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Colorants Detected by HPLC-PDA in Textiles from 13th Century Lieto Ristinpelto, Finland

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Abstract: Organic colorants of textiles found in the female burial of Lieto Ristinpelto, SW Finland, were analyzed by HPLC-PDA. The textiles' visible colors varied from different brownish shades to blueish, greenish, and reddish hues. The aim of the chromatographic analysis was to deepen the current understandings of the dyes used in Finland at the transition between the 12th and 13th centuries AD, i.e., at the beginning of the local Medieval period, and to contribute important new information about dyes and clothing from this less-known period of textile history of Finland. The textile finds consisted of a bronze spiral ornamented shawl, an apron tied at the waist, two tablet-woven bands, and a diagonally plaited band with plaited tassels. A unique find was a textile possibly made using the sprang technique. Other textile finds were an orange wool tabby and twill fragments. Analysis of thirty samples from fourteen different textiles indicated that woad colorants were present in most samples, accompanied with lichen compounds, and dyer's madder was in two visually orange fragments. The visually reddish samples contained luteolin, but no red colorants.

Keywords: organic colorants; woad; dyer's madder; luteolin; unknown orange compounds; HPLC-PDA; wool textiles; Medieval period

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1. Introduction

This study focuses on the dye investigation of wool textiles found in the Lieto Ristinpelto 86 female burial, situated in SW Finland (Figure 1). Thirty samples from 14 different textiles (Figure 2; Table 1) were analyzed for organic dyes at the textile laboratory of the Royal Institute of for Cultural Heritage in Belgium. As chromatographic techniques require only very small samples, this method has been applied widely in archaeological textile research to detect organic colorants [1–3]. Moreover, this method was considered the most suitable for the unique textiles of Lieto Ristinpelto, as it allows direct comparison with the analyzed dye composition made from several Finnish traditional dye plants in the reference collection [4,5]. From an archaeological perspective, this is the second time that dye analyses have been carried out on Finnish textiles from this period, and thus, the current study of Lieto Ristinpelto 86 burial's textiles, together with the dyes found in textiles from Ravattula Ristimäki site (4 km from Ristinpelto), brings an important new contribution to the knowledge of dyes used and available in early Medieval Finland.

Figure 1. Location of Lieto Ristinpelto and Ravattula Ristimäki sites near the city of Turku in southwest Finland.

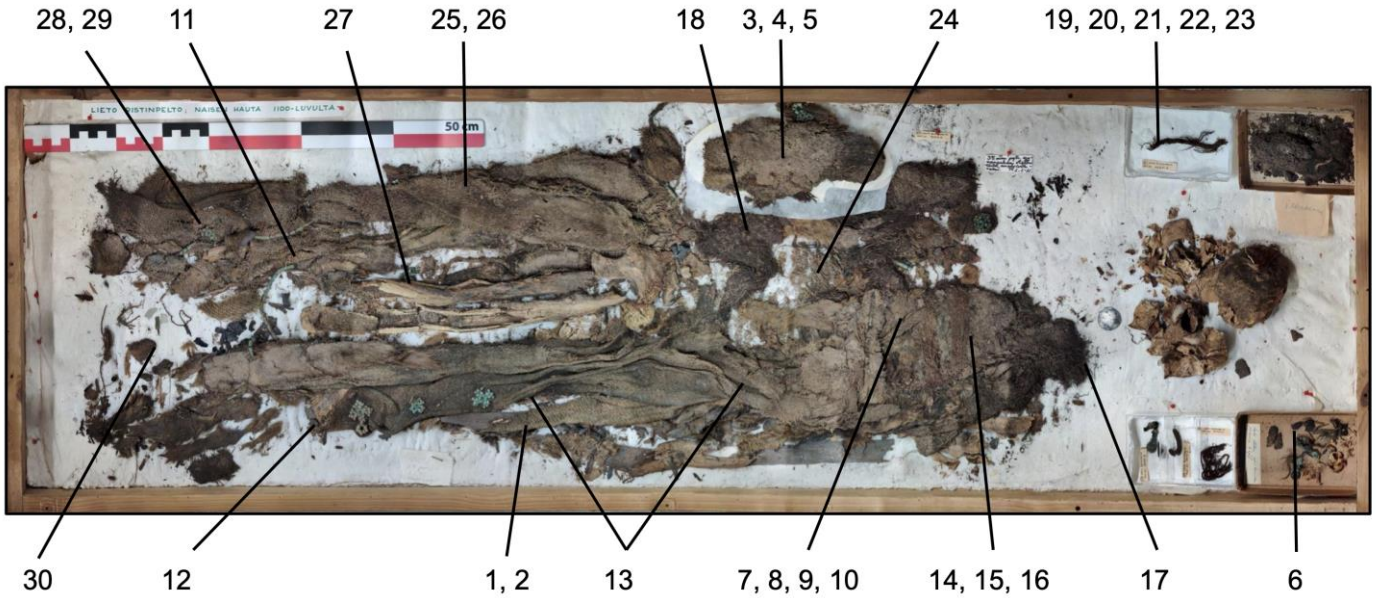

Figure 2. Lieto Ristinpelto grave 86 textiles and the sampling spots for fiber and dye analysis.

Table 1. HPLC results of the wool textile fragments from Lieto Ristinpelto grave 86, with the detected molecules and reference to the possible dye source(s).

No.	Item	Yarn	Detected Molecules	Dye Source(s)
1	Twill dress	Warp	pca, phb, pmb	NDD
2		Weft	Traces of isatin Compounds 1 and 2 pca, phb, pmb	Trace of woad or indigo Compounds 1 and 2

Table 1. *Cont.*

No.	Item	Yarn	Detected Molecules	Dye Source(s)
3	Tablet-woven band with square pattern	Red warp	Trace of luteolin pca, phb, pmb, phc	Trace of luteolin yellow
4		Light warp	pca, phb, pmb	NDD
5		Blue warp	Indigotin, isatin, indirubin Compounds 1 and 2 pca, pmb	Woad or indigo Compounds 1 and 2
6	Diagonally plaited headband of temple ornaments	Warp	-	NDD
7	Diagonally plaited band with plaited tassels	Red warp	Trace of luteolin pca, phb, pmb, phc	Trace of luteolin yellow
8		Blue warp	Indigotin, isatin, indirubin pca, phb	Woad or indigo
9		Light warp	pca, phb, pmb	NDD
10		Blue tassel yarn	pca, phb, pmb	NDD
11	Orange wool tabby	Warp	Alizarin, purpurin (72/28, 255 nm) pca, phb	Dyer's madder
12	Tubular tablet-woven band bordering the bronze spiral decorated shawl	Weft	phb	NDD
13		Warp	Indigotin, isatin, indirubin Compounds 1, 2 and 3 pca, pmb	Woad or indigo Compounds 1, 2 and 3
14	Tablet-woven band from the chest	Red warp	pca, phb	NDD
15		Light warp	pca, phb, pmb	NDD
16		Dark red warp	Luteolin, apigenin pca, phb, pmb, phc	Luteolin yellow
17	Sprang-like textile	Dark yarn	pca, phb, pmb	NDD
18		Light yarn	Trace of isatin, indirubin Compounds 1 and 2 pca, phb	Trace of woad or indigo Compounds 1 and 2
19	Tubular band bordering the sprang-like textile	Red warp	pca, phb, pmb, phc	NDD
20		Dark red warp	Trace of a luteolin pca, phb, pmb, phc, fea	Trace of luteolin yellow
21		Light warp	pca, phb, pmb	NDD
22		Green warp	Indigotin, isatin, indirubin Compound 2 pca, phb, pmb	Woad or indigo Compound 2
23		Yellow weft	Trace of indigotin, isatin Compound 2 pca, phb, pmb	Trace of woad or indigo Compound 2

