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

H. LUKEŠOVÁ†

Universitetsmuseet i Bergen, P.O. Box 7800 5020 Bergen, Norway

H. L. ANDERSEN

Universitetsmuseet i Bergen, P.O. Box 7800 5020 Bergen, Norway

M. KOLÍNOVÁ

Technicka Univerzita v Liberci Fakulta Textilni, Studentská 1402/2, Staré Město 461 17 Liberec, Czech Republic

and B. HOLST

Universitetet i Bergen Det Matematisk-naturvitenskapelige Fakultet, P.O. Box 7800, 5020 Bergen, Norway

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,

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
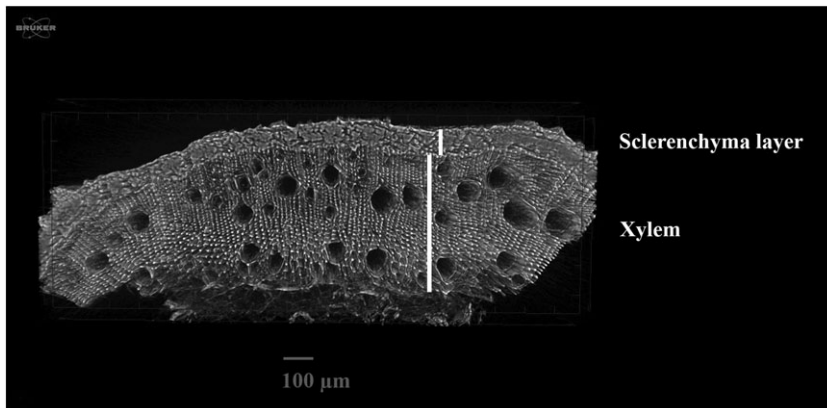

