

Article

Textile Dyes from Gokstad Viking Ship's Grave

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Abstract: The grave from Gokstad in Norway, dating to ca 900 AD, is one of the best-preserved Viking Age ship graves in the world. The grave mound contained a variety of goods along with human remains, buried in a Viking ship. Several textiles, including embroideries and shreds of what might have been the ship's tent, were also found. The colors of the textile fragments are now severely faded, but the high quality of the embroidery made of gold and silk threads is still apparent. The style of the embroidery is exceptional, having no equivalents in other Scandinavian graves. The analyses by HPLC coupled with both diode array and mass spectrometric detectors revealed that the striped "tent" cloth as well as the silk thread used for the embroidery were originally dyed with anthraquinones of plant origin (alizarin, purpurin, pseudopurpurin, and anthragallol), markers of madder-type dyestuffs.

Keywords: Viking Age; dyestuff; textiles; HPLC-DAD-MS



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1. Introduction

The production of natural dyes for textiles was an important economic factor in the early Middle Ages, and a number of written sources mention the production of madder for sale [1].

The period between ca AD 750–1050 is called the Viking Age in Scandinavia. This was a time when people from Norway, Sweden, and Denmark first began travelling to other parts of the world on a large scale and established themselves as a political factor in Europe. Profound social and political change took place in that period, when old religious ideas gave way to new ones and when the Scandinavian countries gradually became unified kingdoms [2]. The impact of long-distance trade and the establishment of urban trading settlements played an important role, bringing the Scandinavians into contact with foreign cultures all over the old world. New trends in arts and crafts fueled the expansion of long-distant trades, including textiles and dyestuffs [3–5].

Given the expansion of the Scandinavian cultural and commercial routes and possible degradation processes that have affected the textiles, it is not easy to predict which could be the natural dyes originally used in the samples retrieved from archaeological sites. The best-preserved textiles from the Viking Age come mainly from bogs or mound contexts. The textiles found in the bogs constitute one of the largest and best-preserved collections of textiles in existence [6]. The chemical study of these fabrics has permitted the identification of a narrow range of natural dyes used in these times. Plants containing luteolin, indigotin, and alizarin were determined in textiles from Søgårds Mose II and Skærso [6]. These components of natural colorants were also frequently reported in the studies performed by Walton P. Rogers on a wide number of textile samples from the Viking Age, along with insect dyes and lichen purples [7–9]. The earliest use of indigotin in Scandinavia dates back to Rebild, Denmark, dated between the 4th and the 3rd century BC, while the earliest evidence of the use of alizarin-containing madder dye in Scandinavia is dated

to the 1st century BC, and was assessed in the textiles collection from Skærsø [10]. The Scandinavian finds indicate that madder became a common dye source in Scandinavia during the Migration period (AD 400–520/540) [11].

Another source of well-preserved textiles are mounds. Some people in the uppermost social strata of society were at this time buried in ships, which were then placed in a large burial mound. The Oseberg ship, on the board of which the bodies of two women with very rich grave goods were found, is one of these. A rich array of textiles, both woolen and silken, designed for a range of uses, were unearthed within this discovery. A very important ship grave, dating to around 900 AD, was also found in Gokstad, Norway [12]. Various textiles from this grave were in good preservation conditions and included an outstanding embroidery in silk and gold (see Figure 1). The same technique has been used in embroideries found in other Scandinavian graves from this period, but the patterns found in these graves are very different [13]. The embroidery from Gokstad is formed in a flower pattern by using a combination of a gold-thread with a silk-core and threads made of pure silk. Traces of a red color are still visible on the thread used to stitch down the gold lamella. When studying the flower pattern in a stereo loupe, it looked like a red and a golden color have been used in combination to form a polychrome pattern [14]. Remnants of a ship's tent were also found in this grave [15–17]. The remnants consist of some 150 fragments of coarse 2/2 twill textiles made of wool, intervened with fragments of rope. Some of the textile fragments bear traces of red color, while others look undyed to the naked eye [14].



Figure 1. Photographs of the embroidery in silk and gold found in the ship grave in Gokstad, Norway. Picture by Ellen C. Holte, Museum of Cultural History, UiO.

