of any fibre identification method on cultural heritage material may be challenging due to several factors as a general condition, brittleness, bad preservation of the crystalline structure, impurities, or even possible contamination that might happen during post-excavation treatments, conservation and/or storage. Cotton is one of the most common pollutants in the museum context [113, 114], however, adhesives and different consolidants may also be present [41, 115]. Caution should be taken during sampling so that only such a sample, which is representative for a research question, is extracted. Another important thing is a thorough consideration of ethical issues since cultural heritage material is irreplaceable and most of the fibre identification methods are micro-invasive. Current museum procedures often require an official application before sampling that is considered by a scientific committee. Documentation of sampling, the method used, results, as well as outcomes in form of publications, should be a part of an object's permanent record as stated by ICOM Code of Ethics for Museums 2004 [116].

3.1 Application of Hop Fibre Method (Article 2)

This article is a follow-up of article 3 on the development of a hop fibre identification method. In this article, the new identification method is applied to two Swedish cultural history objects: a woman's garment from the 19th century and a textile fragment from an 18th-century textile sample book, which was labelled as being made from hops.

Carl von Linné mentions in his "*Flora oeconomica*" the use of hops for textile production. He writes that if the hop stalks are retted, they can be used for yarn similar to hemp [117, p.60-61]. Reading this text was very inspiring and therefore written historical sources mentioning the use of hop fibres for textile production in Scandinavia from around 1600 up to the 19th century were studied [118, p.66-67, 119, p.10-20, 120, p.486]. It seems there was something like a movement to find out

an additional use for hop plant waste that is documented in the record of an experiment published by the Royal Swedish Academy of Sciences showing how to make material like “flax bast” from hops [121, p.214-216]. However, it is obvious the knowledge was spread in Scandinavia already before [118, p.66-67] and the question is if this knowledge is based on a longer tradition of material usage.

In this article, it was proven that the woman’s garment is made with hops and hemp fibres (namely the upper part with hop fibres and the bottom part, which is made of a different fabric is a fibre blend of hop- and hemp fibres). The textile fragment from the textile sample book is made with hops. This study provides the first direct proof on historical objects that hop fibres were used for textiles in the past. The results highlight the importance of careful material analysis of cultural heritage objects, leading to new knowledge regarding the understanding of resource management in the past.

3.2 Application of the Herzog test (Article 4)

The modified Herzog test is still one of the easiest and most reliable ways to distinguish different groups of plant fibre species from each other (the test is explained in chapter 2.2.2). However, as discussed in article 5 (Chapter 2.3.1), the test requires fibres that are not carbonized or mineralized. The inner structure must be well preserved for the test to work. This does not mean that the test does not apply to archaeological material as shown in this study. Basically, if interference colours are possible to distinguish in both orthogonal positions, the test results can be used. If the colours are inconclusive, the results may be enhanced with diluted NaOH as discussed in the article. It is not possible to estimate the extent of the degradation of the fibre’s inner structure based on the visual appearance. Sometimes, fragments that appears heavily degraded visually might still have a relatively well-preserved internal structure and vice versa.

Textile finds in the Late Iron Age Collection of the University Museum of Bergen were studied systematically. The finds come from western Norway mainly. A total of

45 grave finds with more than 100 different weaves was identified in the collection [122]. The burial conditions in Norway are mainly wet and acidic, which is very bad for the preservation of plant fibres.

It was possible to identify ten non-mineralized and non-carbonized finds with fragments of plant fibre material belonging most probably to clothing and accessories [122]. Fibres from these ten finds were investigated using the modified Herzog test. Besides, morphological features were observed carefully. Nine samples were identified as flax, one sample could only be identified as a bast fibre. This study shows that though hemp was used in some cases for fine textile production in Viking Age Scandinavia [123], available remains of plant fibre clothing and accessories coming from Hordaland, Sogn og Fjordane and Rogaland counties are all made of flax.

4. Conclusion and Outlook

The work presented in this thesis highlights the challenges of plant fibre analysis, in particular concerning archaeological and historic objects. The thesis covers several topics, connected through the goal of developing new methods customized to the identification of historical and archaeological fibres. The articles in this thesis can be divided into three groups: i) Two articles concerning the adaptation of plant fibre identification methods on historical and archaeological material and proving of their validity when used in this context, ii) One article on a less known species in the textile context and its characterisation in terms of fibre identification, iii) Two articles on the application of fibre identification methods using optical microscopy on historical and archaeological material.

In this chapter, the results are summarized, and further work is proposed.

4.1 Adaptation of methods on cultural heritage material

Methodologies regarding fibre identification of historical and archaeological fibres have often been derived from the field of textile industry and forensic science. However, it has been shown that fibre identification on cultural heritage is specific and should rather be understood as a sub-discipline, that requires adaptation of the methods on the unique and irreplaceable materials. The reason for this relies on several facts as follows. 1. Not only the species that have been used as commercial fibres were used for textile production throughout history. 2. Historical processing methods, that differs from the modern ones, may have an impact on the fibre's appearance, 3. The material of historical and archaeological objects is often degraded, which requires specific knowledge related to sampling and interpreting of results and limits the methods that can be used (i.e. carbonization prevents the use of transmission light microscopy). 4. Working with cultural heritage material rise ethical issues regarding the number and the size of core samples, which leads to limitations in terms of a possible number of sub-samples and use of statistical evaluation of data. This means that fibre identification of cultural heritage material should strictly

differentiate between characteristic and distinguishing features and the main emphasis in this sub-discipline should go towards further research on distinguishing features – ideally on historical reference samples or at least on artificially aged modern reference samples.

4.2 The need for characterisation of less known species

The material resources of ancient societies differ from the modern ones. This means that the species that were possibly used in the past needs to be pointed out and those that are less known and not yet characterised, need to be described thoroughly. The theoretical set of species that come into question when analysing a cultural heritage object determines the distinguishing diagram that can be used. This means that all possible species that are relevant for an identification process needs to be recorded before the analysis and the diagram used needs to be adapted based on the species that theoretically come into question.

4.3 Application of microscopic identification methods on cultural heritage material

Both light microscopy and SEM as well as other techniques, when necessary, should be used in conjunction, as each will have its advantages for particular circumstances. [30, p.322]. Needless to say, when required, other techniques should be applied as well. In this context, it is important to emphasize the need to do the identification work correctly. During this thesis work, several articles on the identification of ancient fibres were found where either wrong methods were applied or suitable methods were applied wrongly. It is clear, that more teaching material on how to perform fibre identification tests, should be made readily available for the textile archaeology and conservator community. This forms a very important part of future work. It is encouraging that the new COST action CA19131 Europe Through Textiles aiming to bridge current cultural, political and geographical gaps and facilitate interdisciplinary research (https://www.cost.eu/actions/CA19131/) has teaching as one of its priorities.

II Articles

Article 1

1.1 Is Cross-Section Shape a Distinct Feature in Plant Fibre Identification?

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IS CROSS-SECTION SHAPE A DISTINCT FEATURE IN PLANT FIBRE IDENTIFICATION?*

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Correct identification of textile fibres is an important issue in archaeology because the use of different materials can yield crucial information about the society that produced the textiles. Textiles made of plant and animal fibres can normally be easily distinguished, but to distinguish between different types of plant fibres, in particular different types of bast fibres, is difficult. Some years back it was shown that the features fibre diameter, lumen diameter, dislocation (nodes), and cross markings cannot be used on their own to distinguish between the typical bast fibres used for textiles in ancient Europe: flax, hemp, and nettle. Particularly not when only a few fibres are available for an examination so that statistical analysis is not possible, as is often the case in archaeology. The last two characterization features typically used to distinguish between bast fibres are cross-section shape and lumen shape. In this paper, we present a study of retted and unretted fibres (in the stem) of flax, nettle, and hemp, and show that also cross-section shape and lumen shape cannot be used as distinguishing features on their own.

KEYWORDS: PLANT FIBRE IDENTIFICATION, TEXTILES, ARCHAEOLOGY, FLAX, HEMP, NETTLE, CROSS-SECTION

INTRODUCTION

Archaeological evidence suggests that the first textiles were made of tree-bast and wild plant fibres, see for example (Barber 1991; Jørgensen 1992; Good 2001; Hurcombe 2010; Gleba and Mannering 2012), however, the actual use and choice of different textile plants throughout history based on archaeological textile finds has so far not been analysed systematically. The main problem has been that bast fibres (i.e. flax, nettle, and hemp), which were the most common textile fibres available in ancient Europe, look very similar. Unfortunately, there has been a tendency in the literature to identify plant fibres as flax on the sole basis of examinations with standard, white light, compound microscopy (Kvavadze *et al*. 2009; Bergfjord *et al*. 2010; Haugan and Holst 2014). A standard, white light microscopy examination looking at the long axis of fibre is sufficient to distinguish animal fibres and plant fibres (animal fibres have scales). However, as was shown, this is not enough to identify the plant fibre type (Bergfjord and Holst 2010; Bergfjord *et al*. 2012; Haugan and Holst 2014). We note that the same limitation applies to scanning electron microscopy. Thanks to its superior resolution and high depth of field, scanning electron microscopy can produce beautiful microscopy images of archaeological fibres, as demonstrated in a recent publication *Fibres: Microscopy of Archaeological Textiles and Furs* (Rast-

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Eicher 2016), but despite what is sometimes claimed, scanning electron microscopy cannot be used, on its own to identify specific bast fibre species. Wrong identifications will lead to a distorted picture of the relative importance of the various textile plants.

Fortunately, there exist other methods that can be applied in secure plant fibre determination: micro X-ray diffraction (Müller *et al*. 2006), identification of crystals in the associated tissue material of the fibres (Catling and Grayson 1982; Bergfjord and Holst 2010; Bergfjord *et al*. 2012), and the modified Herzog test (Herzog 1922, 1943, 1955; Petraco and Kubik 2004; Haugan and Holst 2013), as well as microchemical tests (Luniak 1953; Wülfert 1999). A recent paper demonstrated that reliable identification of the unusual textile fibre hop can be achieved with a combination of several of the techniques listed above (Lukešová *et al*. 2019). An earlier publication shows how nettle can, in some cases, be distinguished from hemp and flax using polarisation microscopy (Bergfjord and Holst 2010). Ongoing work is applying the use of polarisation microscopy in an attempt to identify plant fibre species outside Europe (Paterson *et al*. 2017; Smith *et al*. 2020). The application of combined tests on historical material can be challenging due to the degradation of fibres (Lukešová *et al*. 2017; Lukešová 2018). Unfortunately DNA analysis, which at first seems an obvious choice, has so far not proven to be a good method for archaeological plant fibres. Fibres contain very little DNA material and the retting process, which releases the bast fibres from the stem, promotes DNA degradation so that even modern fibres are difficult to identify with DNA analysis (Hofreiter *et al*. 2001; Dunbar and Murphy 2009).

Finally, it should be emphasized that when it comes down to proving the use of specific plants, that is, flax or hemp for textile production, the only, true evidence is well preserved textile finds, where a proper fibre identification can be performed. Textile imprints on ceramics and mineralized textile remains cannot be assigned to specific species, because such material estimations are simply unreliable.

The recent research on genetic diversity of flax (*Linum usitatissimum*) confirms its domestication around 10.000 years ago. There are strong indications that at first flax was mainly cultivated for the oil (Allaby *et al*. 2005, 63). Measuring the seed size of flax suggests the presence of different forms of flax for oil and for fibre production since at least the third millennium B.C. (Herbig and Maier 2011; Karg 2011). Early flax processing technology has been studied by several authors (Herbig and Maier 2011; Leuzinger and Rast-Eicher 2011; Maier and Schlichtherle 2011).

In Europe, one of the earliest and largest textile finds is from Late Neolithic lake settlements (4,200–2,800 cal. BC). It includes textiles made of flax (Rast-Eicher 1997; Körber-Grohne and Feldtkeller 1998; Rast-Eicher and Thijsse 2001). Hemp (*Cannabis sativa*) was known and used in the Neolithic period in the northern latitudes, from Europe to East Asia, but textile use of this plant has not been confirmed in the western Europe until the Iron Age (Barber 1991, 17–19). Fibres of the stinging nettle (*Urtica dioica*) are assumed to have been used since the Mesolithic (Hurcombe 2014, 55–57, 63). A direct proof was recently given through fibre identification of the 2,800-year-old Lusehøj Bronze Age Textile from Voldtofte, Denmark (Bergfjord *et al*. 2012).

AIMS AND OBJECTIVES

It has been suggested that standard, white light, compound microscopy may be sufficient to ensure identification if, instead of looking at the long axis of the fibre, fibre cross-sections are examined (Stratmann 1973, p. 108; Catling and Grayson 1982, p. 4). The characteristic features in the cross-section view are cross-section shape and lumen shape.

Table 1 presents an overview of what is considered the typical cross-section features for flax, hemp, and nettle as given in the literature:

Not all literature acknowledges cross-section features as suitable for identification. One author state that fibre cross-section shape of flax and hemp should not be used as a distinguishing feature (Herzog 1955, p. 319), and neither Petraco and Kubik (2004) nor Goodway (1987) refer to typical cross-section shapes of the plant fibres they have studied. Goodway even states that cross-sections of cells from different vegetable fibers tend to look very similar (Goodway 1987, p. 31). The same was the finding of Bergfjord and Holst who stated in their paper on how to distinguish nettle from flax and hemp that large cross-section variations can occur within individual species (Bergfjord and Holst 2010, p. 1192).

In this study we show that it is possible to find all different shapes of fiber cross-section listed above (polygonal, oval, rounded, flattened) in all studied species. It is quite possible that a specific cross-section shape is on average typical for a particular fibre, but growth conditions may alter shapes, and working with cultural heritage objects allows only very limited sample amounts. The risk of poor/wrong statistics is therefore high.

Fibres grow in a compact sclerenchyma layer in plant stems. Retting causes fiber release and swelling. Before starting this study, we speculated that the extraction of fibres from the compact layer through retting may lead to the polygonal shapes becoming rounder. We noticed that some authors comment on fibres in stems (Bodros and Baley, 2008, p. 2144; Catling and Grayson 1982, pp. 12–23; Herzog 1955, p. 319, 335, 345), while others comment on extracted fibres (Carr *et al*, 2008, p. 81; Luniak 1953, p. 109, 124–125; Suomela *et al*. 2017, p. 419; Wülfert 1999, pp. 274–278), which is a potential source of confusion. Therefore, we decided to compare cross-section shapes and lumina of both processed (retted) and unprocessed fibres (in the stem).

Another danger we want to highlight may happen during actual sample preparation. Comparing cross-section shape is only possible when the cross-section is examined perpendicular to the fibre's longitudinal axis. However, historical fibres are often deformed in both spin and weave directions. It may be very difficult to prepare perpendicular cross-sections of degraded fibres because they keep their shape of spin due to loss of flexibility. They also tend to break easily.

Flax is usually harvested for fibre production when the bases of the plants begin to turn from green to yellow (Tobler 1938, p. 31) and seeds begin to ripen (Cook, 1959, p. 7). However, it is described that the exact time of harvesting was dictated by the ultimate use of fibres—green stems were harvested for soft fibres for very fine textiles. For a stronger cloth, the stems were left until they became yellow (Gale and Cutler, 2000, p. 152). Based on these references, we decided to investigate both mature and immature flax (*Linum usitatissimum*) as well as mature hemp (*Cannabis sativa*) and mature stinging nettle (*Urtica dioica*).

EXPERIMENTAL

The first batch of flax plants (*Linum usitatissimum*) was harvested in the botanical garden of the University Museum of Bergen in their immature state when the plants were completely green and started to blossom. A second batch was harvested six weeks later in its mature state when the stems started to be yellow in the lower part and the seeds began to ripen. Hemp (*Cannabis sativa*) was obtained from the botanical garden of the Natural History Museum in Oslo. We investigated a stem from a female individual in its mature state. Wild stinging nettle (*Urtica dioica*) was harvested in Bergen in a mature state.