Table 1. *Cont.*

No.	Item	Yarn	Detected Molecules	Dye Source(s)
24	Threading yarn for bronze spirals bordering the apron	Dark threading yarn	pca, phb	NDD
25	Apron tied at waist	Warp	Traces of isatin, indirubin Compounds 1 and 2 pca, phb, pmb	Trace of woad or indigo + Compounds 1 and 2
26		Weft	Traces of isatin, indirubin Compound 2 pca, phb, pmb	Trace of woad or indigo Compound 2
27	Constructing yarn for a bronze spiral rosette	Threading yarn	Indigotin, isatin, indirubin pca, pmb	Woad or indigo Compound 1 and 2
28	Shawl with bronze spiral rosettes	Warp	Isatin, indirubin Compounds 1 and 2 pca, phb, pmb, phc, fea	Trace of woad or indigo Compounds 1 and 2
29		Weft	Compounds 1 and 2 pca, phb, pmb	Compounds 1 and 2
30	Orange twill	Warp	Alizarin, purpurin (89/11, 255 nm) pca, phb, pmb	Dyer's madder

Previously, dyes of rich textile materials from the Finnish Late Iron Age sites Turku Kirkkomäki, Masku Humikkala, Eura Luistari, and Perniö Yliskylä were analyzed by the means of optical microscopy [6–9] and wet chemistry [10–13]. These analyses detected visually reddish and blue yarns, and indigotin. Absorption spectroscopy and thin layer chromatography (TLC) were applied in Late Iron Age and Medieval textiles from Turku Kirkkomäki, the Turku Åbo Akademi site, and Mikkeli Tuukkala [14–17], with a positive a match for madder or bedstraws, tannin, and indigotin. Recently, HPLC and UHPLC analyses have been applied on archaeological textiles from several inhumation sites from Finland, resulting in the detection of indigoids, anthraquinones of madder or bedstraws, lichen orchil, and several unknown colorants [18–22]. In HPLC analyses of Medieval 14th–15th century textiles from Turku and Masku, the detected dyes were madder anthraquinones, tannin, indigoids, and weld compounds [23–26]. HPLC analyses of Early Modern period textiles from northern Finland have detected madder, indigoids, and unknown red colorants [19,27].

2. Archaeological Background

The textile preservation in the Lieto Ristinpelto grave 86 is exceptionally rich for Finnish materials. Generally, in the local acidic soil, only small pieces of textiles can survive when in direct contact with copper alloy objects. In this grave, the maximum length of some textile fragments is 70 cm; they have survived mainly due to the textiles' bronze spiral decorations. These textiles contain many features found in Finnish Late Iron Age clothing, such as bronze spiral decorated textiles, but also include Medieval influences such as madder dyed fabrics [28–32].

The Lieto Ristinpelto site was archaeologically excavated between 1949 and 1950, when the rich textile and fur materials of the grave 86 were put into storage in the archives of the Finnish Heritage Agency; only the tablet-woven and diagonally plaited bands were analyzed and published [33], while rest of the material remained unexamined until recently.

The only jewelry in the burial was a round silver pendant that imitates a silver coin. Based on the 14C dates from fur [AD 1224–1289 (95.4% probability); 751 ± 30 BP], wool textiles [95.4% probability AD 1044–1086 (26.9%), AD 1093–1105 (3.6%), AD 1118–1219 (64.8%); 896 ± 29 BP], and human bone samples [95.4% probability AD 1034–1178 (92.4%), AD 1192–1202 (2.9%); 926 ± 29 BP] interred in the burial, it dates from the transition between the 12th and 13th centuries AD [34].

Dress finds from 13th century Finland are rare. Accordingly, this study brings remarkable new information about clothing from this less-known period of textile history of Finland. Based on the 150 examined graves in the Lieto Ristinpelto inhumation cemetery, it was used from the 12th to 13th centuries. This period represents a transition between the inhumation practices and rich grave goods of the local Late Iron Age (ca. 800–1150 AD), and the sparse grave goods and churchyard burials of the Medieval period (ca. 1150–1550 AD), with Catholic parishes formed in south-western Finland during the 13th century [35] (pp. 342–344). At Lieto Ristinpelto some individuals were buried in full costume with jewelry, furs, knives, and other supplies, while most burials contained little or no grave goods [36]—a phenomenon acknowledging both the old, local beliefs and the new Christian burial customs. Additionally, the site has the remains of a small building (7 × 7 m), which has been interpreted as serving an ecclesiastical purpose [36,37].

3. Materials and Methods

3.1. Archaeological Materials

A recent survey revealed several textiles in burial 86 (catalogue number KM 8656:15 H15). The textiles were examined and documented in Finland on the premises of the Finnish Heritage Agency using a OnePlus7T mobile phone, and a Leica S6D stereomicroscope with a Leica EC6 camera and Las EZ 3.4.0 software. One-millimeter-long yarn cuts were placed on glass slides, mounted with Entellan New rapid™ and covered with coverslips. These fiber samples were examined in the Nanomicroscopy Center of Aalto University using a Leica 2500 transmitted light microscope (TLM), imaged with a Leica MC190HD camera, and measured with LAS V4.13.0 software to acquire more information about the wool fibers and their natural pigmentation.

All yarns were of wool (Figure 3) according to their morphology and scale patterns [38] (pp. 261–273), except two that were identified as bast fibers. Their identification was based on a three-stage protocol [39], including longitudinal and cross-sectional observation as well as determination of microfibrillar orientation by the modified Herzog test [40] and SFS-EN ISO 20706-1:2019 standard [41]. Threading yarn of the apron's bronze decorations was flax (Figure 4a–d). The tabby veil (or sheet), of which there were only scanty remains, showed a microfibrillar Z orientation, indicating hemp [42].

The dress was 2/2 twill, woven with Sz-plied yarns in the warp and z-spun yarns in the weft (Figure 5a). The shawl's weave and yarns were consistent with the dress and the fabric having a tubular selvedge; the textile was decorated with bronze spiral rosettes, and bordered with a tubular tablet-woven band (Figure 5b). It is likely that when worn, the shawl was held at the shoulders, but in the burial, it was used to cover the deceased. Similar weave and yarn properties were used for the bronze spiral decorated apron, which also had diagonal-plaited bands in its upper corners to tie the apron at waist (Figure 6a). These bands had plaited tassels (Figure 6b), which is a finishing technique found in Finnish Iron Age bands [43].

Two colorful tablet-woven bands rested on the chest area; they were woven using Sz-plied warp yarns and the double-face weaving technique previously only identified in Finland in a single 12th century burial [43,44]. The better-preserved band had a pattern forming squares (Figure 7a), while the pattern was not as apparent in the second band (Figure 7b). Fragments of a diagonally plaited band with hanging temple ornaments and small bronze spiral rosette were found near the skull.

Figure 3. Wool fibers of the shawl's warp band with some visible scales and blue dye.