The most used method for the analysis of dyes in archaeological artefacts is liquid chromatography (HPLC) with different detectors, which, after a suitable sample treatment, allows the separation of the colored molecules and the identification of the original dyes used. The most common setup is HPLC with spectrophotometric detectors, in particular, diode-array (DAD), because it allows the reliable identification of several dyestuffs by comparison with the profiles of reference materials [18]. In the past 15 years, HPLC coupled with mass spectrometry (MS) has been successfully used for dyes analyses, providing a higher sensitivity than HPLC-DAD, which is particularly useful in the analysis of archaeological textiles, where the sample is usually small in size and contains low concentrations of time-altered colorants (see, in general, [19–21]). HPLC-MS also allows, thanks to the acquisition of tandem mass spectra, us to identify unknown species, thus providing information

on dyes not available as reference materials [22–24]. Different reversed phase analytical columns have been employed for dyes analysis with mass spectrometric detection, differing in the length of the hydrophobic chains (C8 or C18), particle size (also capillary columns were employed), and end capping, or even presenting polar embedding, such as RP-amide columns [23,25]. Recently, two-dimensional liquid chromatography was also employed successfully [26].

One of the most important steps in dye analysis is the extraction/separation of the analytes from the sample. The primary objective of the sample treatment is to find the conditions in which the maximum extraction yield and minimum alteration of the molecular profile are achieved, as reviewed in [27,28]. Most extraction methods are thus based on complexation with organic acids such as formic, oxalic, or acetic, alone or combined with EDTA, where mild extraction takes place [29,30]. Various organic solvents have also been applied for the extraction of less hydrophilic compounds, e.g., dimethyl sulfoxide (DMSO), dimethylformamide (DMF), and pyridine [31]. Finally, whenever the mild extraction does not provide any result, harsher hydrolysis treatments may be applied, which were quite diffused in the past, but contraindicated for labile colorants and/or materials unstable in acidic conditions [20,31,32].

This study presents the results obtained for a set of textile samples collected from the Gokstad Viking ship's grave. The identification of the coloring materials was achieved through the application of three increasingly harsh extraction procedures, based on dimethyl sulfoxide, EDTA with DMF extraction of the dyestuff from the yarns, followed by a hydrolysis in acidic condition of the residue; the analysis of the extract was performed with high performance liquid chromatography (HPLC) with diode array detection and subsequently confirmed with HPLC coupled with a high resolution mass spectrometric detector (ESI-Q-ToF). The study led to the identification of the molecular markers of a relevant dyestuff used during the Viking Age.

2. Materials and Methods

2.1. Samples

Samples of colored textiles were subjected to specific procedures to extract the characteristic markers of the materials used during dyeing. The extraction procedures applied are specific for the target molecular markers for each sample and are described in detail in Section 2.2. Table 1 shows the samples information, the extraction procedures chosen, and the instrumental methods applied.

Table 1. Description of the samples.

Sample	Color	N° in Gokstad Collection	Description	Procedure
S1	Red/brown	C10389/37C	Ship's tent	EDTA/DMF and HCl/MeOH + AcOEt extraction
S2	Yellow (white?)	C10389/37C	Ship's tent	DMSO and HCl/MeOH + AcOEt extraction
S3	Red (red silk?)	C10459/b	Red thread silk embroidery	EDTA/DMF and HCl/MeOH + AcOEt on the residue (both fractions injected together)
S4	Red (golden silk?)	C10459/b	Golden thread silk embroidery	EDTA/DMF and HCl/MeOH + AcOEt on the residue (both fractions injected together)

2.2. Analytical Procedures

Three analytical procedures were applied to extract the colored compounds present in the textiles studied. The conditions of each procedure applied are reported below.

2.2.1. EDTA/DMF Procedure

A mild extraction by dimethylformamide (DMF) and 0.1% Na₂EDTA 1:1 (*v/v*) was first applied. Then, 200 µL of mixture solution were added to the sample; extraction was performed at 60 °C for 60 min in ultrasonic bath. The supernatant was filtered with PTFE filter (0.45 µm) and injected in the chromatographic system (20 µL). The procedure

is particularly useful to detect the presence of labile components and in case of fragile materials and degraded textiles [33].

2.2.2. DMSO Extraction

A specific extraction by dimethyl sulfoxide (DMSO) to maximize the recovery of indigoids, and in particular, of photo-labile brominated indigoids, was applied to the yellow faded sample S2, since no hints on the original color could be drawn. The procedure consisted of an extraction assisted by ultrasounds at 60 °C for 5 min followed by filtration with PTFE syringe filter (0.45 µm). The sample was kept in the dark for 5 min and then injected into the chromatographic system.

2.2.3. HCl/MeOH Procedure

The extraction solution was a 30:1 (*v/v*) mixture of hydrochloric acid (HCl) in methanol (MeOH). A total of 300 µL of MeOH/HCl solution were added to the sample and the extraction was performed in an ultrasonic bath for 60 min at 60 °C, followed by liquid-liquid extraction with ethyl acetate (3 × 200 µL). The extract was dried under a gentle stream of nitrogen and dissolved in 200 µL of acetonitrile (ACN) before injection into the chromatographic systems.