Table 1 *Overview of the typical cross-section features for flax, hemp and nettle as given in the literature*

Authors	*Flax Fibre cross-section shape*	*Flax Lumen cross-section shape*	*Hemp Fibre cross-section shape*	*Hemp Lumen cross-section shape*	*Nettle Fibre cross-section shape*	*Nettle Lumen cross-section shape*
Bergfjord and Holst 2010: 1192	Rounded Polygonal	Narrow, round Oval	Rounded Polygonal	Narrow, round Oval	Elongated Band formed	Larger
Bodros and Baley 2008: 2144 (fibres in stems)	-	-	-	-	Polygonal	Channel-like
Carr *et al.* 2008: 81 (ultimate fibres)	Polygonal (3–5 sides), thick cell wall	Small lumen	Polygonal (4–6 sides), cell wall thickness similar to flax	Larger lumen than flax	-	-
Catling and Grayson 1982: 12–23 (fibres in stems)	Pentagonal or hexagonal Rounded in outline	Both narrow and wide (50:50)	Angular with 4, 5 or 6 sides Oval Round	Small Round Elongated Irregular	-	-
Herzog 1955: 319, 335, 345 (fibres in stems)	Flattened Irregular oval Star-shaped Polygonal Rounded Irregular	Line-shaped Wide Elliptic	Flattened oval Polygonal	-	Flattened oval Kidney-shaped Round Band-like	-
Isenberg 1967: 166	Polygonal	Slit-like	Rounded edges	Broad, becomes like a line towards the end of fibre	-	-
Luniak 1953: 109, 124–125 (fibre bundles)	Mainly sharply polygonal Oblong with rounded corners	Mainly narrow round Oval Larger forms	Similar to flax	Similar to flax, often as a mere line and indistinct	-	-
Suomela *et al.* 2017: 419 (fibre bundles)	Polygonal	Round and small	-	-	Oval	Flat and long
Wülfert 1999: 274–278 (fibre bundles)	Regularly polygonal	Mature – thin Immature – thick	Rounded	Not that thin as flax	Irregular oval Flattened	Rather wide with remains of protoplasm

Preparation of cross sections

The first series of cross-sections were prepared from the middle part of the plant stems of flax (*Linum usitatissimum*), hemp (*Cannabis sativa*), and nettle (*Urtica dioica*). Sections of stems were mounted in epoxy resin EpoFix (n_D =1.571), cut with a diamond saw (Buehler IsoMet low-speed precision cutting machine), ground, and polished. Nikon compound microscope Eclipse Ci-POL equipped with CFI TU Plan Fluor EPI P objectives series was used for measurements.

The second series of cross-sections were prepared from retted and combed reference material of flax (*Linum usitatissimum*), hemp (*Cannabis sativa*), and nettle (*Urtica dioica*). The raw material was neither bleached nor spun. Flax and hemp were obtained from the company HempFlax AB from the Netherlands, nettle was obtained from the company NFC GmbH from Germany. We are aware of numerous convarieties/varieties of the described species. However, the identification of textile fibres by means of microscopy does not go below the level of species and the common praxis is to define the reference samples as it is done here.

Cross-section plates, silk embedding fibres, and a razor blade were used for preparing samples. Glycerin (n_D =1.474) was used at the top of the sample as a mounting medium. Leica compound microscope Ortholux II POL-BK equipped with NPL FLuotar P objectives series was used for measurements.

RESULTS

Here we present fibre cross-section shapes and lumen cross-section shapes that we identified in following species flax (*Linum usitatissimum*), hemp (*Cannabis sativa*), and stinging nettle (*Urtica dioica* with unprocessed fibres (fibres in the stem) and processed (retted) fibres. For flax, unprocessed stem fibres are presented for both mature and immature plants.

The results of our measurements are summarized in tables 2 and 3. As can be seen, it is possible to find all identified shapes of cross-sections from table 1 in all studied fibres both in unprocessed and processed fibres. These are polygonal, polygonal slightly rounded, oval, irregular oval, uneven with rounded edges, and flattened. Lumina found in all studied species can be narrow round, oval, or irregular oval. They can also be wide of larger forms, slit-like, indistinct, and flattened.

Table 2 *Cross-section shapes of immature and mature flax, hemp, and nettle fibres*

	Shape of fibre cross-section	*Flax immature/ stem*	*Flax mature/ stem*	*Flax mature/ retted*	*Hemp/ stem*	*Hemp/ retted*	*Nettle/ stem*	*Nettle/ retted*
1	Polygonal	x	x	x	x	x	x	x
2	Polygonal slightly rounded	x	x	x	x	x	x	x
3	Oval	x	x	x	x	x	x	x
4	Irregular oval	x	x	x	x	x	x	x
5	Uneven, rounded edges	x	x	x	x	x	x	x
6	Flattened	x	x	x	x	x	x	x

Table 3 *Lumen shapes of immature and mature flax, hemp, and nettle fibres. The number and letters refer to the microscopy images below where the features are displayed*

	Shape of lumen cross-section	*Flax immature/ stem*	*Flax mature/ stem*	*Flax mature/ retted*	*Hemp/ stem*	*Hemp/ retted*	*Nettle/ stem*	*Nettle/ retted*
A	Narrow round	x	x	x	x	x	x	x
B	Oval	x	x	x	x	x	x	x
C	Irregular oval	x	x	x	x	x	x	x
D	Larger forms, wide	x	x	x	x	x	x	x
E	Slit like	x	x	x	x	x	x	x
F	Indistinct	x	x	x	x	x	x	x
G	Flattened/elongated	x	x	x	x	x	x	x

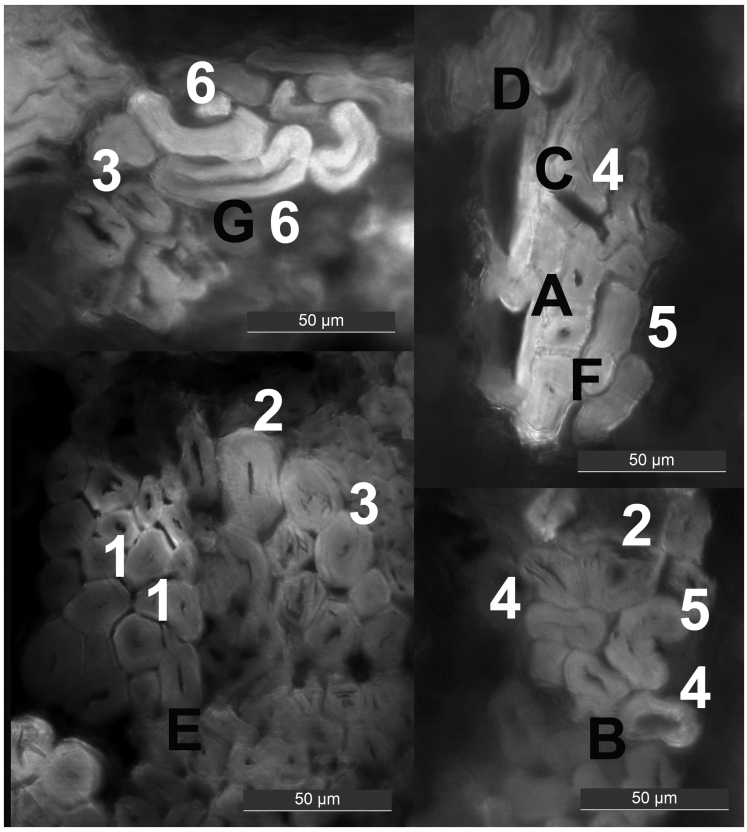

FIGURE 1 *Transmitted light micrograph of retted flax fibres. Numbers refer to the defined cross-section shapes from table 2. Polygonal, oval, flattened as well as all other forms are present. Letters refer to the defined shapes of lumina from table 3. Lumina are mostly narrow round, slit-like, and flattened, but all other forms are also present.*

Retted flax fibres show polygonal, rounded polygonal, oval, flattened, as well as all other forms (Fig. 1: cross-section shapes: 1, 2, 3 and 6). Their lumina are mostly narrow round, slit-like, and flattened, but all other forms are also present (Fig. 1: lumina A, E, G). Retted hemp fibres show polygonal, polygonal rounded, oval, flattened, as well as all other forms (Fig. 2: cross-section shapes: 1, 2, 3 and 6). Their lumina are narrow round but also of wider forms. Slit-like and flattened forms are present, as well as all other forms (Fig. 2: lumina A, D, E, and G). Retted nettle fibres mainly show oval and flattened forms; however, all other shapes are present as well (Fig. 3: cross-section shape 3 and 6). Lumina are often slit-like and flattened, but other forms are also present (Fig. 3: lumina E and G).

Fibres in mature flax and hemp stem typically show rounded polygonal outer shape with a narrow, round, or oval lumen. Fibres in immature flax and nettle stem mostly have a more flattened outer shape with a larger lumen. However, polygonal shapes can show up in nettle and flattened shapes are not unusual in mature flax and hemp. Figures of fibres in stem cross-sections are in supplementary document.

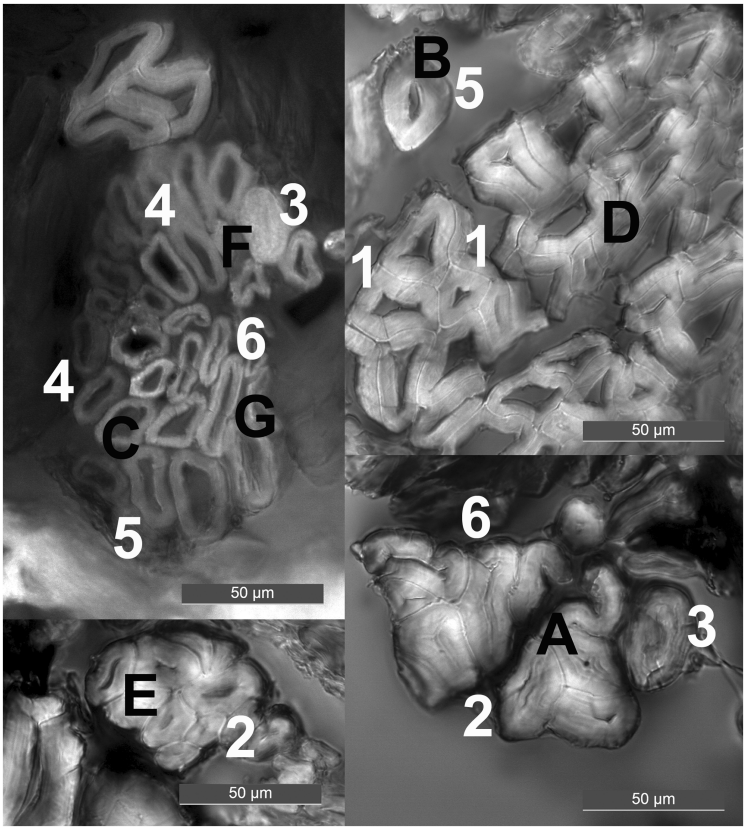

FIGURE 2 *Transmitted light micrograph of retted hemp fibres. Numbers refer to the defined cross-section shapes from table 2. Polygonal, oval, flattened as well as all other forms are present. Letters refer to the defined shapes of lumina from table 3. Lumina are often of wider forms, slit-like, and flattened are present, as well as all other forms.*

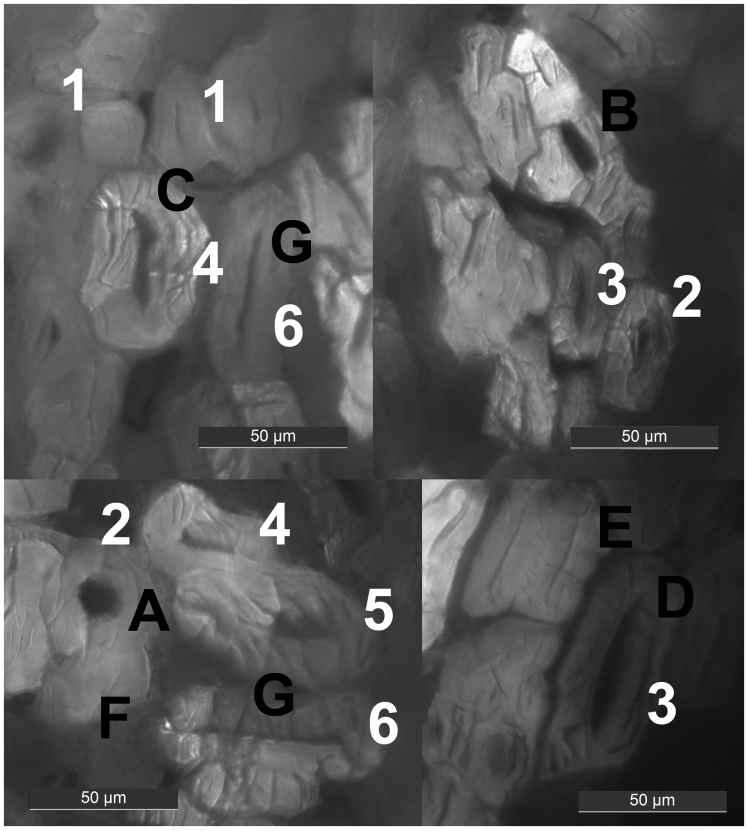

FIGURE 3 *Transmitted light micrograph of retted nettle fibres. Numbers refer to the defined cross-section shapes from table 2. Oval and flattened forms are common; however, all other forms are present as well. Letters refer to the defined shapes of lumina from table 3. Lumina are often slit-like and flattened, but other forms are also present.*

Our study shows that even cross-section shapes of unprocessed stems (that cannot be disturbed by processing or deformed due to spinning and/or weaving) cannot be used as a reliable feature for distinguishing species.

DISCUSSION

We have conducted a comparative study of fibre cross-section shapes and lumen cross-section shapes of three plant species (flax, hemp and nettle), and we summarize that the two criteria are inconclusive for identification on their own because fibres with non-characteristic shapes can be found for all species. We note that looking at the different images one does tend to recognize an overweight of fibres with what may be considered characteristic features, such as the rounded polygonal shape with a small lumen for mature flax and hemp, and flattened shape with a slit-like and flattened lumen for unmatured flax and nettle. However, the point we want to emphasize is that with only a very limited sample material that does not allow for a proper statistical analysis, it is very difficult to conclude. Also, for archaeological samples, nothing can be known

about the growth conditions, and as we see, the very small lumen is characteristic for mature flax only.

CONCLUSION

In this study, we show that all identified shapes of fibre cross-sections and their lumina in flax (*Linum usitatissimum*), hemp (*Cannabis sativa*), and stinging nettle (*Urtica dioica*), can be found in all three species. Flax was examined in two stages of ripeness: immature and mature because both were used for textile production. All identified shapes were found both in unprocessed and processed fibres. Mature flax and hemp typically show rounded polygonal outer shape with a narrow, round, or oval lumen. Immature flax and nettle mostly have a more flattened outer shape with a larger lumen. However, polygonal shapes can show up in nettle and flattened shapes are not unusual in flax and hemp.

We conclude that cross-section shape and lumen shape cannot be used on their own as a distinct feature for plant fibre identification. Proper identification is only possible by the combination of several methods, as highlighted in the introduction, and even then, secure identification cannot always be ensured. Precise knowledge of material use in cultural heritage collections is important for understanding resource management in the past. Hence, it is important to keep searching for new ways to identify plant fibre species in historical objects.

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SUPPORTING INFORMATION

Additional supporting information may be found online in the Supporting Information section at the end of the article.

Table S1: Cross-section shapes of immature and mature flax, hemp, and nettle fibres.
Table S2: Lumen shapes of immature and mature flax, hemp, and nettle fibres.
Figure S1: Reflected light micrograph of immature flax stem: the numbers refer to table 1. Oval and flattened shapes remind very much nettle fibres, polygonal- and polygonal, slightly rounded shapes are present as well as other types of shapes.
Figure S2: Reflected light micrograph of immature flax stem. The letters refer to table 2. Lumina are slit-like, flattened of wider forms, but all other forms are also present.
Figure S3: Reflected light micrograph of mature flax stem. The numbers refer to table 1. Polygonal- and polygonal slightly rounded cross section shapes can be observed, but oval and uneven shapes as well as all other forms are present.
Figure S4: Reflected light micrograph of mature flax stem: The letters refer to table 2. Narrow round, slit-like; oval and wide lumina are common but all other shapes are also present.
Figure S5: Reflected light micrograph of hemp stem. The numbers refer to table 1. Polygonal rounded shapes and oval cross-section shapes are common. All other shapes are present as well.
Figure S6: Reflected light micrograph of hemp stem: The letters refer to table 2. Narrow round lumina, oval and slit-like lumina are present as well as all other shapes.
Figure S7: Reflected light micrograph of nettle stem: The numbers refer to able 1. Oval and flattened cross-section shapes are very common, but polygonal and polygonal slightly rounded come often for as well as other shapes.
Figure S8: Reflected light micrograph of nettle stem: The letters refer to table 2. Lumina are mostly large and wide, slit-like, and flattened lumina show up as well as all other shapes.