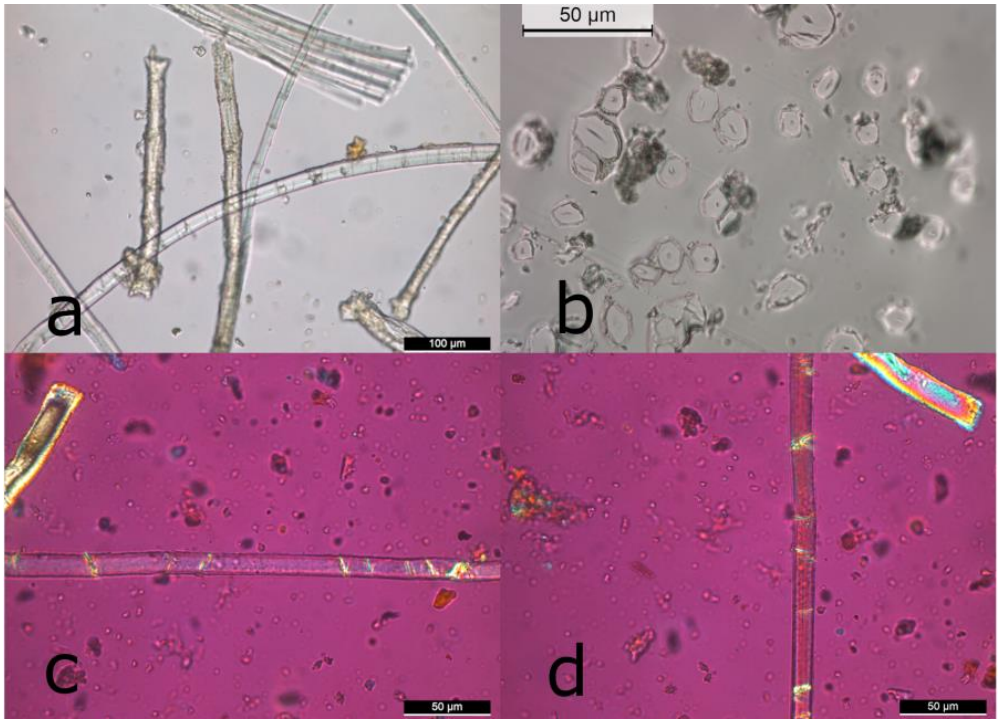

Figure 4. Flax (*Linum usitatissimum* L.). (**a**) Longitudinal view, (**b**) cross-sections showing fiber's polygonal shape, (**c**,**d**) color changes in the Herzog's test revealing microfibrillar S orientation.

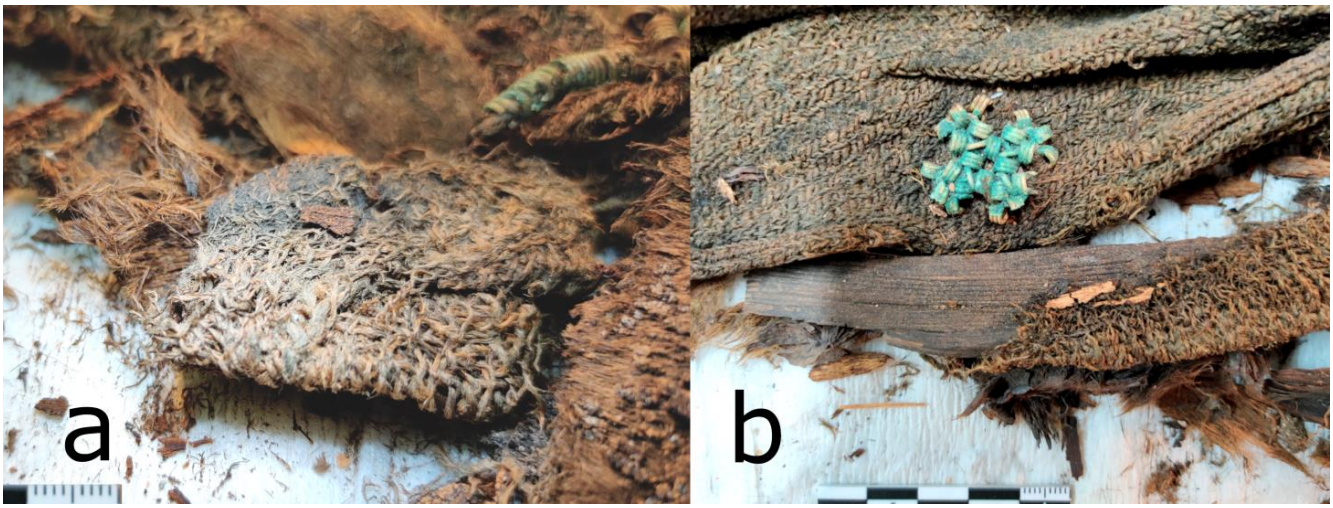

Figure 5. Twill fabrics. (**a**) Dress fabric with altering blue and undyed yarns. (**b**) Shawl, decorated with bronze spiral ornaments and bordered with a tubular tablet-woven band.

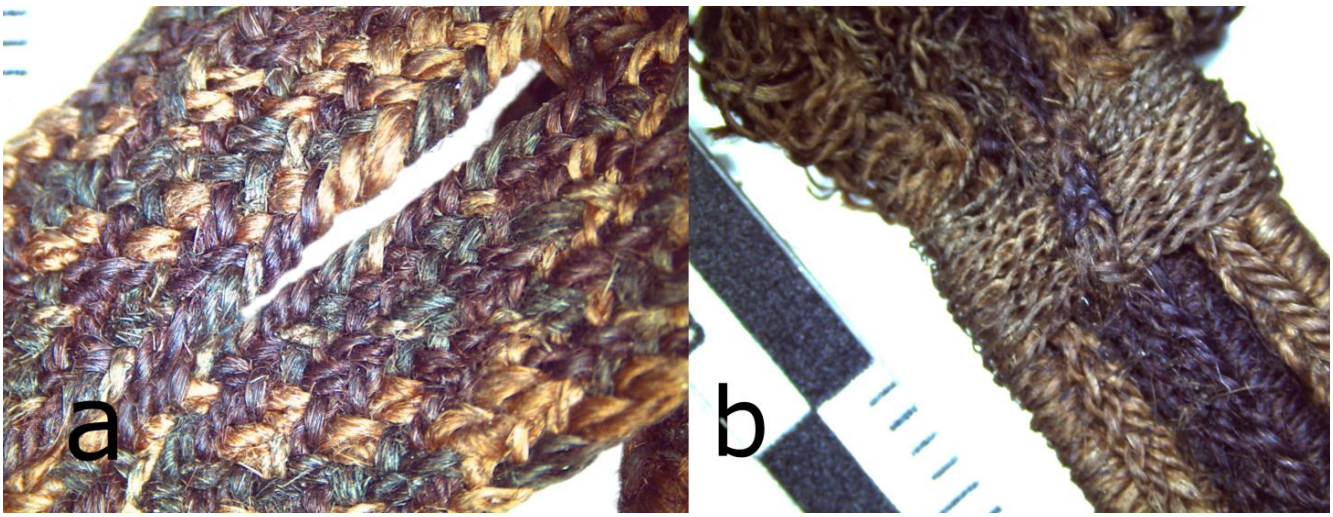

Figure 6. Three-colored bands. (**a**) Diagonally plaited apron band. (**b**) Plaited tassel finishing of the band.