Figure 2   *A transverse section of a hop stem fragment. The fibre bundles are in the sclerenchyma layer. The wooden xylem constitutes the biggest part of the stem. (Copyright © Marcela Kolínová, the Technical University in Liberec.)*
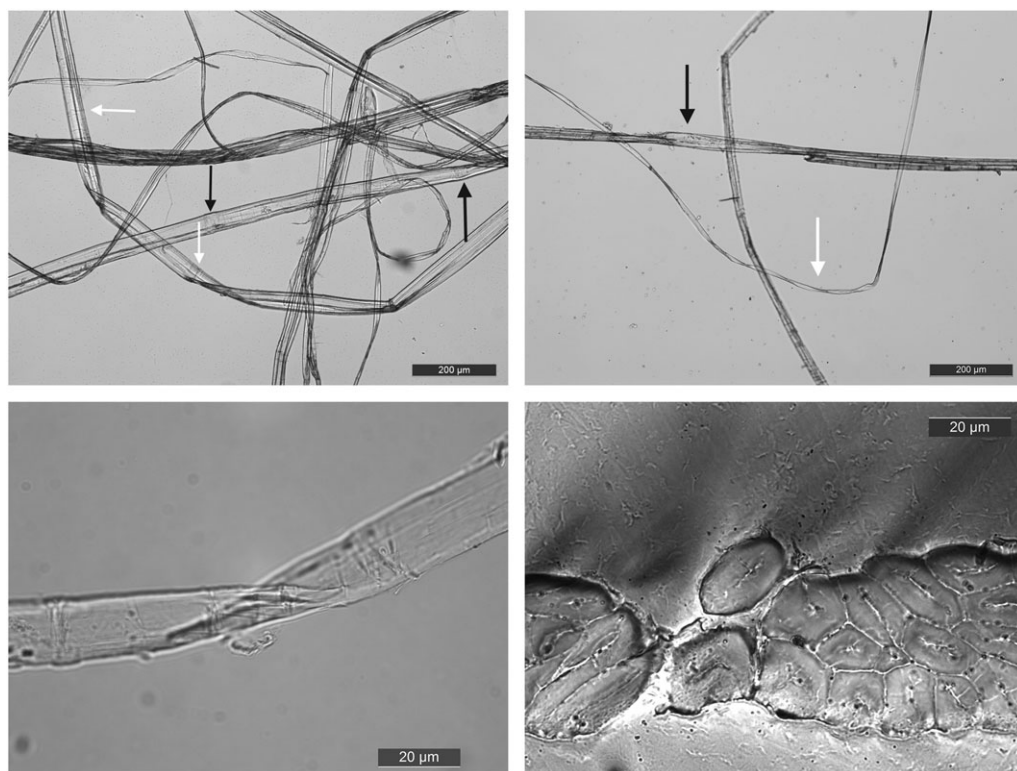
Figure 3   *Top left: cross marks (marked with white arrows) and dislocations (marked with black arrows) are typical for bast fibres. However, hop shows a big variation in the appearance of the fibres. Top right: a thick flat region (marked with a black arrow) and a thin, undulated fibre (marked with 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
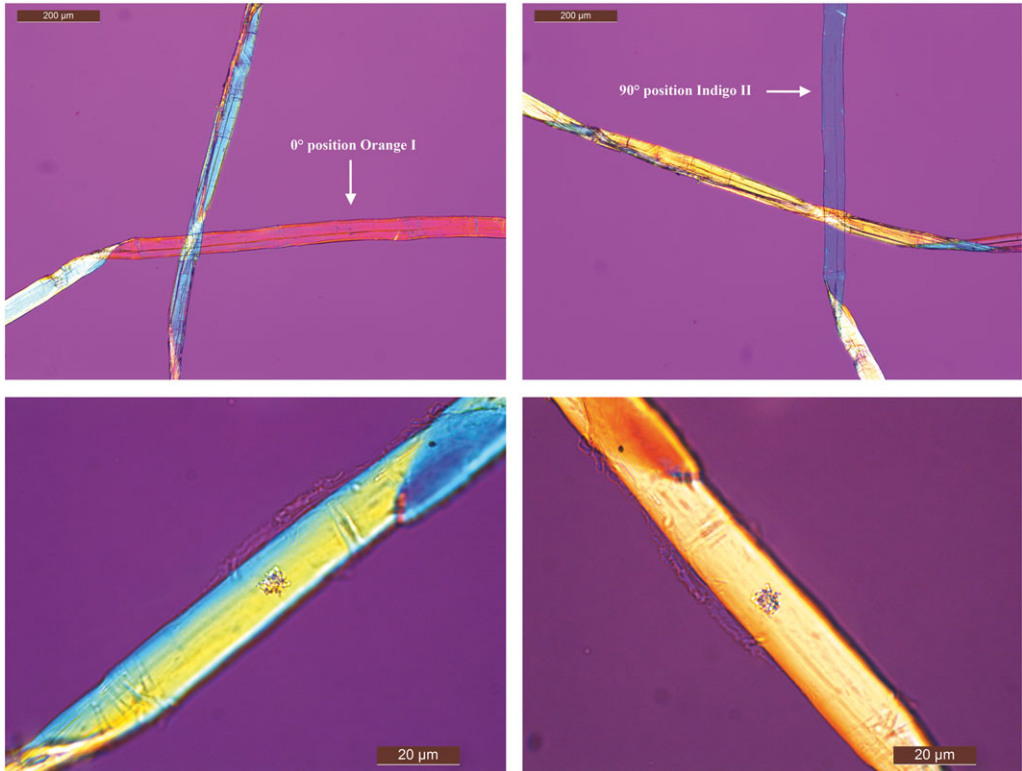
Figure 4 *Top left: hop fibre in the 0° position shows Orange I. Top right: hop fibre in the 90° position shows Indigo II. Bottom left: large-crystal druse in the 45° position. Bottom right: large-crystal druse in the −45° position. Note that the polarization filters are oriented according to DIN 58879 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:
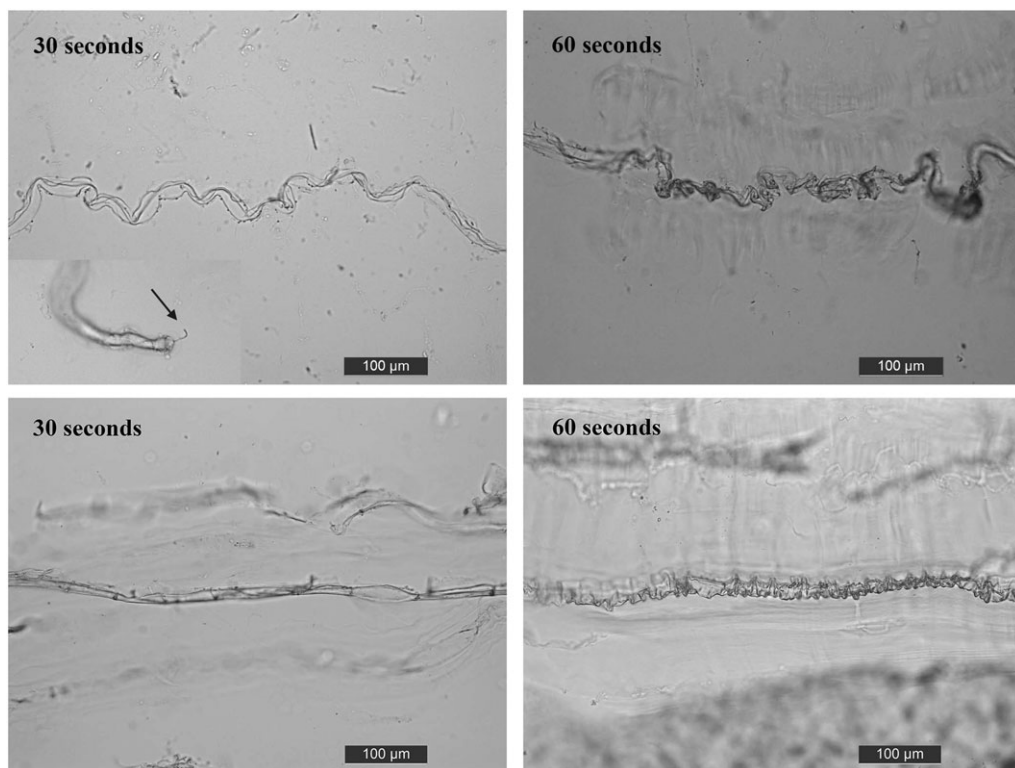
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Figure 5   *Top left: hop fibre dissolving in cuoxam in 30 s. The remains of the protoplasm are sticking out of the fibre end (detail). Top right: the fibre is undulating in an irregular way. Bottom left: hemp fibres are swelling slowly. Bottom right: 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.
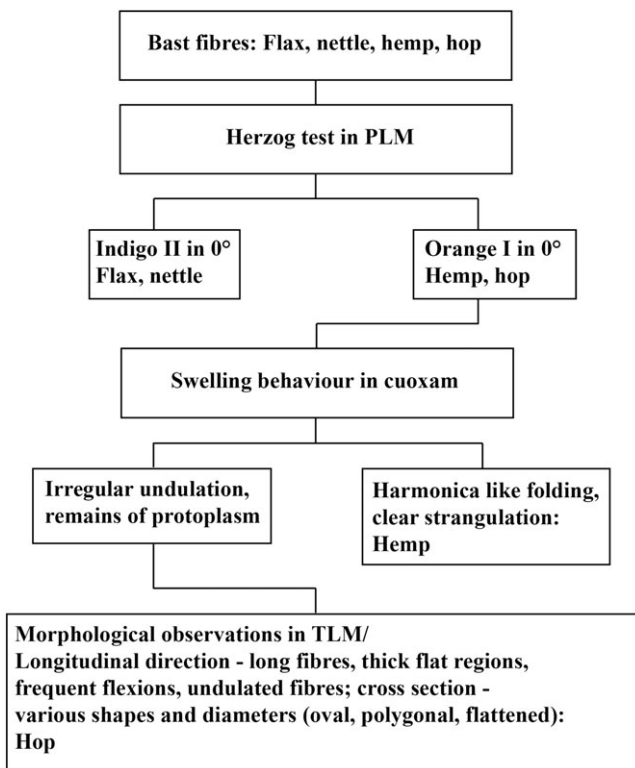
Figure 6   *A characterization procedure showing how to distinguish hop fibres from flax, nettle and hemp. The polarization filters are oriented according to DIN 58879 and the lambda plate is inserted at –45° for the Herzog test. (Copyright © Hana Lukešová, the University Museum of Bergen.)*

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 $1.05 \pm 0.28 - 1.27 \pm 0.30$ 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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