Supplementary

	Shape of fibre cross-section	Flax immature/ stem	Flax mature/ stem	Flax mature/ retted	Hemp/ stem	Hemp/ retted	Nettle/ stem	Nettle/ retted
1	Polygonal	x	x	x	x	x	x	x
2	Polygonal slightly rounded	x	x	x	x	x	x	x
3	Oval	x	x	x	x	x	x	x
4	Irregular oval	x	x	x	x	x	x	x
5	Uneven, rounded edges	x	x	x	x	x	x	x
6	Flattened	x	x	x	x	x	x	x

Table 1/ supplementary: Cross-section shapes of immature and mature flax, hemp and nettle fibres.

	Shape of lumen cross-section	Flax immature/ stem	Flax mature/ stem	Flax mature/ retted	Hemp/ stem	Hemp/ retted	Nettle/ stem	Nettle/ retted
A	Narrow round	x	x	x	x	x	x	x
B	Oval	x	x	x	x	x	x	x
C	Irregular oval	x	x	x	x	x	x	x
D	Larger forms, wide	x	x	x	x	x	x	x
E	Slit-like	x	x	x	x	x	x	x
F	Indistinct	x	x	x	x	x	x	x
G	Flattened/elongated	x	x	x	x	x	x	x

Table 2/ supplementary: Lumen shapes of immature and mature flax, hemp and nettle fibres.

Fibres in mature flax- and hemp stem typically show rounded polygonal outer shape (Fig. 3 and Fig. 5: cross-section shape 2) with a narrow, round or oval lumen (Fig. 4 and Fig. 6: lumen A and B). Fibres in immature flax and nettle stem mostly have a more flattened outer shape (Fig. 1 and Fig. 7: cross-section shape 6) with a larger lumen (Fig. 2 and Fig. 8: lumen D, E, G). However, polygonal shapes can show up in nettle (Fig. 7: cross-section shape 1) and flattened shapes are not unusual in mature flax and hemp (Fig. 3 and Fig. 5: cross-section shape 6).

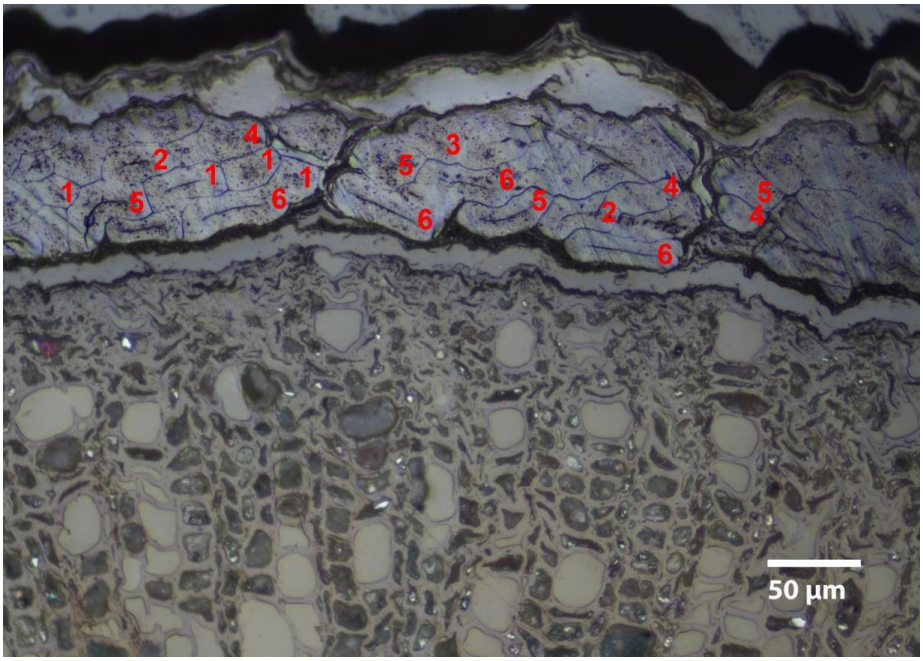

Figure 1/ supplementary: Reflected light micrograph of immature flax stem: the numbers refer to Table 1. Oval and flattened shapes remind very much nettle fibres, polygonal- and polygonal, slightly rounded shapes are present as well as other types of shapes.

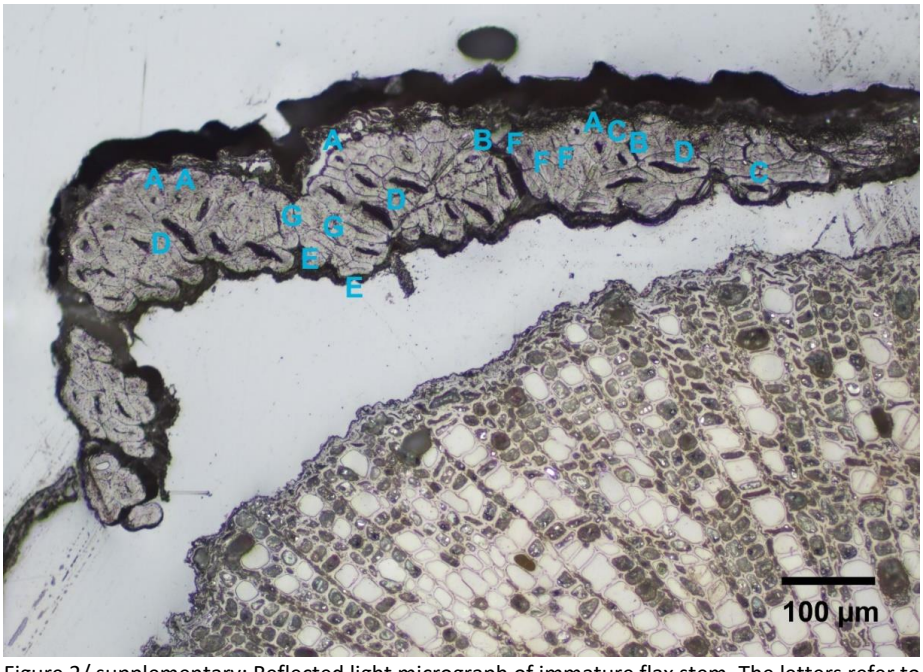

Figure 2/ supplementary: Reflected light micrograph of immature flax stem. The letters refer to Table 2. Lumina are slit-like, flattened of wider forms, but all other forms are also present.

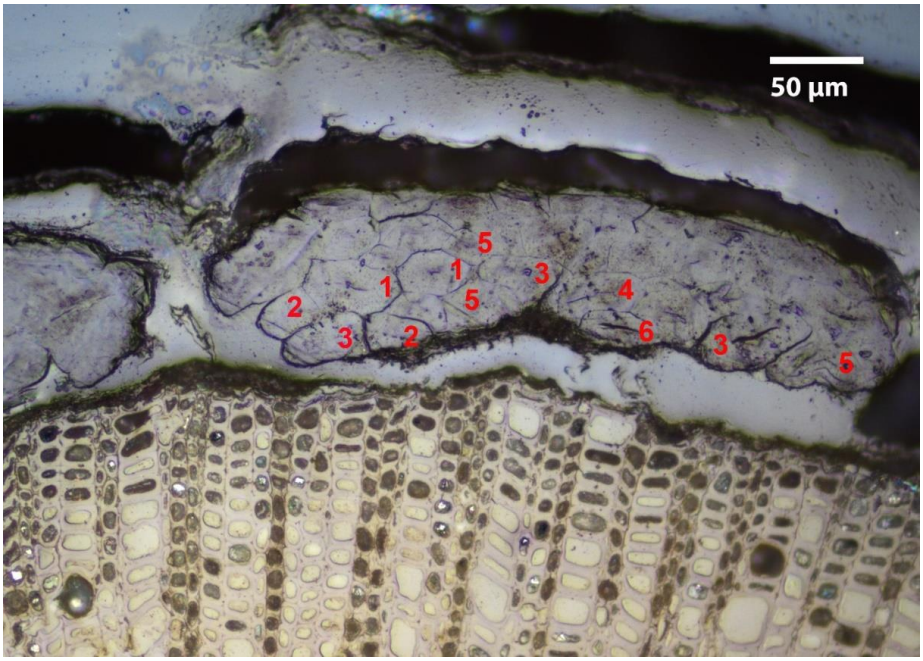

Figure 3/ supplementary: Reflected light micrograph of mature flax stem. The numbers refer to Table 1. Polygonal- and polygonal, slightly rounded cross section shapes can be observed, but oval and uneven shapes as well as all other forms are present.

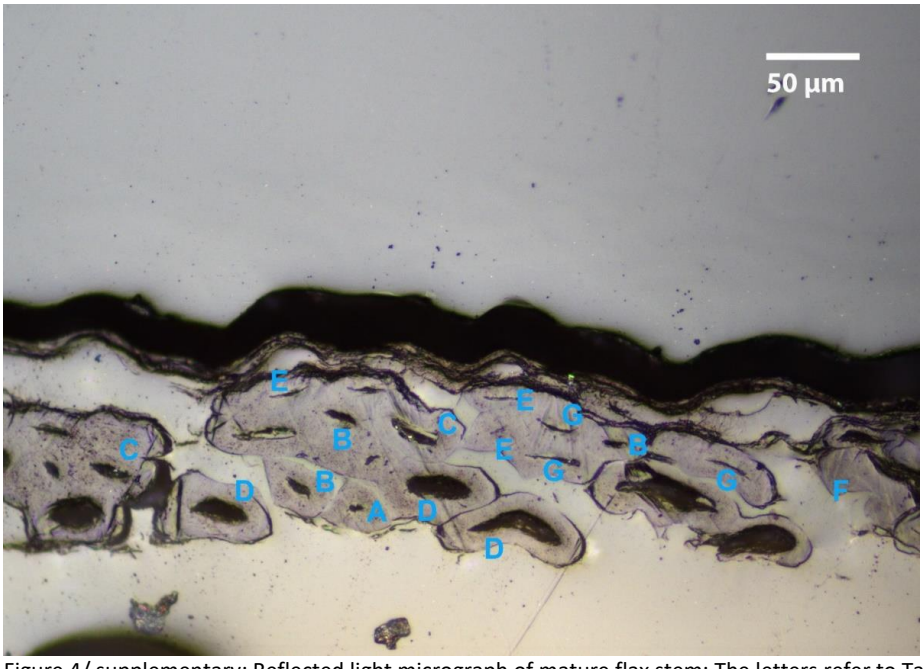

Figure 4/ supplementary: Reflected light micrograph of mature flax stem: The letters refer to Table 2. Narrow round, slit-like; oval and wide lumina are common but all other shapes are also present.

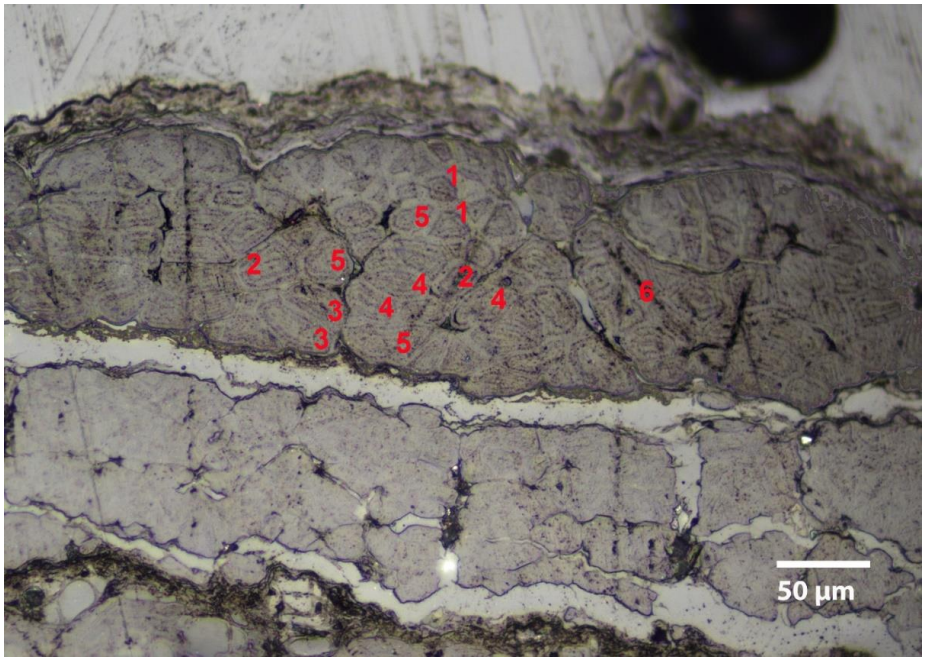

Figure 5/ supplementary: Reflected light micrograph of hemp stem. The numbers refer to Table 1. Polygonal rounded shapes and oval cross-section shapes are common. All other shapes are present as well.

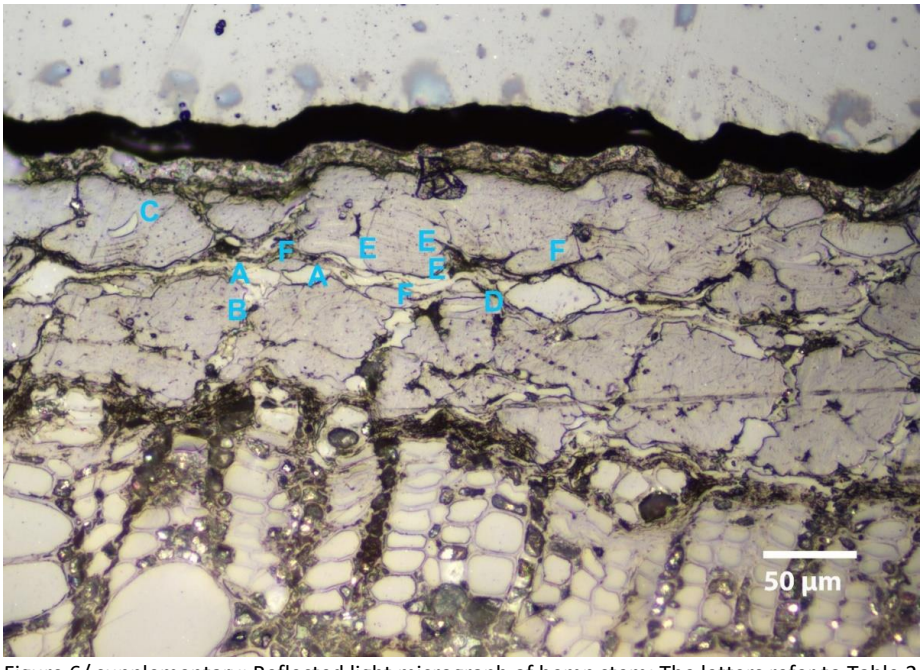

Figure 6/ supplementary: Reflected light micrograph of hemp stem: The letters refer to Table 2. Narrow round lumina, oval and slit-like lumina are present as well as all other shapes.

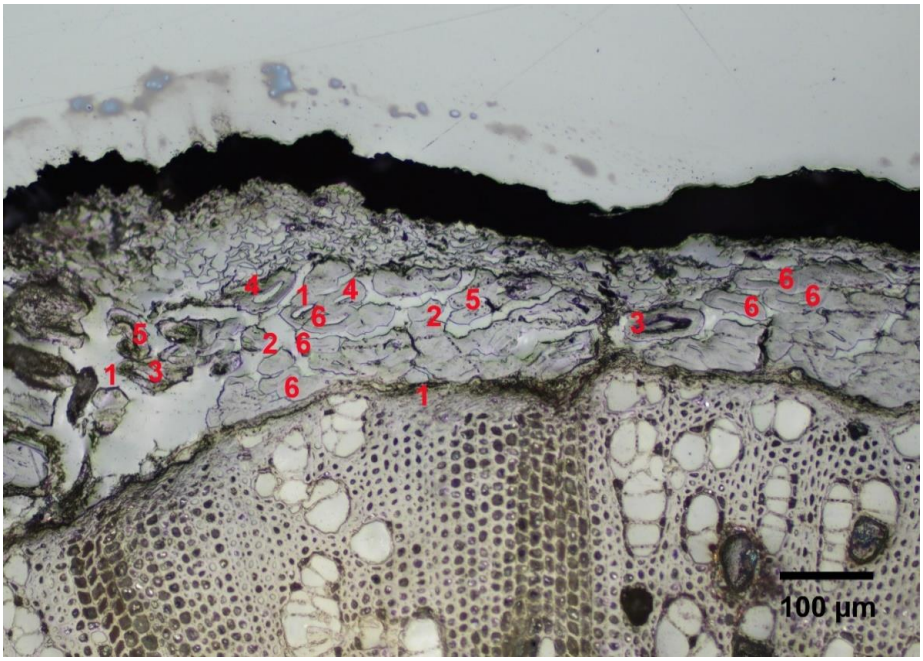

Figure 7/ supplementary: Reflected light micrograph of nettle stem: The numbers refer to table 1. Oval and flattened cross-section shapes are very common, but polygonal and polygonal slightly rounded come often for as well as other shapes.