The orange tabby and twill were woven using single-plied s- and z-spun yarns (Figure 8a). These small fragments were found near the feet, and may be sock remnants. A unique find was a sprang-like textile, placed on the upper body. It was crafted using Sz-plied dark- and light-colored yarns, and bordered with a colorful tubular band (Figure 8b). Since there are no other archaeological sprang textiles from Finland, the structure of this textile was difficult to completely parse.

3.2. Chromatography

The analysis method used was high-pressure liquid chromatography and photodiode array detection (HPLC-DAD), made using Water's ACQUITY Arc system with a column heater/cooler and a quaternary solvent manager, a 2998 PDA with low dispersion flow-cell detection system, and Empower3 data handling software. The solvents used were (A) methanol (for HPLC > 99.8%), (B) a mixture of 1/9 (v/v) methanol/Milli-Q water, and (C) 0.5% phosphoric acid (85 wt% p.a.). The analysis was performed at a flow rate of 1.2 mL/min with the following gradient: isocratic state 23A/67B/10C for 0–3 min, linear gradient to 90A/0B/10C between 3–29 min, and isocratic 23A/67B/10C from 30–35 min.

For the stationary phase, a temperature-controlled column of LiChrosorb RP-18 with a 125 mm × 4 mm diameter end cap with 5 µm particle size and 100 Å pore diameter was used.

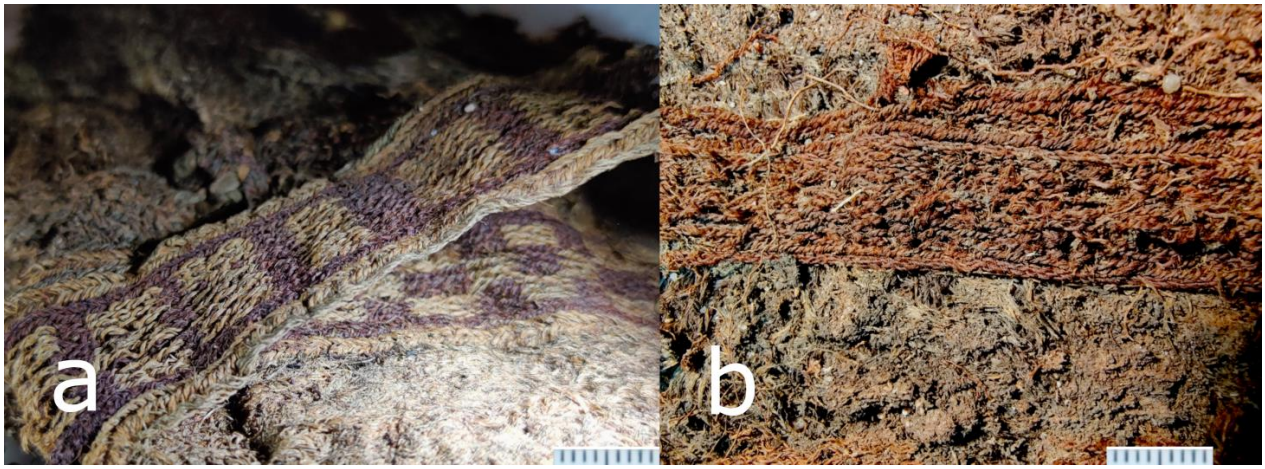

Figure 7. Tablet-woven bands. (**a**) Band with a square pattern. (**b**) Visually reddish tablet-woven band.

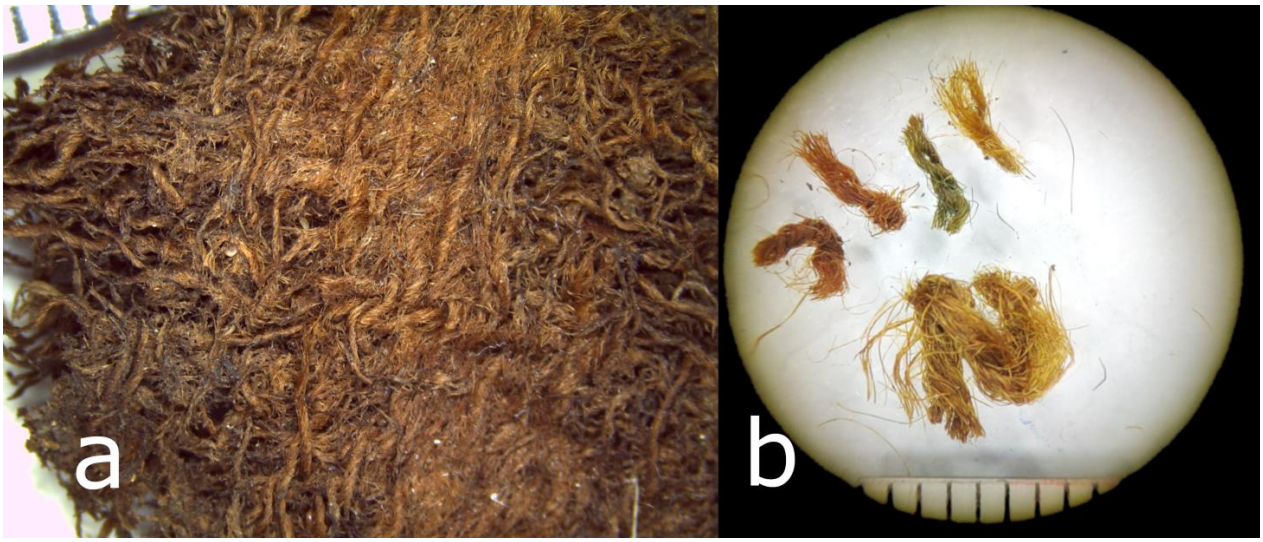

Figure 8. Visually orange and red yarns. (**a**) Orange twill fabric. (**b**) Yarn samples from the tubular band bordering the sprang-like textile.

3.3. Dye Extraction

Before analysis, the small 4 to 6 mm-long thread samples were examined under a Zeiss Stemi stereomicroscope to determine fiber type, morphology, color or hue, and sample condition. Where necessary and to the extent possible, surface contaminants were removed. The color extraction procedure was then chosen based on these initial observations. The dyes were extracted by treating the fibers in 250 µL water/methanol/37% hydrochloric acid (1/1/2, *v*/*v*/*v*) for 10 min at 105 °C in open Pyrex tubes in a heating block. The dye solution was further purified by adding 500 µL ethyl acetate. After a few minutes, the upper phase was decanted, and the ethyl acetate solution was dried in a vacuum evaporator. The dry residue was dissolved in 30/30 µL methanol/water, of which 20 µL was injected. [1]. If the initial sample size allowed for a second extraction, the remaining sample was analyzed after extraction in 250 µL water/methanol/37% hydrochloric acid (1/1/2, *v*/*v*/*v*) alone. After 10 min in open Pyrex tubes at 105 °C, the sample was filtered and vacuum evaporated. Hereafter, the residue was dissolved in 30/30 µL methanol/water, of which 20 µL was

injected. Visually green and blue yarns (Samples 5, 8, 13, 22) were analyzed after extraction in dimethyl sulfoxide for 10 min at 80 °C, followed by immediate injection. When possible, the dry residue sample was subsequently extracted with hydrochloric acid and a second analysis performed on the solution obtained.

4. Results

Of the 30 analyzed yarns, colorants were present in 18 cases, either as dye or at the trace level. No dyes (NDD) were detected in 12 samples (Table 1) [45].

4.1. Indigoids for Blue

Indigoids (Figure 9A,B) were present in 11 samples: the detected compounds were indigotin (the main marker), indirubin (the isomer of indigotin), and /or isatin (a precursor). Indigotin was used to dye the border yarns blue in the tablet-woven band with square patterns (Sample 5; Figure 7a), to form a zig-zag pattern in the diagonally plaited band (Sample 8; Figure 6a,b) and in the warp yarn of the tubular tablet-woven band (Sample 13, Figure 5b). The threading yarn for the apron's bronze spirals (Sample 27) was dyed blue, as well as the green-looking warp yarn (Sample 22; Figure 8b) of the tubular band of the sprang-like textile. Minimal amounts of indigoids also occurred in the weft of the dress (Sample 2; Figure 5a), the light-colored yarn of the sprang-like textile (Sample 18), the tubular band's yellow weft (Sample 23, Figure 8b), both the warp and weft of the apron (Samples 25 and 26), and the shawl's warp (Sample 28; Figure 5b).

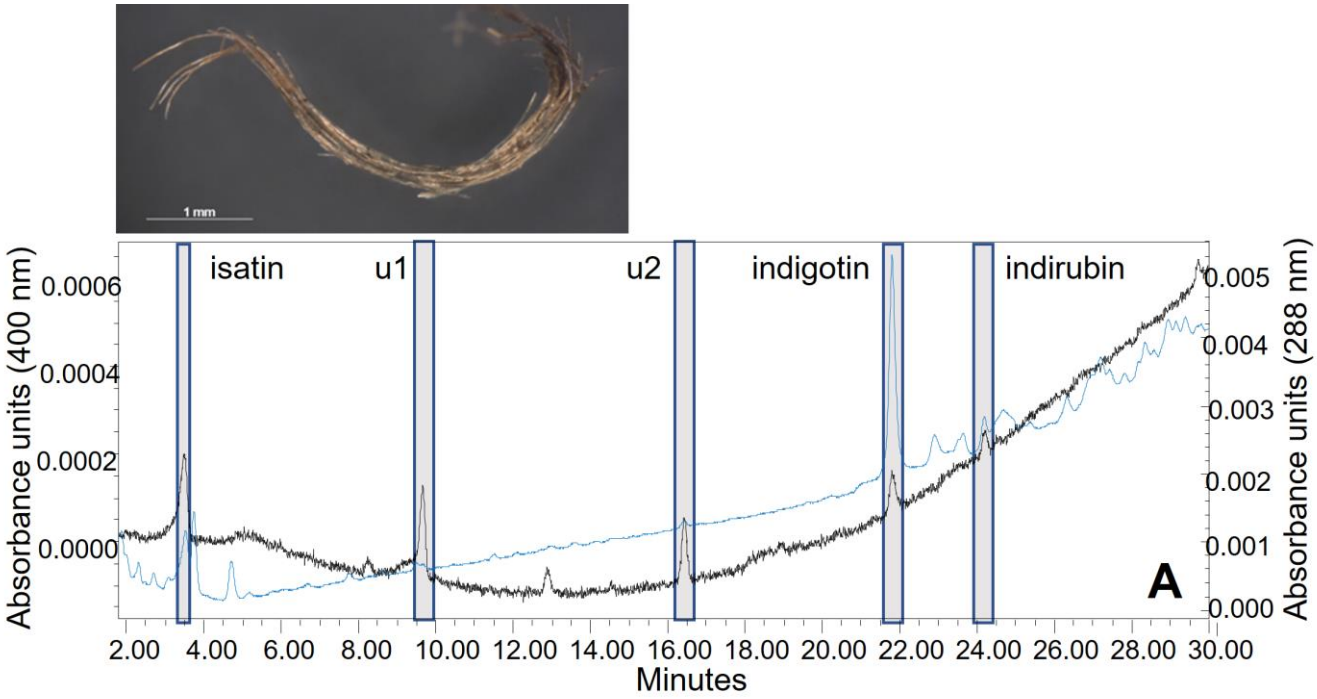

Figure 9. *Cont.*

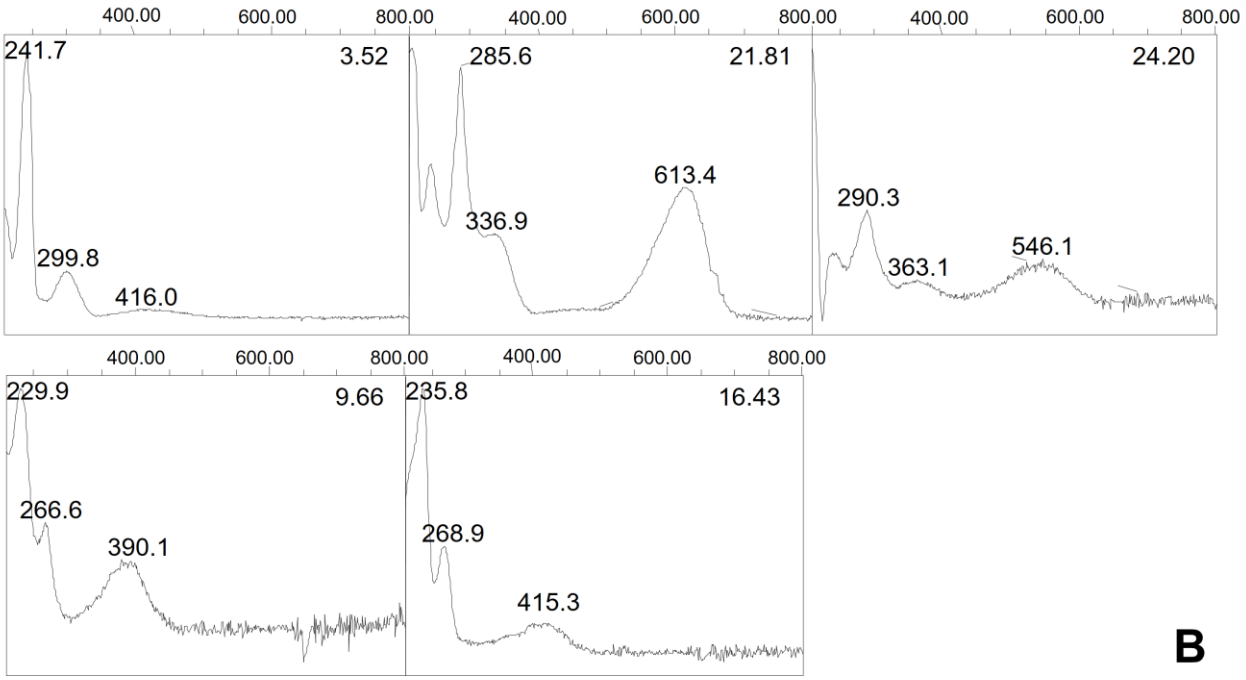

Figure 9. Indigoids and Compounds u1 and u2. (**A**) Chromatograms at 400 nm (black) and 288 nm (blue) of the dye extract obtained with hydrochloric acid and ethyl acetate from the blue yarn of a tablet-woven band (Sample 5) with (**B**) the absorbance spectra between 200–800 nm of the detected dye compounds isatin, indigotin, indirubin, and Compounds 1 and 2 (u1, u2).