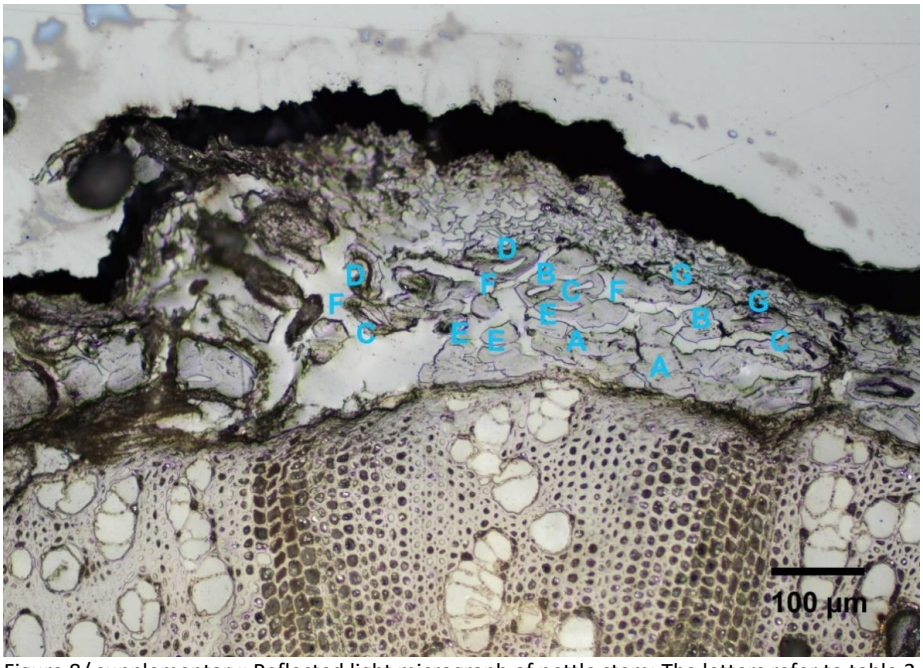

Figure 8/ supplementary: Reflected light micrograph of nettle stem: The letters refer to table 2. Lumina are mostly large and wide, slit-like and flattened lumina show up as well as all other shapes.

Article 2

First experimental evidence of hop fibres in historical textiles

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ORIGINAL PAPER

First experimental evidence of hop fibres in historical textiles

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Abstract
Hop (*Humulus lupulus*) has been used in Scandinavia since at least the ninth century AD, as documented through archaeological findings and written, historical records. The written records are mainly focused on the use of cone-shaped flowers for beer brewing and medical purposes, but there are also records, for example, from the famous Swedish botanist Carl von Linne, who mentions the use of hop fibres for textile production. However, until now no experimental investigations have been published on the use of hop fibres in cultural heritage objects. A major reason for this has been the lack of a suitable characterization method. Hop is a bast fibre, just as flax and hemp and bast fibres cannot be distinguished from each other by simple optical inspection. Recently a new identification method for hop fibres was published by the authors of this article. Here we apply the new method in an investigation of two Swedish cultural heritage objects: (i) a woman's garment from the nineteenth century, which was labelled as having an upper section made from coarse linen and a bottom section made of hemp and hop and (ii) a textile fragment from an eighteenth-century textile sample book, which was labelled as being made from hop. We show that the woman's garment is made with hop and hemp fibres and the textile fragment from the textile sample book is made with hop. Our work provides the first direct proof that hop fibres were used for textiles in the past.

Keywords Fibre identification · Hop · *Humulus lupulus* · Historical textiles · Herzog test · Cuoxam

Introduction

Hop (*Humulus lupulus*) is an ancient perennial crop plant, native to the Northern hemisphere. The oldest cultivated archaeological findings from Scandinavia, where it is clear that the findings are hop, are macrofossils from Birka, dating back to the ninth century AD (Hansson 1996, 129). Hop is frequently mentioned in historical, written records. The main emphasis is on the use of hop flowers for beer brewing, but other applications are also mentioned: hop flowers were applied for medical purposes (i.e. sleeping draughts) and for embalming and placed in burial coffins, for example, as filling in pillow cases (Strese and Tollin 2015, 263–273).

One of the oldest parts of the Frostathing law (*Frostatingsloven*), coming from the twelfth century, mentions cultivation of hop Book XIII, no. 11 (Hagland and Sandnes 1994, 93). In the Middle Ages in Norway, it was a duty for all farm owners to cultivate a certain amount of hop plants (Høeg 1976, 385). The same was the case in Sweden, where hop growing was obligatory from 1414 until 1860 (Karlsson Strese et al. 2014). On the other hand, records of cases of restrictions, where hop production was forbidden within certain areas and time periods, can also be found (Lankester 1840, 68).

It is documented through written records that hop fibres extracted from the plant stem were used for textile production in Scandinavia from around 1600 up to the nineteenth century (Bromelio 1687, 66–67; Schissler 1750, 214–216; Hald 1980, 130; Strese and Tollin 2015, 255–256). Carl von Linne mentions in his *Flora oeconomica* the use of hop for textile production. He writes that if the hop stalks are retted, they can be used for yarn similar to hemp (Linné and Aspelin 1749, 60–61). In 1773, the Norwegian topographer Gerald Shoning describes a travel to Surnadal (Norway). He mentions that in 1758, hemp, hop and also linen goods were imported to Trondheim (Schøning 1778, 10). He also states that flax, wool, hemp and hop were used to make fabrics (Schøning

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1778, 20). In 1781, Fischerström (1781) comments that hop stalks are normally thrown away but that one ought to do as in Jamtland and Medelpad, where they are used to make a weave, which is stronger than flax and hemp. (Fischerström 1781, 486). A fairly recent source mentions that hop fibre quality can vary a lot (Tobler 1938, 84–87). Experiments with substitute materials for textiles were also referred by Freund (1972, 7).

The most widely used textile plants in Scandinavia until the beginning of the twentieth century were flax (*Linum usitatissimum*) and hemp (*Cannabis sativa*). Hemp was used for cordage and coarse textiles, but there are also examples of the use of hemp for finer fabrics (Skoglund et al. 2013; Skoglund 2016). A few cases are documented, where stinging nettle (*Urtica dioica*) has been used for textile production (Hald 1942, 29–49; Bergfjord et al. 2012). Hop fibres were most likely not a very commonly used material compared with other textile materials.

Flax, nettle, hemp and hop are all bast fibres and it is not possible to distinguish them by simple optical inspection (Bergfjord and Holst 2010; Bergfjord et al. 2010; Haugan and Holst 2014). This may well have led to some textiles being incorrectly labelled as made of flax in various museum collections. It should also be noted that during the eighteenth century in Scandinavia, the term *linne* (Swedish) and *lin* (Norwegian) became common as a term to describe a plain weave textile irrespective of what it was made of. Earlier, a plain weave textile was often referred to as *lærred or lärft* in Swedish (Geijer 1979, 17). The terms *linne and lin* are however also used specifically for textiles made from flax. The modern word for flax is *lin* in both Swedish and Norwegian. The difficulties in terminology concerning linen also apply to the German *Leinwand* (Küster-Heise and Mitschke 2011, 159).

In order to find out what plants have been used to produce historical textiles, systematic investigations of objects in cultural heritage collections using the appropriate identification methods are necessary (see, e.g. Lukešová et al. 2017). Precise knowledge of material use in cultural heritage collections is important because it will enable better understanding of resource management in the past.

In this article, we present the first investigation on cultural heritage objects performed with the specific aim of finding out if they are made of hop; we use a very recently developed identification method (Lukešová et al. 2019).

The samples investigated

We investigate two historical objects in this article: the first object is a woman's garment from Jamtland County in Sweden (NM.0131474, left), belonging to the Nordic Museum in Stockholm (Fig. 1). It was donated to the museum in 1917. According to the museum accession record, the donor stated that the upper section is made of coarse linen fabric and the bottom section of hemp and hop (Redogörelse för Nordiska museets utveckling och förvaltning år 1919, p.11). The garment was probably produced in the middle of the nineteenth century. It is written in accession record that it was around 65 years old when it was donated to the Nordic Museum (https://digitaltmuseum.se/011023635901/overdelssark, downloaded 28.4.2020).

The upper section is made of twill fabric, which is rather greyish and soft in its appearance compared with the bottom section which is made of coarse tabby with a yellow tinge. The object is described in Skoglund (2016). It is stated here that a Herzog test fibre analysis suggests an upper section made with a mixture of flax and hemp and/or hop and a bottom section made with hemp and/or hop. No further details to the analysis are presented (i.e. regarding thickness of the fibres investigated).

The second object is a textile fragment glued onto a sheet of paper in a Swedish textile sample book (NM.0405398+) from 1766 (Fig. 1, right). The book presents a sample collection of textiles produced in the eighteenth century. The purpose of assembling the collection was to inspire an increase in Swedish textile production.

Methods, including sample preparation

Samples of both objects—the woman's garment from Jamtland and the textile fragment from the textile sample book—were carefully extracted and investigated by white light transmission and polarized light microscopy. In addition, microchemical tests using cuoxam-tetraamminediaquacopper dihydroxide $[Cu(NH_3)_4(H_2O)_2](OH)_2$ were performed at the end of investigation, in order to investigate the swelling behaviour (Luniak 1953, 80; Wülfert 1999, 281–282, 320; Stratmann 1973, 58–62). The investigations were performed following the recently developed identification method for hop fibres (Lukešová et al. 2019). See also Fig. 2.

For the Jamtland garment, four core samples were extracted since it was made of two different fabrics: two samples of the weft and warp system from the upper section (samples 1 and 2) and two samples of the weft and warp system from the bottom section (samples 3 and 4). For the textile fragment from the textile sample book, we only sampled the thread system of the shorter side (sample 5); it is so small. Both thread systems (warp and weft) show similar thread thickness, spin direction and colour when observed by stereomicroscope. We carefully evaluated ethical issues when sampling and concluded we perform the tests on one thread system only.

Five sub-samples (consisting of single fibres) were made from each core sample. Two of them were mounted in Meltmount (n_D = 1662) (labelled samples 1.1, 1.2, 2.1, 2.2,

Fig.1 (Left) The female upper garment from Jamtland county in Sweden (NM.0131474), 89 × 130 cm; (right) the fabric sample in a Swedish fabric sample book from 1766 (NM.0405398+), the lower sample was investigated (© The Nordic Museum in Stockholm)

3.1, 3.2, 4.1, 4.2, 5.1 and 5.2). The three remaining sub-samples from each core sample were mounted in distilled water according to an established protocol (Wülfert 1999, 325). These sub-samples (labelled sample 1.3, 1.4, 1.5, 2.3, 2.4, 2.5, 3.3, 3.4, 3.5, 4.3, 4.4, 4.5, 5.3, 5.4 and 5.5) were subsequently used for microchemical tests in cuoxam. Sample preparation was done using a stereomicroscope to be able to separate fibre bundles. Very fine tweezers and tungsten needles were used when manipulating single fibres; for a detailed description of fibre sample handling and mounting, see Lukešová (2018).

The samples were investigated using a polarized light microscope Leica DM750 P. A full wave compensator (λ = 530 nm) was used for the modified Herzog test (Herzog 1922, 1943; Haugan and Holst 2013). Photographs were taken using the camera Leica MC170 HD and software LAS V4,9. Fibres

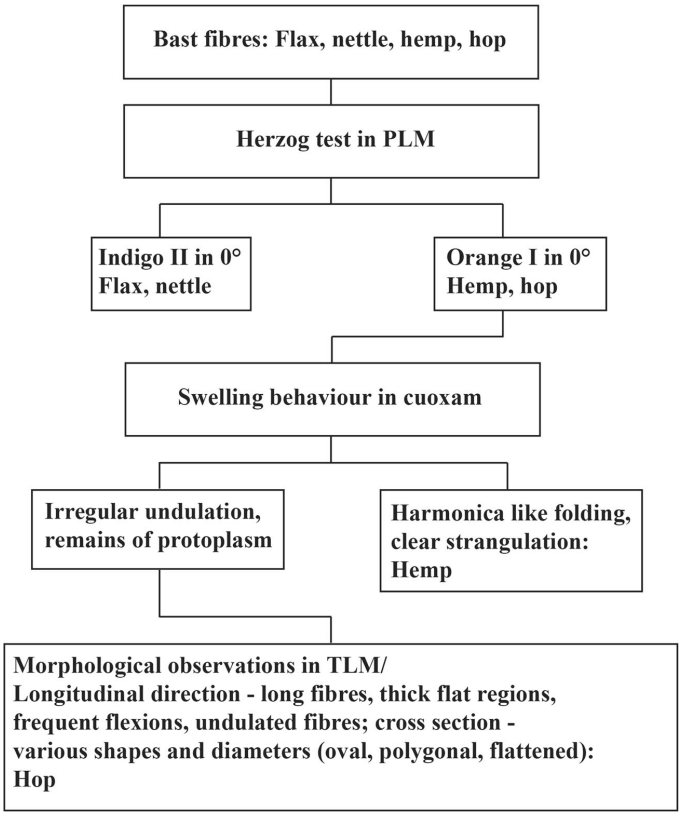

Fig. 2 Diagram for identification of hop fibres, reproduced from Lukešová et al. (2019), Herzog test, modified Herzog test; PLM, polarized light microscopy; TLM, transmission light microscopy

were first observed in transmitted white light. Polarized light was used for performing the modified Herzog test. Fibres thinner than 20 μm were not used for the Herzog test since an experience with reference samples has shown that they may give misleading results. A demonstration video on how to perform the Herzog test can be found on https://youtu.be/sC9GlUKjBDE.

Results

We followed the diagram elaborated for the identification of hop fibres shown in Fig. 2.

Polarization microscopy and the modified Herzog test

All samples except sample 1.5, which had no suitable region for testing, show Orange I in 0° and Indigo II in 90° position according to Michel-Levy birefringence chart when performing the modified Herzog test (Fig. 3).

Numerous crystals, probably calcium oxalates or other phytoliths, were clearly visible in all sub-samples except sample 1.4 (this is not used as an identification criterion).

Microchemical tests using cuoxam

Cuoxam, also called Schweizer's reagent, is an established tool for fibre identification since it causes swelling typical for species. All tested sub-samples show irregular undulation when swelled in cuoxam (Fig. 4, upper left and right) which together with the Herzog test result indicates hop. Samples 3.5; 4.3 and 4.4 show in addition harmonica-like folding of the middle lamella on some fibres, which indicates hemp (Fig. 4, lower left and right).

All sub-samples except 3.5 show clearly visible remains of protoplasm in the lumen. Sub-samples 1.3; 1.5; 2.4; 3.4; 4.3 and 5.4 showed a typical rounded edge of a fibre with a plasma thread sticking out (Fig. 4, upper right).

White light microscopy

All samples show strong, irregular thickness variations along the fibre lengths. This is one of the most characteristic features for hop (Fig. 5, lower left). There are wide flattened regions without cross marks that are even and smooth (Fig. 5, upper left and right). These often alternate with regions containing frequent cross marks and dislocations. All original samples show frequent flexions (Fig. 5, lower right). Undulated fibres (many twist flexion after each other) that might remind one of cotton fibres are also common.

We conclude that the upper section of the woman's garment NM.0131474 is made with hop (*Humulus lupulus*) and the bottom part is made with a fibre blend of hop and hemp (*Humulus lupulus* and *Cannabis sativa*). The textile fragment from the textile sample book (NM.0405398+) is made with hop (*Humulus lupulus*)—only one of the thread systems was investigated, because of the limited amount of original material.