4.2. Orange Compounds

In eleven of the thirty yarns analyzed, two chromophoric compounds were found (Compounds 1 and 2). They appeared at 9.6 and 16.4 min and had absorption spectra with a maximum absorbance in the visual light wavelengths at 390 and 415 nanometers, respectively, suggesting an orange-red color (Figure 10). In one sample (Sample 13), they were accompanied by a third, minor compound with a retention time of 19.5 min and absorption maximum at 390 nanometers (Compound 3). In this set of analyses, they were detected ten times in the presence of indigotin, isatin, or indirubin.

4.3. Anthraquinones for Red

Alizarin and purpurin, which are red anthraquinone colorants, were detected in both the wool tabby's (Sample 11) and orange twill's (Sample 30; Figure 8b) warp yarns. In both samples, the alizarin and purpurin were detected in relative peak area ratios of alizarin to purpurin of 72/28 and 89/11 (calculated at a wavelength of 255 nm), respectively (Figure 10A,B).

4.4. Yellow Colorants

The flavone dye molecules luteolin, and to a much lesser extent apigenin, were found in the dark (red) yarn of the tablet-woven band (Sample 16) (Figure 11A,B; Figure 7b). Minimal amounts of luteolin were also detected in three other cases, namely in the reddish yarn of the square patterned tablet-woven band (Sample 3; Figure 7a), in the dark red warp yarn of the tubular band of the sprang like-textile (Sample 20; Figure 8b), and in the yarn of the diagonally plaited band fragment (Sample 7; Figure 6a).

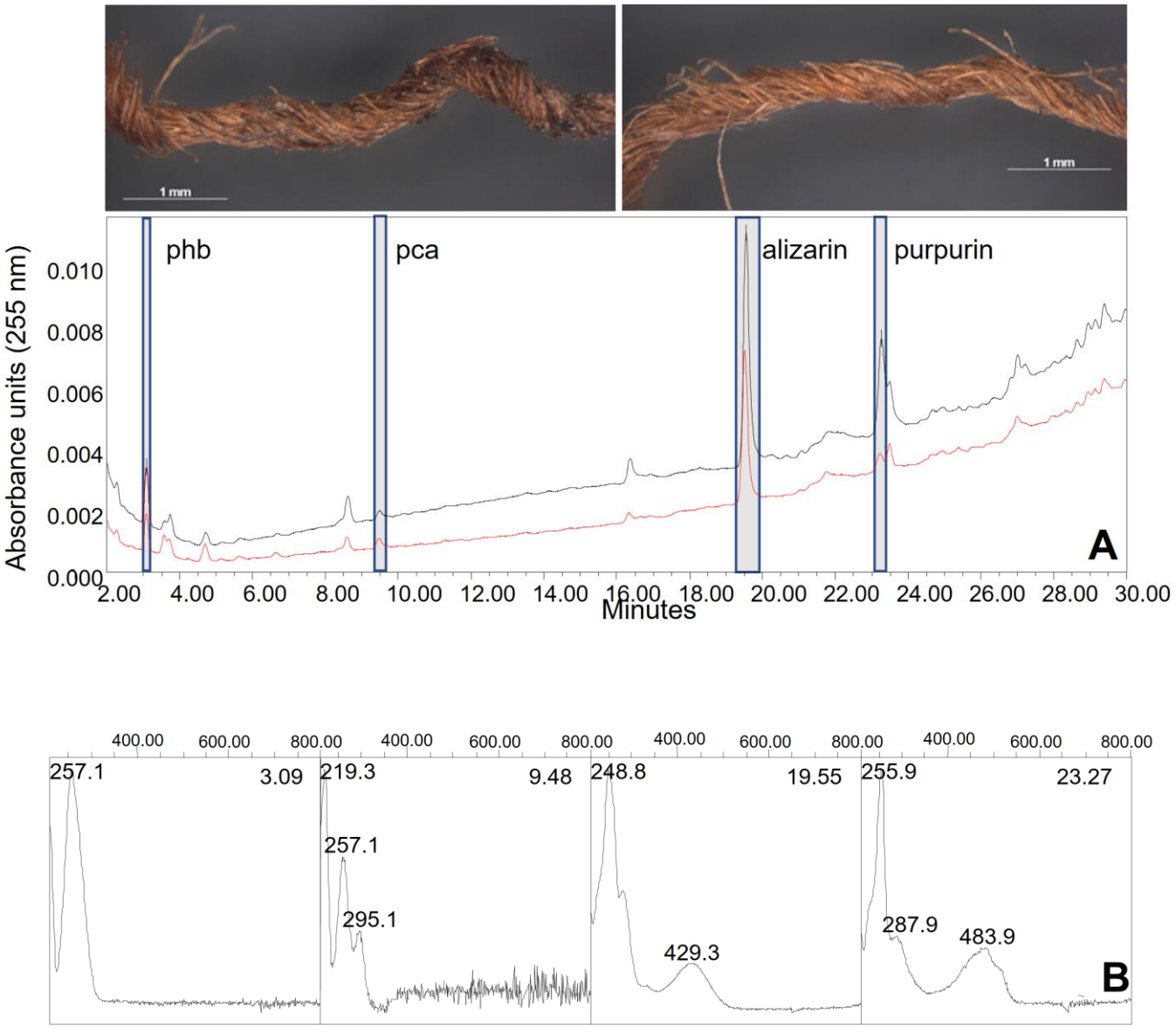

Figure 10. Anthraquinone reds and benzoic acid derivatives. (**A**) Chromatograms at 255 nm of the dye extract obtained with hydrochloric acid and ethyl acetate from the warp yarns of the orange tabby (Sample 11, in black) and the twill fabric (Sample 30, in red), with (**B**) the absorbance spectra between 200–800 nm of the detected benzoic acid derivatives (phb, pca), alizarin, and purpurin.

4.5. Other Compounds

Benzoic (phb, pmb, pca) and cinnamic acid (fea, phc) derivatives were detected in many samples. These are colorless compounds which cannot be related directly to any dye source. Their occurrence, in terms of the peak heights, was rather moderate.

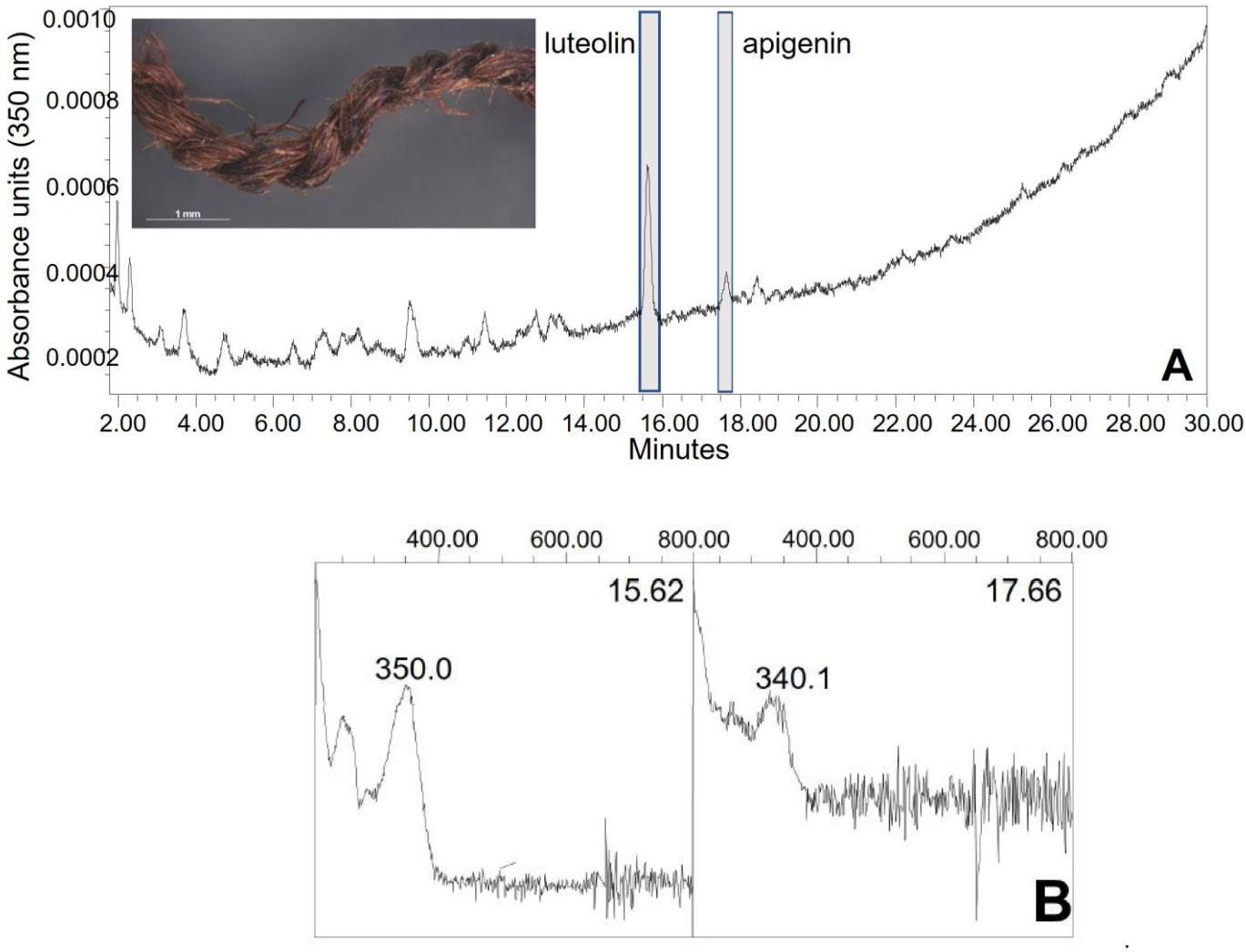

Figure 11. Yellow colorants. (**A**) Chromatogram at 350 nm of the dye extract obtained with hydrochloric acid and ethyl acetate from the dark (red) yarn of a tablet-woven band (Sample 16), with (**B**) the absorbance spectra between 200–800 nm of the detected dye compounds luteolin and apigenin.

5. Discussion

The dye composition found in the HPLC-PDA analysis of the Lieto Ristinpelto yarns does not distinguish between woad (*Isatis tinctoria* L.) or tropical indigo shrub (*Isatis Indigofera* L.). The geographical context (Northern Europe) and the date of the textiles (13th century AD) cannot be used to exclude the use of imported tropical indigo, since tropical indigo was already traded to Europe at this time [46] (p. 364). However, European woad is more likely: it has already been used in blue dyeing in Bronze Age to dye the Hallstatt textiles [47] and it was widely cultivated in at least in Germany by the Middle Ages, and traded all over Europe [16] (p. 368). Woad was probably never cultivated locally in Finland, even though it now grows wild along the sandy shores of Finland's southern coast. Woad was most likely traded to Finland as woad balls by the Iron Age [20,48].

Of the analyzed textiles, only four yarns were certainly dyed blue (Samples 5, 8, 22, 27), based on the clear presence of indigotin, indirubin, and isatin. The yarns were also in good condition, due to their placement in the grave. The tablet-woven band (Sample 5) with square patterns survived due to its direct contact with a bronze spiral rosette, while the diagonally plaited band (Sample 8) survived due its proximity to the apron's spiral bordering, as did the wool yarn threaded inside the apron's bronze spirals (Sample 27). The greenish warp yarn (Sample 22) of the tubular tablet-woven band survived inside the tightly woven band's wefts.

The dress fragments were very degraded, which can explain the trace level of indigoids detected in Sample 2. Additionally, the microscopic examination revealed that the twill dress was woven using plied blue yarns in every second warp, as well as single-plied blue yarns in every second weft. Every second yarn in both systems was undyed, like Sample 1. Similar-type twill, with two differently colored yarns in the warp and weft was recently found in Ravattula Ristimäki grave 41, although this example was woven by alternating naturally pigmented and white wool yarns [29,49]. The trace level of indigoids detected in the apron yarns (Samples 25, 26), as well as in the shawl's warp (Sample 28) and the sprang-like textile's lighter yarn (Sample 18) can be explained by the sampling location near the fragments' faded edges. TLM observations showed the fibers were very eroded, but some were blue in color. It is likely that indigotin was used to dye the shawl's warps, the sprang-like textile, and the apron, as well as the wrapping yarn of the tassel (Sample 10), following the pattern of utilizing blue in textiles in Finnish Late Iron Age attire [7,13,50,51]. The indigoids in the tubular band's yellowish weft (Sample 23) were most likely contaminated by the band's greenish warp (Sample 22), since no blue hue was found in TLM observation.

The orange-red colorants detected with indigoids in 10 samples indicate the use of a combined dyeing with an indigoid blue and another dye source, unidentified so far. These could possibly be lichens, but other dye sources with unknown or degraded dye components under the given environmental or analytical conditions cannot be ruled out. Lichen dyes that have been detected in woad-dyed Late Iron Age textiles found in Finland and Estonia [20,48]. This dye combination (woad and lichens) was also known elsewhere in the Medieval Europe [46] (pp. 499–501). In the weft yarn of the shawl (Sample 29) only orange-red dyes found, forming a vivid texture with the warp yarns that contained indigoids as well.

Orange dye components have been regularly found in Scandinavian archaeological textiles, but they not been identified or attributed to any dye class [1,19]. There is a lot of variation in absorption spectra and retention times which prevents further identification of the specific dye molecules. These are probably spectra of less stable dyes, which have degraded differently under the influence of the specific burial conditions. On the other hand, the mode of dye extraction and/or the chromatographic method may have caused changes in the composition and spectral characteristics of these (less stable) dye(s). The larger number of dye analyses performed on Finnish archaeological textiles during the last decade also bring up similarities; the orange dyes (Compounds 1 and 2) were previously found combined with dyes from indigo/woad in multiple samples from the cemetery of Ravattula Ristimäki near Turku, dated AD 1100–1250 [49], while also unknown orange-red compounds are reported in the presence of indigo/woad in samples from the contemporary sites Turku Kirkkomäki and Masku Humikkala [19,52]. Although the spectra from the latter unknown compounds (analyzed under slightly different extraction and analysis conditions) is not exactly the same as those from textiles found at the Ristinpelto and Ristimäki sites, the detection of such orange compounds together with indigotin might well indicate that it must have been a common dyestuff combination in that period.