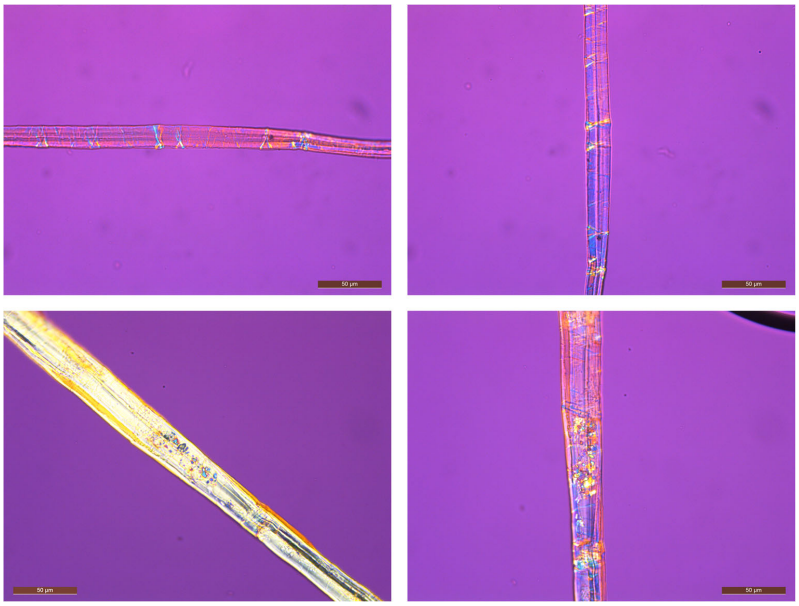

Fig. 3 (Upper left and upper right) Sample 1.2 showing Orange I in 0° and Indigo II in 90° position; (lower left and lower right) sample 3.1 showing numerous small crystals, probably calcium oxalates or other phytoliths, which are visible as small areas with pronounced, strongly varying interference colours (the objective HI PLAN POL × 40/0,65 used for all four figures)

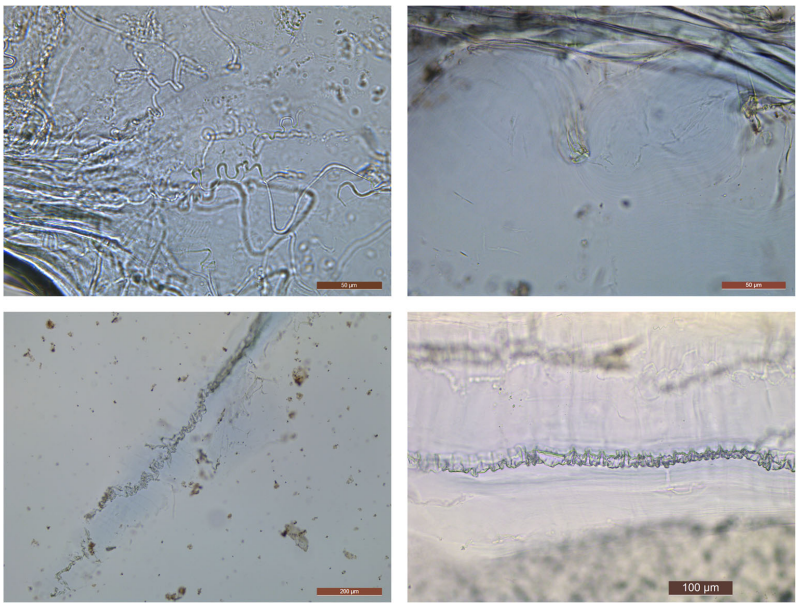

Fig. 4 (Upper left) Sample 4.3 in cuoxam showing ribbon-like pattern typical for hop fibres; (upper right) sample 1.3 showing plasma thread sticking out of rounded edge of a fibre, which is typical for hop fibres (the objective HI PLAN POL × 40/0,65 used for both figures). (Lower left) Sample 4.3 showing harmonica-like folding of cell wall in cuoxam typical for hemp (the objective HI PLAN POL × 10/0,25 used); (lower right) hemp reference fibre: typical harmonica-like folding in cuoxam (the objective HI PLAN POL × 20/0,40 used)

Discussion

We have conducted a fibre identification analysis of two historical objects: a woman's garment (NM.0131474) and a textile fragment from a textile sample book (NM.0405398+). Based on the behaviour of fibre samples from the two objects in polarized light, characteristic swelling in cuoxam and distinctive fibre morphology using the identification method (Lukešová et al. 2019), we conclude that for the garment NM.0131474, the upper part is made with hop fibres and the

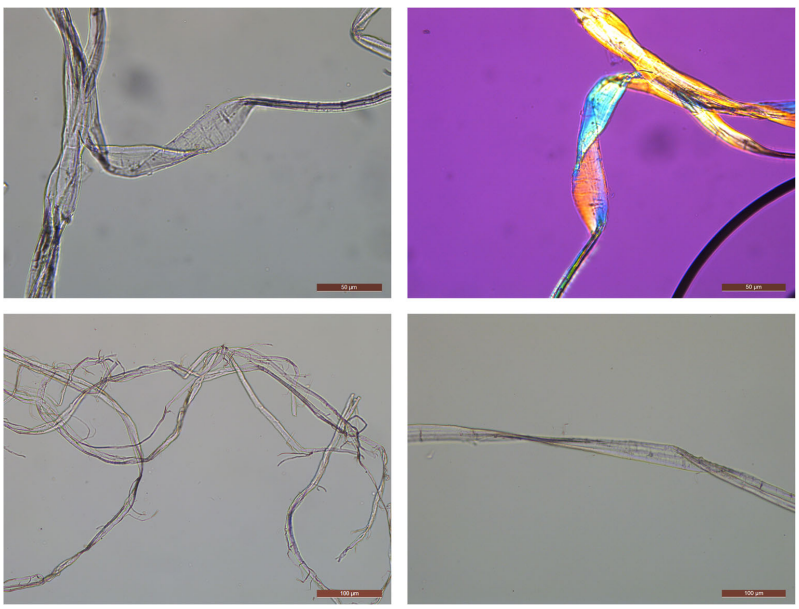

Fig. 5 Sample 1.1. (Upper left) Wide, flattened regions without cross marks are typical for hop fibres; (upper right) the same micrograph in crossed polars, with full wave compensator inserted. These flattened regions often show strong interference colours (the objective HI PLAN POL × 40/0,65 used for both figures). (Lower left) Thickness variations along the fibre's length in an irregular way; (lower right) twists typical for hop fibres (the objective HI PLAN POL × 20/0,40 used for both figures)

bottom section is made with a fibre blend of hop and hemp fibres. The quality of the upper section is rather soft and fine compared with the bottom section. This shows that the textile quality is a result of fibre processing and selection and is not an inherent quality of the plant fibre species used.

Our fibre analysis result for the upper section differs from that of Skoglund (2016, p. 88), who claims that flax is also present. Of course, we cannot exclude that sampling on two different sections of the garment may contain different fibres. Alternatively, if very thin fibres (less than 20 μm diameter) were investigated, a false result is possible as investigation on reference samples have shown. It is important to take into consideration that methods in microscopy, such as fibre analysis and microchemical tests, are comparative studies that build upon each other. Note also that the identification method used here (Lukešová et al. 2019) is for cultivated hop (*Humulus lupulus*). Wild hop has not been investigated. It is very probable that the objects investigated here are made of cultivated hop, but we cannot exclude wild hop completely.

We note that the original museum accession record states that the upper section is made of coarse linen and the bottom section of hemp and hop. Strictly spoken this is not wrong, since linen just refers to the weave, but as mentioned in the introduction, linen is often taken to mean flax, and when other types of fibres are explicitly mentioned, flax is the natural association. Another recent report states that the upper section is made of hemp (Frankow 1992, p.75), which is incorrect. The fabric sample from the textile sample book (NM.0405398+) is made with hop fibres. This agrees with the information in the textile sample book.

Conclusion

In this paper, we present the first experimental evidence of hop fibres in historical textiles. The fibre identification is based on the behaviour of fibres in polarized light, characteristic swelling in cuoxam and fibre morphology following (Lukešová et al. 2019). Both objects are investigated: the woman's garment (NM.0131474) and the textile fragment from the textile sample book (NM.0405398+) confirm the use of hop fibres. Our results highlight the importance of careful material analysis of cultural objects. Precise knowledge of material use in cultural heritage collections is crucial because it is necessary for understanding resource management in the past.

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Article 3

Is It Hop? Identifying Hop Fibres in a European Historical Context

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IS IT HOP? IDENTIFYING HOP FIBRES IN A EUROPEAN HISTORICAL CONTEXT*

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Hop (Humulus lupulus L.*) is an ancient perennial crop plant, native to the Northern Hemisphere. The archaeological evidence dates back to at least the sixth century* AD *in Europe. Hop has been used for beer brewing, in sleeping draughts, as bedding and for antibacterial purposes. Less known is that hop fibres have also been used for textiles and paper. However, it is difficult to distinguish hop from other bast fibres. Here, we present a set of fibre features, which, when found together in an archaeological/historical material within a European context, provide a strong indication that the fibres are hop.*

KEYWORDS: FIBRE IDENTIFICATION, BAST FIBRES, TEXTILE, PAPER, HOP, HEMP, HERZOG TEST

INTRODUCTION

An identification of plant fibres in archaeological and historical material provides important information about resource exploitation, agriculture, textile technology and cultural development. In recent years, there has been an increased interest in the scientific identification of archaeological and historical plant fibre material (see, e.g., Bergfjord *et al*. 2012; Haugan and Holst 2013, 2014; Skoglund *et al*. 2013; Lukešová 2017; Lukešová *et al*. 2017; Paterson *et al*. 2017; Suomela *et al*. 2018).

The main plants grown for fibre production in Europe are hemp (*Cannabis sativa* L.), flax (*Linum usitatissimum* L.) and nettle (*Urtica dioica* L.) (Wild 1970; Bergfjord and Holst 2010; Laws 2010; Bergfjord *et al*. 2012; Gleba and Mannering 2012; Skoglund 2016). All these plants contain bast fibres that can be extracted from the phloem in the plant stems by retting.

Hop belongs to the *Cannabis* family (Cannabaceae), with several species of plants; that is, hemp (*Cannabis sativa* L.) containing bast fibres within the phloem in the stems (Simpson 2011, 334,

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336). Hop is an indigenous, herbaceous, perennial climbing liana that may climb up to 10 m high (van Wyk 2005, 211). It requires fertile, humus-rich soil for its cultivation in northern and central Europe, Asia and North America (Elzebroek and Wind 2008, 281–3). Hop is dioecious, which means that it develops male and female flowers on separate plants (van Wyk 2005, 211). The resins and essential oils needed for beer brewing are in the female plant flowers—called strobili. The resins and oils are situated in the beaker-like lupulin glands. Cultivated hop has more lupulin glands and produces much more resins, tannins, and bitter agents (Humulon and Lupulon) than wild hop (Barth 2013). Plant propagation is usually done by root cuttings. That is why it is possible and more profitable to cultivate female plants (van Wyk 2005, 211).

Archaeological finds suggest that hop has been used for beer brewing in Europe from at least the sixth century AD (Behre 1998, 1999). A field bottle found in the famous Trossingen grave 58 from the sixth century in the Tuttlingen district of Baden-Württemberg, Germany (Rösch and Fischer 2004; Rösch 2010) sheltered remains of hopped barley beer (Rösch 2008). The earliest European written sources discussing hop cultivation are from the eighth century AD at Geisenfeld in the Hallertau region of Bavaria, Germany (Hornsey 2003, 304). Abbess Hildegard of Bingen refers to beer brewing and describes the antibacterial use of the plant in her *Physica sacra* of *c*.1150 (Laws 2010, 110–13). In the Middle Ages in Norway, it was a duty for all farm owners to cultivate a certain amount of hops (Høeg 1976, 385). The same was the case in Sweden, where the growing of hop plants was obligatory for every farm according to law from 1414 until 1860 (Karlsson Strese *et al*. 2014).

The oldest archaeological findings, where it is clear that the findings are *Humulus lupulus*, are macrofossils from Birka, located on the island of Björkö in Lake Mälaren, Uppland, Sweden, which date back to the ninth century AD (Hansson 1996, 129). One of the oldest parts of the Frostathing law (*Frostatingsloven*, book XIII, no. 11), coming from the 12th century, mentions the cultivation of hops (Hagland and Sandnes 1994, 93).

Pillows containing hops were used as a traditional medicine to ward off insomnia. The inventory of the Ratsapotheke of the Hanseatic town of Lüneburg from 1475 lists 'aqua lupuli', which points out the use of hop as tea and as a water solution for pharmaceutical purposes (Lonitzer 1679; Wiethold 2005). In Norway, the so-called *humlevann* (hop water) was used to treat catarrh until the second half of the 20th century (Høeg 1976, 386).

The reuse of hop plants, both stems and flowers, for pillow and blanket padding seems to have occurred in Danish farms at least until the 19th century (Skougaard 1983). It has also been preserved as bedding in early modern graves (Karg 2001; Wiethold 2005). Hop was even used for paper- and rope-making (Laws 2010, 110). It is very probable that people tried to find a way to reuse the waste material from beer brewing.

There exist at least two historical weaves in Swedish museums that are labelled as being partially made of hop: a chemise from Jämtland, NM131474 (Skoglund 2016, 88) and another textile sample from a sample book (Nordiska museet, Stockholm NM.0405398+). Both objects date from the 18th century. Beer production leads to a mass waste of hop plant material, since it is only the female flowers (cone-shaped strobili) that are used. Hence, it is probable that people tried to use the leftover material, including the bast stems, in different ways. However, hop fibres were most likely not a very commonly used material, one of the reasons being (as we learned during the Hop project) that the retting process of the long, branched herbaceous climbing plant is difficult compared to straight stems of flax, hemp and nettle, which it is possible to bundle into sheaves easily.

Bast fibres from different species are difficult to identify and so a careful investigation is necessary to ensure correct identification (Bergfjord *et al*. 2010; Haugan and Holst 2014). In this paper, we present an experimental study of modern hop fibres using standard, white light and

polarization microscopy—the modified Herzog test (Herzog 1922, 1943; Petraco and Kubic 2004; Haugan and Holst 2013) and microchemical tests (Luniak 1953; Stratmann 1973; Wülfert 1999).

AIMS AND OBJECTIVES

The aim of this study has been to identify features that can distinguish hop fibres from the main European plant bast fibres (flax, nettle and hemp) as well as cotton. An additional aspiration for us was to develop a test based on readily available experimental techniques (Luniak 1953; Stratmann 1973; Goodway 1987; Wülfert 1999; Petraco and Kubic 2004; Catling and Grayson 2007). We use standard, white-light microscopy combined with polarization microscopy—the modified Herzog test (Haugan and Holst 2013). The Herzog test was developed by the textile engineer Alois Herzog in the 1920s (Herzog 1922). It identifies the twist direction of the cellulose microfibrils in the first layer of the secondary cell wall ($S2_1$) of a bast fibre. Flax and hemp have opposite twist directions in the $S2_1$ layer and can therefore be distinguished from each other (Wülfert 1999, 257). A right-handed helix is referred to as Z twist, while a left-handed helix is referred to as S twist. The test depends on a correct interpretation of the interference colours that occur when using a polarization microscope. Cotton, though not a bast fibre, can easily be distinguished from bast fibres with the Herzog test setup as pointed out by Herzog himself (Herzog 1943—see also Luniak 1953; Haugan and Holst 2013).

Very little work has been done on the microscopic investigation of hop fibres. Herzog makes a brief mention of the xylem cross-section of the hop stem in PLM (Herzog 1943, 176). Hop fibres were recently investigated using various techniques (Reddy and Yang 2009); however, they did not describe the morphology of hop fibres. The study shows that hop fibres have higher cellulose content than hemp and that the crystal structure is similar, but that hop has lower crystallinity—a relatively smaller amount of ordered cellulose microfibrils (Reddy and Yang 2009).

METHODS AND EXPERIMENTAL APPROACH

Hop plants (*Humulus lupulus* L.) from the Botanical Garden of the University Museum of Bergen were harvested in November in their mature state. We investigated one female individual (Fig. 1). The stem was cut into ~20 cm long pieces. Three parts of the plant were studied: the bottom (close to roots), middle and top stems.

A micro-CT scan was performed on a hop stem coming from the middle area of the plant in order to visualize the distribution of fibres in the stem. We used Bruker micro-CT Skyscan 1272 compact X-ray microtomography with a 50 kV X-ray source and a cooled 1.3 megapixel X-ray camera that goes down to 6 μm 3D spatial resolution. It allows 3D image analysis and realistic visualization.

The method of fibre extraction was carefully considered, because any fibre processing can potentially change the fibre structure and morphology. We decided on a traditional retting process because this is likely to be the method used when fibres were processed for textiles and paper. The stems were retted in a water bath for 2 weeks at 20°C and extracted mechanically from the half-dried stems by hand. As mentioned earlier, it was more difficult to extract fibres from hop than from flax, hemp or nettle.

We used hemp, flax and nettle reference samples for comparison with hop fibres. All reference samples were extracted by water retting. Before we move on to present the experiments, we wish to make the point that it was not difficult to find non-typical bast fibres in the available fibre material. There were many typical fibres in the sample material but also many non-typical fibres.

Figure 1 *The hop plant (Humulus lupulus L.) in a dried state—one female individual, (Copyright © Hana Lukešová, the University Museum of Bergen.)*

The fibres presented here were selected as particularly suitable for demonstrating the points that we want to make, but they were in no way unique. When we observed our samples in the longitudinal direction, every field of view (using a 20× lens) offered many areas of a fibre that were special and different from other types of plant bast fibres—we explain this further in the 'Results' section. Some areas were similar in appearance to other plant bast fibres and cotton fibres, but we did not need to separate the fibres with special features from others to be able to identify them. Every random sample contained enough special features needed for distinguishing hop fibres from other plant bast fibres.

Sample preparation

The separation of fibre bundles was done in a wet state using fine tweezers, tungsten needles and a stereo microscope (Lukešová 2017). Single fibres were mounted in Meltmount ($n_D = 1.662$) on a glass slide with a cover glass according to a protocol (Wülfert 1999, 325). Cross-sections were mounted in EpoFix ($n_D = 1.571$), cut with a diamond saw, ground and polished down to a thickness of about 80 µm. Samples used for microchemical tests were mounted in water ($n_D = 1.333$).

Microscopy

The samples were investigated using a Leica DM750 P polarized light microscope. The microscope was equipped with HI PLAN POL (10×/0.25, 20×/0.40, 40×/0.65 and 100×) objectives. A full-wave compensator ($\lambda = 530$ nm) oriented at –45° was used for the modified Herzog test. Photographs were taken using a Leica MC170 HD camera and the LAS V4.9 software. Fibres were first observed in transmitted white light both in longitudinal direction and in cross-section. Polarized light was used for performing the modified Herzog test. A demonstration video on how to perform the Herzog test can be found at https://youtu.be/sC9GlUKjBDE.

Finally, we performed series of microchemical tests using cuoxam, in order to define a specific swelling behaviour of hop fibres. The test has been used as a standard procedure for plant fibre identification (Luniak 1953, 80; Stratmann 1973, 58–62; Wülfert 1999, 281–2, 320). Cuoxam, which is also called Schweizer's reagent, is tetraamminediaquacopper dihydroxide $[Cu(NH_3)_4(H_2O)_2](OH)_2$. It cannot be stored and has to be prepared fresh. We used the following protocol: 13 g of

copper (II) sulphate was dissolved in 50 ml of distilled water while being heated up until 50°C. The solution was cooled down and 8.6 ml of 30% sodium hydroxide was added while stirring. The blue precipitate of copper hydroxide was filtered and washed with cold distilled water. An amount of 40 ml of 25% ammonia was added to the moist copper hydroxide until dissolved.

RESULTS

The fibre bundles constitute a thin layer called sclerenchyma (~60 μm) hidden under the primary wall. The main part of the stem consists of the wooden part—xylem—and air. A micro-CT scan of a hop stem fragment shows the sclerenchyma layer with fibre bundles clearly (Fig. 2).

White-light microscopy

The hop fibre specimens examined show strong variation in the diameter, changing the size along the length of the fibre in an irregular way. This is the first and most characteristic feature (Fig. 3, top left). We find that fibres can be up to 85 mm long and show rather oval cross-sections, but there are polygonal shapes as well (Fig. 3, bottom right). The wide flattened regions can easily be found in the cross-sections. The fibre diameter is typically between 5 and 60 μm. The longer axis of the oval and/or flattened fibres was measured.

The next characteristic is that there are fibre parts with frequent cross marks and dislocations, which alternate with typically thick and very flattened regions (Fig. 3, top right). These regions rarely show dislocations and are rather even and smooth. The third typical feature is frequent twists that might remind one of cotton (Fig. 3, bottom left).

Large crystal druses, probably calcium oxalates or other phytoliths, can be found. The fibre tips are pointed. The size of lumen versus the cell wall is irregular along the length of the fibre.

Polarization microscopy and the modified Herzog test

Hop fibres are birefringent due to the ordered cellulose chains in the microfibrils. However, this investigation shows that only some parts of the fibres can be used for the modified Herzog test. The crystallinity (the fraction of ordered cellulose chains, compared to disordered cellulose) is

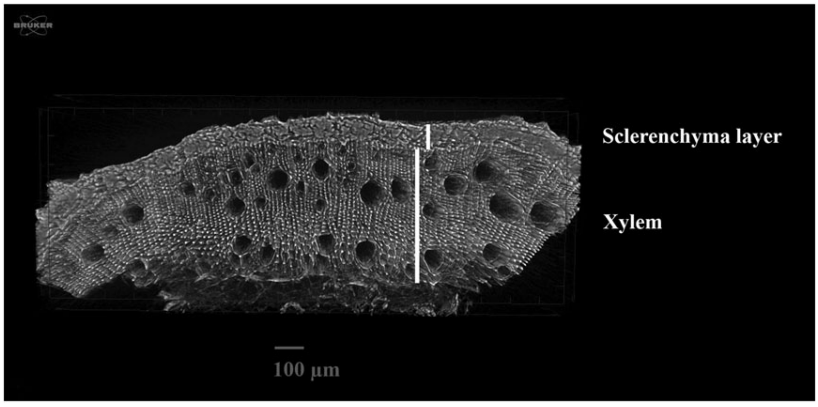

Figure 2 *A transverse section of a hop stem fragment. The fibre bundles are in the sclerenchyma layer. The wooden xylem constitutes the biggest part of the stem. (Copyright © Marcela Kolínová, the Technical University in Liberec.)*

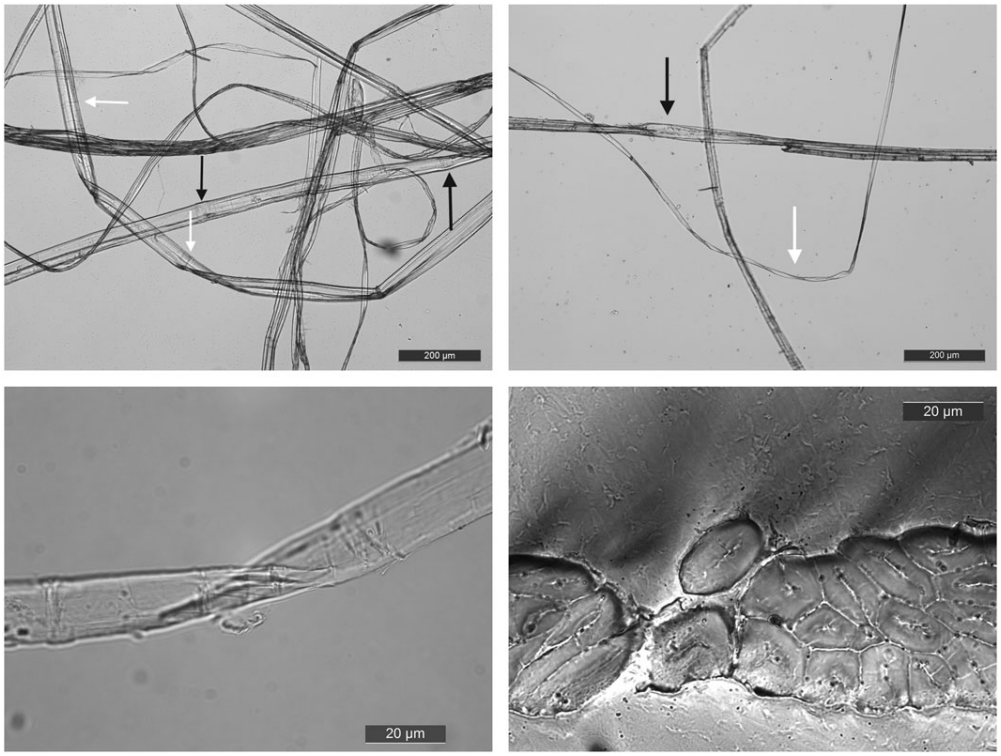

Figure 3 *Top left: cross marks (marked with white arrows) and dislocations (marked with black arrows) are typical for bast fibres. However, hop shows a big variation in the appearance of the fibres. Top right: a thick flat region (marked with a black arrow) and a thin, undulated fibre (marked with a white arrow). Bottom left: frequent, 'cotton like' twists. Bottom right: oval, polygonal and flattened shapes in cross-section, with a large variation in the fibre diameter. (Copyright © Hana Lukešová, the University Museum of Bergen.)*

lower in hops than in hemp (Reddy and Yang 2009, 900). This may be the reason why only some parts of fibres show dark grey extinction. If the extinction in crossed polars is dark enough, the interference colours can be very pronounced when the full-wave compensator is inserted (oriented at –45°). In this case, the interference colours in the orthogonal positions are as follows: 0° position, Orange I and 90° position, Indigo II, according to the Michel–Lévy birefringence chart (Fig. 4). The orientation of microfibril helix in the $S2_1$ layer is therefore in Z-twist, which differentiates hop from flax and nettle but is similar to hemp, as one would expect given that hop and hemp belong to the same plant genus.

Thick flattened regions show often shimmering Orange I and Indigo II in both the 0° and the 90° positions, crossed polars and full-wave compensator at –45°, and are therefore not suitable for the Herzog test.

Additional microchemical tests

Cuoxam—tetraamminediaquacopper dihydroxide $[Cu(NH_3)_4(H_2O)_2](OH)_2$ We compared hop, hemp, flax and nettle behaviour in cuoxam, with the result that hop shows different swelling than

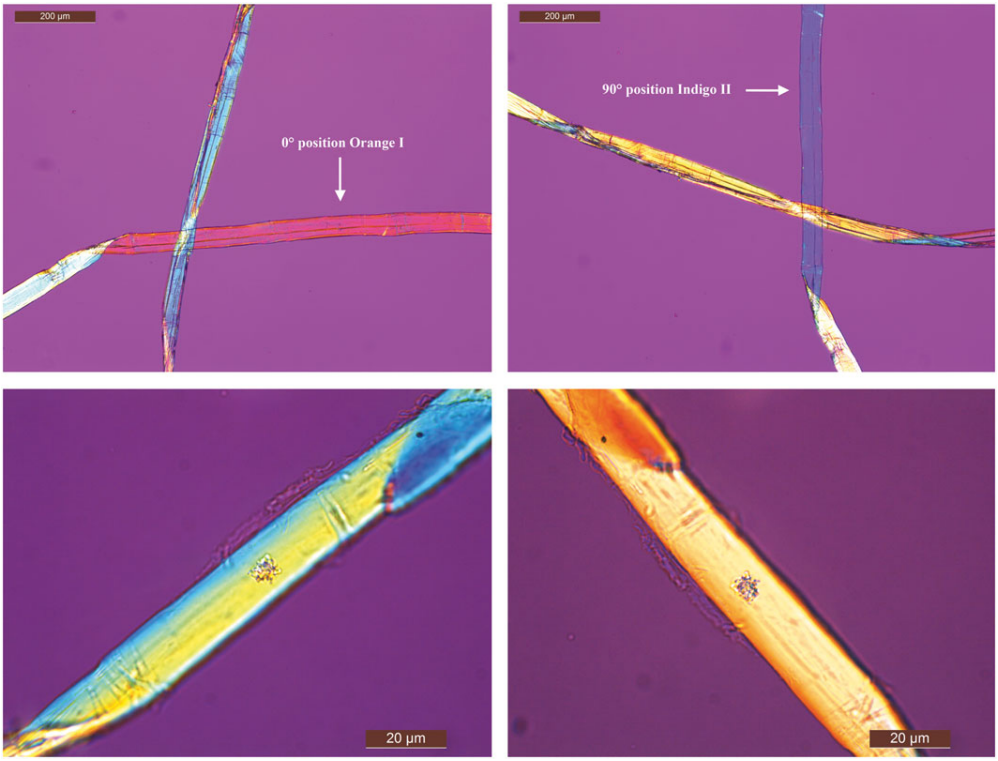

Figure 4 *Top left: hop fibre in the 0° position shows Orange I. Top right: hop fibre in the 90° position shows Indigo II. Bottom left: large-crystal druse in the 45° position. Bottom right: large-crystal druse in the –45° position. Note that the polarization filters are oriented according to DIN 58879 and the lambda plate is inserted at –45° for the Herzog test. (Copyright © Hana Lukešová, the University Museum of Bergen.)*

hemp, flax and nettle. It is possible to observe the different swelling behaviour between hop and hemp in particular. For the tests, we used fibres with a similar diameter (~40 µm).

Hop fibres are undulated in an irregular way when swelled in cuoxam (Fig. 5, top right). Remains of protoplasm in the lumen are clearly visible especially at the end of a hop fibre, which has a rounded edge (Fig. 5, top left). Hop swells slowly (the first changes after around 20 s) and does not dissolve completely over a period of minutes. This contrasts strongly with flax, which shows complete dissolution.

Hemp fibres show typical constrictions and/or even strangulation as well as 'harmonica-like' folding of the cell walls (Fig. 5, bottom right). Hemp swells slowly in comparison to flax and does not dissolve completely. Flax fibres swell uniformly. There are remains of protoplasm in the lumen that look like a wavy thread and can stick out from a 'trumpet-like' final edge of a fibre. Flax swells fast in comparison to hemp and hop and dissolves completely, as mentioned earlier. Nettle fibres show clear striation of the cell wall. They swell fast in comparison to hemp and hop; and can dissolve completely (Luniak 1953, 124; Wülfert 1999, 281).

The following characteristic features were found and elaborated into a diagram (Fig. 6) as a procedure for how to identify hop fibres:

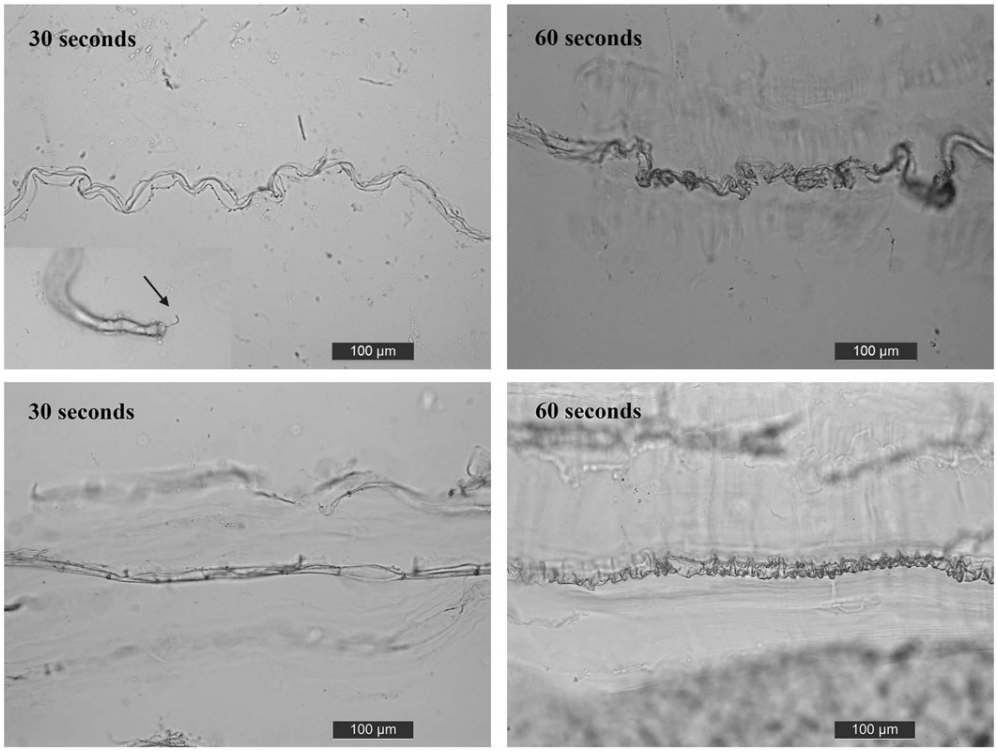

Figure 5 *Top left: hop fibre dissolving in cuoxam in 30 s. The remains of the protoplasm are sticking out of the fibre end (detail). Top right: the fibre is undulating in an irregular way. Bottom left: hemp fibres are swelling slowly. Bottom right: hemp in cuoxam shows typical harmonica-like folding of the middle lamella. (Copyright © Hana Lukešová, the University Museum of Bergen.)*

1. Hop fibres are very long (up to 85 mm) and vary strongly in diameter along the length. Variations from typically 5 to 60 µm were observed in this study.
2. Some fibre regions having frequent cross marks and dislocations often alternate with typical thick and very flat regions. These regions do not show dislocations very often and are rather smooth.
3. 'Cotton-like' flexions that can sometimes cause fibre undulation are often observed.
4. Large crystal druses, up to 10 µm, can be found.
5. The shape and size of the cross-section varies (see point 1). The cross-section is mostly oval but polygonal shapes are possible. The wide, flattened regions can be seen easily in the cross-sections.
6. Fibres display Z-twist in the modified Herzog test; however, the flattened regions show a mixture of Orange I and Indigo II in both the 0° and the 90° positions, crossed polars and full-wave compensator at –45°.
7. Cuoxam causes fibre swelling followed by a typical fibre undulation that differs clearly from the swelling of hemp. Protoplasm sticking out from a rounded fibre edge is common.