The detected anthraquinones alizarin and purpurin indicate that the rhizomes of a plant from the Rubiaceae family were used for red dyeing (Samples 11, 30). Since alizarin was the predominant compound, the dye source was probably dyer's madder (*Rubia tinctorum* L.) instead of local bedstraws (*Galium* species) or woodruffs (*Asperula* species) [53]; the latter two were important local dye plants for stable red from the Late Iron Age to the 19th century, especially in the Nordic countries and Estonia [54]. Although madder dyed textiles were well-known in Scandinavia during the Early and Late Iron Age and the Early Medieval period [55–57], anthraquinone dyes are not commonly found amongst Finnish Late Iron Age or early Medieval textiles. Traces of alizarin was previously detected in the band of a leg wrap from the Turku Kirkkomäki inhumation cemetery, dated to the 11th–12th centuries AD [58]. This might be an indication of the use of dyer's madder. Additionally, local Rubiaceae dyes such as bedstraws (*Galium* sp.) or dyer's woodruff

(*Asperula tinctoria* L.) were detected in 11th century AD textiles from a female burial in the Eura Luistari inhumation cemetery, also located in southwest Finland [59].

In the Lieto Ristinpelto burial 86, dyer's madder was identified in two textile fragments. These were the visually orange tabby and twill fragments, located near the feet. These textile types are not common among Finnish archaeological textiles, which are usually twills with a plied warp yarn and single-plied weft yarn [7,13]. Madder dye, however, has been found in locally woven wool twill socks in Ravattula Ristimäki burial 41 [21,29]. Recent studies of the textiles from Turku, founded in the late 13th century [60], have provided new information of Medieval Finnish red dyes. Dyer's madder was identified in three imported textiles from a relic assemblage at Turku Cathedral. The items have been 14C dated to the 13th–14th centuries, and consist of one cotton and two silk fabrics, presumably of oriental origin [25]. Dyer's madder, bedstraw colorants, tannin, indigotin, and flavonoids of weld (*Reseda luteola* L.) have also been detected in urban textiles from Medieval Turku [23,24,30].

The presence of luteolin in four yarns (Samples 3, 7, 16, 20) indicate the use of a luteolin-based yellow dye source. Without other accompanying dye molecules, a wide range of yellow dye plants might have been used. Together with apigenin, the source of luteolin could be weld (*Reseda luteola* L.), dyer's broom (*Genista tinctoria* L.), chamomile (Anthemis species), and sawwort (*Serratula tinctoria* L.) [61] (pp. 346–357). These plants have a long tradition in the Central European textile dyeing from the Bronze Age [2,47] to the Medieval time [46] (pp. 168–180). In Scandinavian Iron Age textiles luteolin has been detected in several cases [1]. All these plants have luteolin as the main coloring component and some contain apigenin as a minor compound. None of these are native to Finland except scentless chamomile (*Anthemis arvensis* L.); dyer's chamomile (*Anthemis tinctoria* L.) arrived in Finland during the local Middle Ages.

In general, the preservation of flavone dye molecules in Finnish archaeological textiles is very exceptional. Previous research has found apigenin and luteolin only once in Late Iron Age textiles, namely in a three-colored imported textile found in the Mikkeli Tuukkala inhumation cemetery, dated to the early 14th century. Yellow dyes have been connected to the deep cultural changes in Finland at the beginning of the Medieval period due to Christianization and Swedish rule. New influences increased the import of new dye stuffs and alum that was not available in Finnish bedrock [20] (p. 63).

Surprisingly, luteolin-based dye was present in the square-patterned tablet-woven band's visually red warps (Sample 3), in the reddish warp of the diagonally plaited band (Sample 7), the dark red warp of the tablet-woven-band which rested on the chest (Sample 16), and in the dark red warp of the tubular band of the sprang-like textile (Sample 20). In two slightly differently colored visually red yarns, no dyes were detected (Samples 14 and 19). This suggests a colorant that cannot be detected using the HPLC method. Only parallel for these reddish yarns with a luteolin-based dye has been detected in a textile from a 11th century child burial from Turku Kirkkomäki [62]. According to the TLM examination, the wool in all these yarns was unpigmented, so a pigmentation cannot explain the shade of color in these yarns. Other warp yarns in these bands were undyed white wool (Samples 4, 9, 21) and blue dyed wool. When weaving colorful bands, it is mandatory to keep the yarns in order, to create certain patterns. With warps having only slight visual color differences, such as yellow and natural white wool, weaving becomes very demanding. Because of that, it seems unlikely that the reddish hue is the result of the burial context or degradation of yellow dyes.

The detected luteolin-based dye unfortunately cannot explain the several unknown red colorants in the visually red yarns, found in previous Finnish research of archaeological textiles. These compounds have appeared in HPLC analysis in Late Iron Age textiles from southern Finland, as well as in 18th century funerary textiles from northern Finland [19,27] with no parallels in other European archaeological finds. Alder buckthorn (*Rhamnus frangula* L.) bark has been suggested a source of the unknown red compounds detected in Finnish Iron Age textiles [20] while the unknown red colorants found in Medieval textiles

from Turku have been connected to common alder (*Alnus glutinosa* L.) bark dye, which was used in conjunction with iron to produce a black dye [32].

The sprang-like textile's dark yarn and spiral rosette's construction yarn (Samples 17, 24) were undyed, naturally pigmented wool. The square-patterned tablet-woven band's light-colored warp (Sample 4), light warp of the tubular band of sprang-like textile (Sample 7), and the headband (Sample 6) were spun of undyed, white wool.

6. Conclusions

The dye analysis of the textiles in Lieto Ristinpelto burial 86, dating to the transition of the Late Iron Age to the Medieval period, identified several colorants in 18 of the 30 analyzed samples. Most textiles had natural dyed yarns. The headband was not dyed and contained only white wool, and the natural pigmented constructing yarn of a spiral rosette was also undyed.

Indigotin, indirubin, or isatin, likely originating from woad, were detected in 11 samples. This suggests that the bronze rosette decorated shawl was blue, as well as the apron and shawl's tubular tablet-woven band. Blue warps were also present in the diagonally plaited band, and the border warps of the tablet-woven band with a square pattern. The dress was woven with blue-dyed and undyed yarns in both the warp and weft. Blue colorants were accompanied by three orange-red compounds, indicating the use of woad with a second dye source, possibly lichen. Some other Finnish samples from other archaeological sites have contained these same colorants, suggesting a local dyeing tradition.

Dyer's madder was present in two samples, namely in the orange tabby and twill. As a rare dye finding in the Finnish context, this suggests imported textiles. In six visually reddish yarns originating from the bands, no red colorants were detected. Four of these contained the yellow colorant luteolin, once with apigenin. This suggests a luteolin-based dye source, and/or an unknown local dye source that cannot be detected by the HPLC-PDA method.

The textile and yarn structures originating from the Finnish Late Iron Age and present in the Lieto Ristinpelto shawl, apron, and diagonally plaited and tablet-woven bands indicate that locally spun wool yarns were used in these textiles. These were dyed with imported dyes, which may have been used alongside local colorants. The less common textile types, such as the orange tabby and twill, suggest that these madder dyed fabrics were imported. Woad and dyer's madder, along with weld, sawwort, and dyer's chamomile were widely used in Medieval Europe. These dye plants were not cultivated in Medieval Finland, but were already being traded during the late 12th and 13th century.

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Ethics: In this study: human bone was used for ^{14}C dating. It was treated with respect, although according to Finland's Antiquities Act, all materials over 100 years old (including human remains) are seen as objects.

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