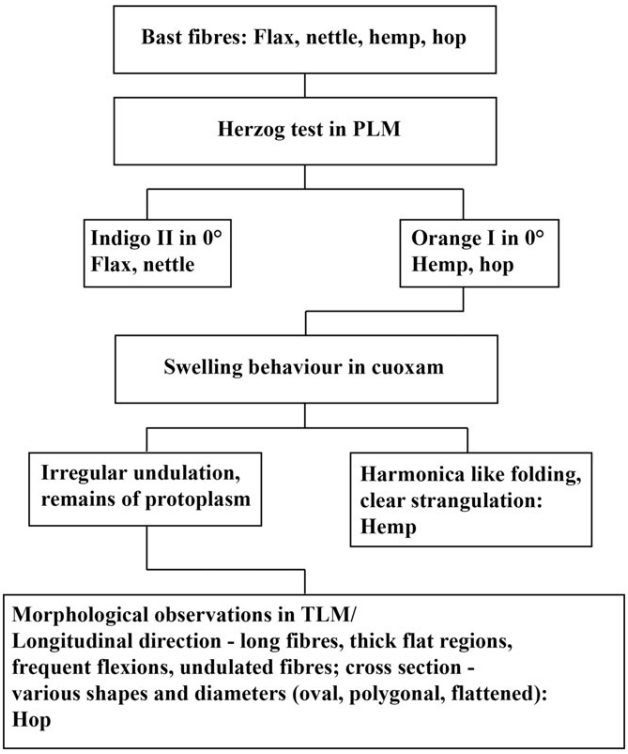

Figure 6 *A characterization procedure showing how to distinguish hop fibres from flax, nettle and hemp. The polarization filters are oriented according to DIN 58879 and the lambda plate is inserted at –45° for the Herzog test. (Copyright © Hana Lukešová, the University Museum of Bergen.)*

DISCUSSION

We have presented several characteristic features for distinguishing hop fibres from other bast fibres that are naturally growing in Europe and have been used for centuries in various types of objects. It is possible to draw conclusions based on typical morphological features, behaviour in polarized light and characteristic swelling in cuoxam. A characterization procedure is presented in Figure 6.

Hop has lower crystallinity—a relatively smaller amount of ordered cellulose microfibrils compared to hemp (Reddy and Yang 2009). From this, one would expect that the interference colours, observed in the modified Herzog test, should be less intense than those observed in hemp. Our experience is that there are fibre regions with very saturated interference colours, but also regions that shows faded results, which cannot be used for analytical purposes. This probably means that the degree of crystallinity varies within a fibre, which can make the difference.

Every randomly selected sample contained enough special features needed for distinguishing hop fibres from other plant bast fibres. However, superficial observation and/or testing that would skip any part of the presented diagram might lead to misinterpretations, since every sample also contained areas that were very similar to commonly used bast fibres—especially hemp.

The characterization procedure presented here is applicable to modern, historical and/or archaeological material. We have found that the modified Herzog test can be applied to degraded,

archaeological material from the Scandinavian Viking Age (Skoglund *et al*. 2013; Lukešová *et al*. 2017). However, the inner fibre structure has to be preserved, which is why charred, mineralized and/or fully metal replaced material cannot yield results (Lukešová 2017). The degree of degradation might also influence the speed of swelling when using cuoxam.

We are aware that plants vary and evolve within one species in different regions and epochs. However, DNA studies indicate that wild hops (*Humulus lupulus* L.) deviated into the European haplotype about 1.05 ± 0.28 – 1.27 ± 0.30 million years ago and show a low genetic variation (Murakami *et al*. 2006, 66). Female flowers that were not fertilized were of better quality for brewing, which is why we have used female plants for our experiments. The reason why cultivated hop has changed little in the past can thus be attributed to the mainly asexual reproduction by root cuttings, since only female individuals are needed for beer brewing, resulting in very few genetic recombination events over time (Karlsson Strese *et al*. 2010, 2012, 2014).

CONCLUSION

In this paper, we present the first detailed, morphological investigation of hop bast fibres using a range of microscopy and chemical methods.

We compare the results with the other native European bast fibres: flax (*Linum usitatissimum* L.), nettle (*Urtica dioica* L.) and hemp (*Cannabis sativa* L.). We present a procedure that allows hop fibres to be distinguished from these other fibres. The procedure is described in Figure 6. The method has many advantages: it is cost-effective, relatively simple, fast and appropriate for systematic investigations when numerous samples are needed. However, it is a destructive method, although a very small amount of sample is needed. Samples with a badly preserved inner structure do not yield results.

With the work presented here, it is now possible to identify the presence of hop in European historical and archaeological contexts. Important factors for obtaining a reliable result are not only the condition of the studied material but also the quality of sample preparation and proper execution of the various tests.

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Article 4

Identifying Plant Fibre Textiles from Norwegian Merovingian Period and Viking Age Graves

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Identifying plant fibre textiles from Norwegian Merovingian Period and Viking Age graves: The Late Iron Age Collection of the University Museum of Bergen

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ABSTRACT

The investigation of textiles and textile production can yield important information about the infrastructure and resource management in ancient societies. Before the 19th century textiles made of plant material in Scandinavia were mainly made from locally available raw materials: nettle, hemp and flax. In 2012, an investigation of ten Scandinavian Viking Age and Early Middle Age wall hangings showed that four of these, including the famous Överhogdal wall hanging, are in fact made with hemp. This investigation demonstrates that hemp, in some cases at least, was also used for fine textile production in Viking Age Scandinavia. The aim of this paper is to investigate this topic further. In order to do so we examined all known Merovingian Period (560/570–800 CE) and Viking Age (800–1066 CE) textile finds in the Late Iron Age Collection of the University Museum of Bergen. The collection is extensive and belongs to one of the oldest archaeological collections in Norway. It contains finds from western Norway mainly. We identified a total of 45 grave finds with more than 100 different weaves in the collection. Plant fibres do not keep well under the burial conditions in Norway, but we managed to identify ten non-mineralized and non-charred finds with fragments of plant fibre material belonging most probably to clothing and accessories. Fibres from these ten finds were investigated using the modified Herzog test. In addition, morphological features were observed carefully. Nine samples were identified as flax, one sample could only be identified as a bast fibre. Our finds show that though hemp was used in some cases for fine textile production in Viking Age Scandinavia, available remains of plant fibre clothing and accessories coming from Hordaland, Sogn og Fjordane and Rogaland counties are made of flax.

1. Introduction

The research on textiles and textile production is an important part of archaeological and anthropological research, because textiles is such an essential part of human culture, be it as clothing or as household articles or as tools such as fishing nets, sail cloth and rope. While plant fibres and animal fibres can easily be distinguished from each other, it is difficult to distinguish between different species within the two groups (Jakes, 2000). Ancient plant fibre textiles have frequently been identified as flax on the basis of superficial examinations. This may have caused a distorted view of the relative importance of flax, nettle and hemp in ancient textile production (Bergfjord et al., 2009; Holm-Olsen, 1976; Bergfjord et al., 2012). Caution should always be taken when looking for example at the identification given in old collection databases.

In the case of the Late Iron Age Scandinavia the situation is comparatively simple, because the only plant fibres available were hemp, flax and nettle since plant material such as ramie, jute or bamboo came only later (Cook, 1968; Kozłowski et al., 2012). Theoretically, cotton might be possible as a Roman import. However, we have not found any reference for such finds in the literature. The question of which plant fibre is used is important, because it can give information about the infra-structure and resource management in the ancient society where the textiles were used. Textiles made of nettle and remains of their production as e.g. retting pits show that wild plants were used (Andresen, Karg, 2011; Bergfjord et al., 2012). In the case of hemp and flax which can both be cultivated, they do to some extend thrive in different growth conditions and give different yields. However, both plants are harvested and processed in a similar manner (Andresen, Karg, 2011; Cook, 1968).

In order to address the issue of textile production properly, a systematic investigation of all textile fragments preserved is required. Here we present an investigation of all known textile fragments made of plant material in the Late Iron Age Collection of the University Museum of Bergen. We examined a total of 45 finds with more than 100 different weaves that were excavated in Hordaland, Sogn og Fjordane and

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Fig. 1. Viking Age textile purse for scale weights coming from a male grave in Jåtten, Hetland, Rogaland B4772_d, sample no.9. The size of the purse is 6,5 × 6,5 cm. (Photograph Svein Skare, © University Museum of Bergen).

Rogaland counties (Lukešová, H., 2011, 2015). Most of the finds consist of remains of clothing and accessories (Fig. 1). The chronology and the division of the Late Iron Age in Norway into the Merovingian Period (560/570–800 CE) and Viking Age (800–1066 CE) is based on the recently republished book from Solberg (2006).

2. Methods

2.1. Identification of textile fragments made of plant material

The first step was to identify textile fragments made of plant material in the finds listed in the Appendix. A detailed examination of the archaeological material was carried out. Previous research of the collection was a great help at the beginning of the project (Bender-Jørgensen, 1986). We started our investigation with visual inspections of the textile remains using a stereo microscope. In the cases where plant material was suspected, fibre samples were taken from the find, mounted (see Section 2.2.) and investigated using transmission (polarisation) microscopy. A typical picture of animal hair on a macroscopic scale coming from an archaeological context in Scandinavia shows round and smooth fibres with a glossy surface whereas plant material often seems dimmed, shapeless, flabby and pasted together. It should be emphasized that a proper identification requires a proper microscopical investigation. However, due to the fragility of the textile fragments and the limited amount of material, we decided only to remove material for transmission (polarisation) microscopy in the cases where the visual inspection of the textile fragments on a macroscopic scale under a stereo microscope suggested plant material. Metal-replaced and/or charred textiles were not included for ethical reasons, since only very small fragments were preserved.

Fibre damage and optical appearance of degraded archaeological textiles is discussed in (Cooke, 1990). Different textile materials have typical light reflection and lustre (Morton, Hearle, 2008) which may be specific even in a degraded condition. It was such optical properties of materials especially that were decisive when looking for plant textiles in the collection. Knowledge of microstratigraphy and grave context was also important when searching. Most of the textile finds were preserved in connection to metal objects as e.g. metal brooches used as functional accessories (Fig. 2).

2.2. Sampling and instruments

Samples of threads, about 1–2 mm long were collected from each textile find. When thread is removed in this way it is irreversible. This is an important ethical aspect which we considered carefully when sampling in the following way: Care was taken to remove as little material as possible, further the exact place where thread was removed was documented and finally the fibre samples were all mounted using a long lasting mounting material so that new investigations can be carried out in the future without distorting the textile fragments further. Single fibres were extracted from each sample and mounted on microscope slides using Meltmount® ($n_D = 1662$) following the procedure described by (Wülfert, 1999). For three samples it was necessary to carry out a second series of tests because no clear results could be obtained. For the second series of tests we used a 3% solution of sodium hydroxide in distilled water as a mountant. The reason is given in Section 2.3. It is important that the mounting is done with great care, the archaeological textile material tends to be very light and brittle which makes the procedure intricate. In addition, fibres are often dirty and contain residues of corrosion products that are difficult to remove.

All experiments were done twice using an Olympus BX-51P compound microscope (equipped with objectives of the type UiS2 series, Ach N and a full wave compensator of wavelength 530 nm) and a Leica Ortholux II POL-BK microscope (equipped with objectives NPL Fluotar series and a full wave compensator of wavelength 530 nm). Several samples from each textile were examined by two independent performers.

2.3. Performing the modified Herzog test

Hemp, flax and nettle are very similar in appearance as discussed above, but fortunately there are several characteristic features, which can be used for identification purposes. The cell walls in bast fibres

Fig. 2. 10th century oval brooches from a richly equipped woman's grave from Vinjo, Aurland, Sogn og Fjordane, B7731_a. The brooches were found together with textile remains B7731_z, sample no.2.
(Photograph Svein Skare, © University Museum of Bergen).

Table 1
A list of all Merovingian Period and Viking Age burial-finds in the University Museum of Bergen that were found to contain non-mineralized and non-charred plant fibre textiles. F/female; M/male; UC/unreliable find context. Sex determining of a buried person is based upon preserved grave goods and the entire grave context, http://www.unimus.no. There were found objects typical for female graves as e.g. oval brooches and other jewelry belonging to the women's costume and objects typical for male graves as e.g. weapons. Dating the textile finds is based upon grave goods and the entire grave context. We used information both from the Museum catalogue http://www.unimus.no and from (Bender-Jørgensen, 1986).

Inv. No.	Provenience	Sex	Fragment structure	Interpretation	Dating
B4772_d	Jåttå, Hetland, Rogaland	UC	Tabby	A purse (Fig. 1)	Viking Age
B4864_g,h	Hyrt, Voss, Hordaland	M,F	Tabby	Women's shift (Fig. 4)	Viking Age, the 10th century
B7731_z	Vinjo, Aurland, Sogn og Fjordane	F	Lozenge twill I.	Women's clothing	Viking Age, the 10th century
B7732_a_2	Skjervheim, Voss, Hordaland	F	Tabby	Women's shift	Viking Age, the 10th century
B7761_s	Hopperstad, Vik, Sogn og Fjordane	F	Tabby	Women's clothing	Viking Age, the 10th century
B8953_a_2	Eide, Stryn, Sogn og Fjordane	F	Tabby	Women's shift	Viking Age, the 10th century
B9014_s	Sanddalen, Gloppen, Sogn og Fjordane	F	Lozenge twill II.	Women's clothing	Merovingian Period
B9765_Id_2	Korsvoll, Gaular, Sogn og Fjordane	M	Tabby	Man's clothing -possibly shirt	Viking Age
B12131	Målsnes, Balestrand, Sogn og Fjordane	F	Tabby	Women's shift	Viking Age, the 10th century
B17186/1/2	Spurkeland, Lindås, Hordaland	F	Tabby	Women's shift	Viking Age, the first half of the 10th century

contain bundles of cellulose chains, the so called fibrils. Cellulose bundles rotate around the fibre interior in successive cell walls in different directions. This is referred to as S-twist or *Z*-twist of a cell wall. Flax, nettle and ramie show S-twist in S_1 (the first layer coming after the primary wall) whereas hemp and jute show Z-twist in the corresponding layer (Wülfert, 1999). The so-called modified Herzog test or red plate test can be used to measure this fibrillar orientation as a reliable analytical method (Bergfjord, Holst, 2010; Haugan, Holst, 2014; Haugan, Holst, 2013; Skoglund et al., 2013). In the modified Herzog test the fibre sample is placed between crossed polars and rotated to extinction (sample turns black). A red plate compensator is then inserted. A colour change, dependent on the fibrillar orientation, will then occur in the sample part that was at extinction before. A fibre having S-twist in the cell wall S_1 will turn yellow/orange (Orange I in the Michel-Lévy Birefringence Chart) and a fibre having *Z*-twist in the corresponding cell wall will turn blue (Indigo II in the Michel-Lévy birefringence Chart) when close to parallel to the analyser (NS- or 90° position). The exact colour change and angle will vary depending on the thickness of the fibre, cell membrane, degradation grade etc. A nice feature of the Herzog test is that cotton fibres, which might have entered as contamination can easily be distinguished (Wülfert, 1999; Haugan, Holst, 2013).

New series of tests were performed when a result of the modified Herzog-test was not clear enough, that is when one of the two performers perceived birefringence colours as not corresponding completely to the Michel-Lévy Chart. We used a different mountant (3% solution of sodium hydroxide in distilled water). Diluted sodium hydroxide enhances the Herzog effect due to swallowing of the sample material (Wülfert, 1999). The disadvantage is that the test has to be done quickly and the sample is not durable.

2.4. Other observations

All fibres examined by Herzog test except one sample which could not be identified were found to have S-twist which means flax or nettle in the context of Scandinavian archaeological material. We searched for calcium oxalate crystals under a polarisation microscope and the fibre morphology was observed carefully to test whether the fibres were flax or nettle. The presence of calcium oxalate crystals would show that the fibres were nettle; however the absence of calcium oxalate crystals can be due to fibre processing and thus cannot be taken in itself as a proof that the fibres are flax (Bergfjord, Holst, 2010). We also searched for "cotton-like" twists in a longitudinal direction that are typical for nettle (Wülfert, 1999).

3. Results

A list of all Merovingian Period and Viking Age burial finds in the University Museum of Bergen that were found to contain plant fibre textiles is presented below (Table 1). Information about provenience, sex of a buried person, fragment structure, fragment group interpretation and dating is listed in the table as well.

An overview of results using transmitted- and polarised light microscopy (modified Herzog-test) is presented in Table 2. Seven of ten samples provided a reliable result using the modified Herzog-test

Table 2
An overview of results using transmitted- and polarised light microscopy.

Sample No.	Inv. No.	Herzog test/series I.	Herzog test/series II.	Morphology	Comments	Result
1	B4864_g,h	S-twist	–	Fibre bundles with cross-markings, fissures; no cotton like twists in longitudinal direction	No calcium oxalate crystals	Flax
2	B7731_z	S-twist	–	Fibre bundles with cross-markings, fissures; no cotton like twists in longitudinal direction	No calcium oxalate crystals	Flax
3	B7732_a_2	No result	No result	Fibre bundles with cross-markings, fissures; no cotton like twists in longitudinal direction	No calcium oxalate crystals; reddish residues of corrosion products	Bast fibres
4	B7761_s	S-twist	–	Fibre bundles with cross-markings, fissures; no cotton like twists in longitudinal direction	No calcium oxalate crystals	Flax
5	B8953_a_2	S-twist	–	Fibre bundles with cross-markings, fissures; no cotton like twists in longitudinal direction	No calcium oxalate crystals	Flax
6	B9014_s	S-twist	–	Fibre bundles with cross-markings, fissures; no cotton like twists in longitudinal direction	No calcium oxalate crystals	Flax
7	B9765_Id_2	S-twist	–	Fibre bundles with cross-markings, fissures; no cotton like twists in longitudinal direction	No calcium oxalate crystals	Flax
8	B12131/1/2	Possibly S-twist	S-twist	Fibre bundles with cross-markings, fissures; no cotton like twists in longitudinal direction	No calcium oxalate crystals	Flax
9	B4772_d	S-twist	–	Fibre bundles with cross-markings, fissures; no cotton like twists in longitudinal direction	No calcium oxalate crystals	Flax
10	B17186/1/2	Possibly S-twist	S-twist	Fibre bundles with cross-markings, fissures; no cotton like twists in longitudinal direction	No calcium oxalate crystals; fibres are brittle, additional dirt is difficult to wash out	Flax

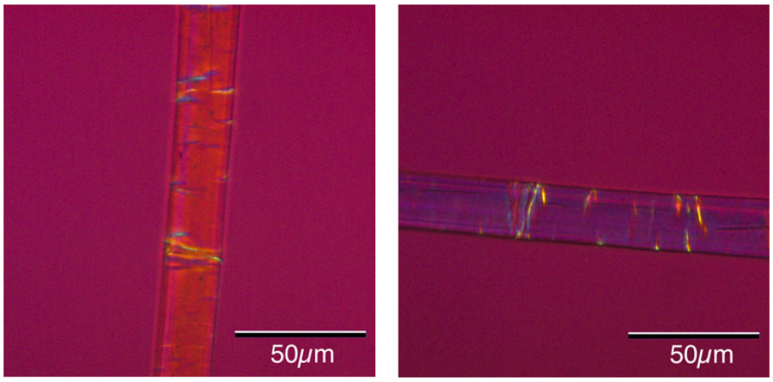

Fig. 3. Result of the Herzog test performed on sample no.1, left: Orange I in 90°- position, right: Indigo II in 0°- position.

during test series I. Test series II was performed for sample no. 3, 8 and 10. Sample no. 8 and 10 provided a reliable result during the second series.

All samples that gave a result were identified as flax (*Linum usitatissimum* L.).

Only sample no. 3 did not show any result for the Herzog-test. However, it is possible to state that the material is bast fibre due to clear morphological features. It is known that the modified Herzog test occasionally does not work even on seemingly well preserved fibre material. This is discussed in (Haugan and Holst, 2013); see also the discussion in Section 4.

Fig. 3 shows sample no.1 with typical results using the modified Herzog test. The colour change shows that it is an S-twist fibre.

We considered the possibility of fibre blends or impurities as far as the limited sampling material allowed. We investigated at least 10 elementary fibres from each thread that showed the same result. Due to the very limited amount of material only one thread system was sampled. The direction of the weave structure was documented. The state of the material does not make it possible to differentiate between warp and weft.

4. Discussion

We have presented the results for textile finds of plant origin coming from Merovingian Period and Viking Age grave finds in western Norway. It is possible to draw conclusions on material use due to previous research (Lukešová, 2011, 2015). We can see that not only plain weave (tabby) but also lozenge twills are identified as plant fibres. Five of the eight tabbies were identified as a layer closest to the body of the buried person (Lukešová, 2015). Flax might have been a preferable material for underwear because unlike animal hair (typically used for upper garments) it does not contain scales and therefore is not itchy.

Fig. 4. Blue tabby made of fine flax fibres interpreted as remains of women's shift (a shirt worn under a suspended dress), sample no.1.

An identification determining the material used in ancient textile production has to be based on analytical methods since superficial surface estimates, based on a subjective visual and/or haptic impression, are not credible. Only such contributions, which are based on reliable data collecting, may open insights into textile production and resource management in ancient societies. Although there are many challenges when identifying archaeological textile material (degradation, minimal amounts of sample material, contamination etc.), it is in many cases possible to get reliable results using a relative easy and low-cost method as we demonstrate here. The method might even have another use: It can be applied for estimation of degradation grade since the modified Herzog test shows results based upon the preserved inner structure of the studied material. If the bundles of cellulose chains are split and the successive cell walls do not present a regular "grid-like" structure any more, the modified Herzog test will not work. Hence a failure of the test may indicate a high degradation grade.

5. Conclusion

In this paper we present an investigation of fibre textiles from the Late Iron Age Collection in the University Museum of Bergen. A total of ten all non-mineralized and non-charred plant textile finds made of plant material were found. Nine samples were identified as flax (*Linum usitatissimum* L.). One sample could only be identified as bast fibre. A recent investigation has shown that hemp has been used for fine textile production in Viking Age Scandinavia (Skoglund et al., 2013). The finds that we have examined stem largely from rich graves. Our results suggest that the preferred material for plant fibre clothes and accessories of high ranked people in Merovingian Period and Viking Age Western Norway is likely to have been flax. Hemp fibres can be as fine as flax fibres (Catling, Grayson, 1982; Wülfert, 1999), so fibre quality alone cannot explain this. The reasons may be related to material availability and local tradition. A full explanation will require a systematic investigation of all Merovingian Period and Viking Age textile finds preserved in Scandinavia. This paper is a contribution towards the topic.

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Conflict of interest

The authors declare that they have no conflict of interest.

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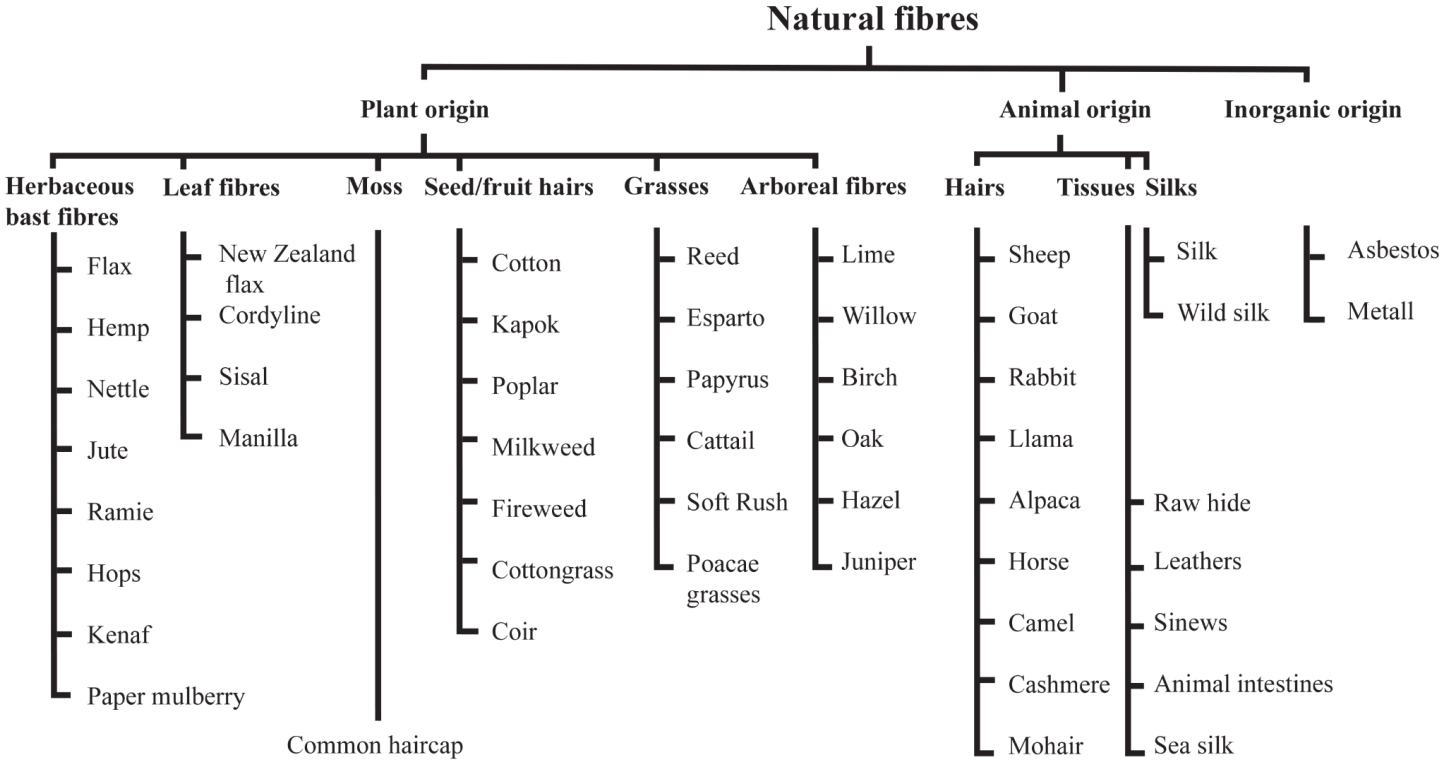
III Appendices

Appendix A


Natural fibres
Plant origin
Animal origin
Inorganic origin
Herbaceous bast fibres
Leaf fibres
Moss
Seed/fruit hairs
Grasses
Arboreal fibres
Hairs
Tissues
Silks
Flax
Hemp
Nettle
Jute
Ramie
Hops
Kenaf
Paper mulberry
New Zealand flax
Cordyline
Sisal
Manilla
Common haircap
Cotton
Kapok
Poplar
Milkweed
Fireweed
Cottongrass
Coir
Reed
Esparto
Papyrus
Cattail
Soft Rush
Poacae grasses
Lime
Willow
Birch
Oak
Hazel
Juniper
Sheep
Goat
Rabbit
Llama
Alpaca
Horse
Camel
Cashmere
Mohair
Raw hide
Leathers
Sinews
Animal intestines
Sea silk
Silk
Wild silk
Asbestos
Metall

An overview of some selected fibres and fibrous materials used for textiles and cultural heritage objects in past [6, 13, 16, 20, 58, 67, 71, 104, 124-126].

Common name	***Latin name***	**Reference**	**Material type**
Alpaca	*Vicugna pacos*	[126]	Animal hairs
Asbestos	*Amphibole*	[20]	Inorganic
Birch	*Betula sp.*	[58]	Arboreal fibres
Camel	*Camelus bactrianus*	[71]	Animal hairs
Cashmere goat	*Capra hircus laniger*	[71]	Animal hairs
Cattail	*Typha latifolia*	[125]	Grasses
Coir	*Cocos nucifera*	[71]	Seed/fruit hairs
Common haircap	*Polytrichum commune*	[104]	Moss
Cordyline	*Cordyline australis*	[67]	Leaf fibres
Cotton	*Gossypium arboreum*	[71]	Seed/fruit hairs
Cotton grass	*Eriophorum angustifolium*	[125]	Seed/fruit hairs
Esparto	*Stipa tenacissima*	[71]	Grasses
Fireweed	*Epilobium angustifolium*	[125]	Seed/fruit hairs
Flax	*Linum usitatissimum*	[71]	Herbaceous bast
Goat	*Capra aegagrus hircus*	[71]	Animal hairs
Hazel	*Corylus avellana*	[58]	Arboreal fibres

Hemp	*Cannabis sativa*	[71]	Herbaceous bast
Hops	*Humulus lupulus*	[124]	Herbaceous bast
Horse	*Equus ferus caballus*	[71]	Animal hairs
Juniper	*Juniperus communis*	[58]	Arboreal fibres
Jute	*Corchus olitorius*	[71]	Herbaceous bast
Kapok	*Ceiba pentandra*	[71]	Seed/fruit hairs
Kenaf	*Hibiscus cannabinus*	[71]	Herbaceous bast
Lime	*Tilia sp.*	[58]	Arboreal fibres
Llama	*Lama glama*	[126]	Animal hairs
Manilla	*Musa textilis*	[125]	Leaf fibres
Milkweed	*Asclepias speciosa*	[125]	Seed/fruit hairs
Mohair	*Capra hircus aegagrus*	[71]	Animal hairs
Nettle	*Urtica dioica*	[71]	Herbaceous bast
New Zealand flax	*Phormium tenax*	[67]	Leaf fibres
Oak	*Quercus sp.*	[58]	Arboreal fibres
Paper mulberry	*Broussonetia papyrifera*	[16]	Herbaceous bast

Papyrus	*Cyperus papyrus*	[71]	Grasses
Poacae grasses	*Poacae sp*	[13]	Grasses
Poplar	*Populus balsamifera*	[125]	Seed/fruit hairs
Rabbit	*Oryctolagus cuniculus*	[71]	Animal hairs
Ramie	*Boehmeria nivea*	[71]	Herbaceous bast
Reed	*Phragmites australis*	[71]	Grasses
Sea silk	*Pinna nobilis*	[71]	Silks
Sheep	*Ovis orientalis aries*	[71]	Animal hairs
Silk	*Bombix mori*	[71]	Silks
Sisal	*Agave sisalana*	[125]	Leaf fibres
Soft Rush	*Juncus sp.*	[71]	Grasses
Wild silk/ Tussah	*Antheraea assamensis*	[71]	Silks
Willow	*Salix sp.*	[58]	Arboreal fibres

Appendix B

An overview of the evaluation of different morphological features commented in literature is presented in the following table [26-28, 32, 36, 49, 50, 58-60, 127, 128]

Morphological feature	**Evaluated as diagnostic**	**Use with caution or as indication**	**Refuted**
Fibre cell length	Gale & Cutler 2000, 412 Carr et al. 2008, 79-83	Luniak 1953, 121 Wülfert 1999, 280 Petraco & Kubik 2004, 89	Catling & Grayson 1982, 78
Fibre cell ends	Gale & Cutler 2000, 412		Herzog 1955, 319 Catling & Grayson 1982, 2
Dislocations and cross-markings	Gale & Cutler 2000, 412	Wülfert 1999, 280 Petraco & Kubik 2004, 89	Luniak 1953, 122 Catling & Grayson 1982, 2 Haugan & Holst 2014, 957
Cross-section diameter	Carr et al. 2008, 79-83 Gale & Cutler 2000, 412	Luniak 1953, 121 Wülfert 1999, 280 Petraco & Kubik 2004, 89	Catling & Grayson 1982, 78 Bergfjord & Holst 2010, 1194
Lumen diameter (ev. the thickness of cell wall)	Catling & Grayson 1982, 2 Gale & Cutler 2000, 17	Luniak 1953, 121 Petraco & Kubik 2004, 89	Bergfjord & Holst 2010, 1194

Cross-section shape	Luniak 1953, 122 Catling & Grayson 1982, 4 Gale & Cutler 2000, 412 Carr et al. 2008, 79-83	Wülfert 1999, 280 Petraco & Kubik 2004, 89	Luniak 1955, 319 Lukesova & Holst 2021, 224
Lumen shape	Luniak 1953, 122 Carr et al. 2008, 79-83	Wülfert 1999, 280	Lukesova & Holst 2021, 224
Cell structure/ **Convolutions/flexions**	Gale & Cutler 2000, 412 Carr et al. 2008, 79-83 Lukesova et al 2019	Wülfert 1999, 280 Petraco & Kubik 2004, 89	
Crystals/ **Crystal shapes**	Catling & Grayson 1982, 3 Luniak 1953, 125 Gale & Cutler 2000, 412 Petraco & Kubik 2004, 107 Carr et al. 2008, 79-83 Bergfjord & Holst, 2010, 1193 Marková 2019, 26		
Adhering tissues **as spiral elements,** **vessels and parenchyma cells, epidermal cells**	Herzog 1955, 253 Catling & Grayson 1982, 3 Luniak 1953, 125 Gale & Cutler 2000, 412		

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