2.3. Instrumentation

2.3.1. HPLC-DAD

For the analysis of the extracts, a High Performance Liquid Chromatography system equipped with a quaternary pump with degasser PU-2089 was used, with an AS-950 autosampler and coupled to a diode array spectrophotometric detector MD-2010 (all modules were Jasco International Co., Tokyo, Japan). Data were acquired and processed using the ChromNav software. The working conditions were as follows: acquisition of the spectra in the range 200–650 nm with a resolution of 1 nm; chromatographic separation conducted at room temperature (25 °C) on an analytical column TC-C18 (2) (4.6 × 150 mm, particle size 5 µm, Agilent) with a guard column TC-C18 (2) (4.6 × 12.5 mm, particle size 5 µm, Agilent), flow rate 1 mL/min. The injection volume was 20 µL. The two eluent solutions were: A: trifluoroacetic acid (TFA 0.1% *v/v*) in bi-distilled water; B: TFA (0.1% *v/v*) in acetonitrile (ACN, HPLC grade). The elution program started at 15% B, held for 5 min, then a linear gradient to 50% B was applied in 25 min; re-equilibration time took 15 min.

2.3.2. HPLC-ESI-Q-ToF System

HPLC-ESI-Q-ToF MS analyses were carried out using a 1200 Infinity HPLC, coupled with a Jet Stream ESI interface with a Quadrupole-Time of Flight tandem mass spectrometer 6530 Infinity Q-ToF detector (all modules were Agilent Technologies, Santa Clara, CA, USA). The ESI operating conditions were: drying gas (N₂, purity >98%): 350 °C and 10 L/min; capillary voltage 4.5 KV; nebulizer gas 35 psig; sheath gas (N₂, purity >98%): 375 °C and 11 L/min. The nozzle, skimmer, and octapole were set at 1000 V, 65 V, and 750 V, respectively. High resolution MS and MS/MS spectra were acquired in negative mode in the range 100–1000 *m/z* with a scan rate of 1.04 spectra/sec, employing the AutoMS/MS acquisition mode (1 precursor per cycle, CID voltage for tandem mass spectra 30 V, collision gas N₂, purity 99.999%, FWHM (Full Width Half Maximum) of quadrupole mass bandpass used during MS/MS precursor isolation 4 *m/z*). MassHunter[®] Workstation Software (B.04.00) was used to carry out mass spectrometer control, data acquisition, and data analysis. The mass spectrometer was calibrated daily using Agilent tuning mix HP0321. An Agilent Zorbax Extend-C18 column (2.1 × 30 mm, particle size 1.8 µm) with precolumn Extend-C18 column (2.1 × 12.5 mm, particle size 1.8 µm) was used for the chromatographic separation. The injection volume was 20 µL and the flow rate was 0.2 mL/min.

The two eluent solutions were: A: trifluoroacetic acid (FA, 0.1% *v/v*) in bi-distilled water; B: FA (0.1% *v/v*) in acetonitrile (ACN, HPLC grade). The elution program started at

15% B, held for 1 min, then a linear gradient to 50% B was applied in 5 min, then to 70% B in 2 min and to 90%B in 7 min; 100% was reached after further 5 min; re-equilibration time took 5 min.

3. Results and Discussion

HPLC/DAD and HPLC-ESI-Q-ToF MS analyses were performed in order to determine the dyestuff used in the Viking Age textiles from the Gokstad ship's grave.

The chromatograms obtained from the extracts after DMSO extraction did not show any relevant peaks. The extracts obtained after EDTA/DMF procedure gave satisfactory results for sample S1, providing the highest peaks in HPLC-ESI-Q-ToF MS analysis. Only the harsh extraction based on hydrolysis followed by extraction in ethylacetate provided results for sample S2, S3, and S4, and was also successfully applied to S1.

The three samples S1, S3, and S4 showed (Figures 2, 4 and 5) a very similar composition, consisting of the presence of anthraquinones typical of madder-type dyestuffs.

In detail, Figure 2 shows the Extracted Ion Chromatograms for the HPLC-ESI-Q-ToF MS chromatograms of the EDTA/DMF extract of sample S1 from the ship's tent, where anthragallol, alizarin, munjistin, purpurin, and pseudopurpurin were detected. The detected anthraquinones are typical of a madder-dye source. The overall molecular profile was not typical of a specific dye source, although the highest content in alizarin respect to the other components may point to *Rubia tinctorum* as the original raw material. The good results obtained by the mild extraction might suggest that a relatively high amount of dye survived in the sample, or that a different recipe was used to obtain the desired color, with respect to the other samples. For comparison, the inset presents the results obtained by HPLC-DAD (shown at 254 nm) relative to sample S1 after hydrolysis followed by extraction in ethylacetate, which provided the highest peaks in the UV-Vis range.

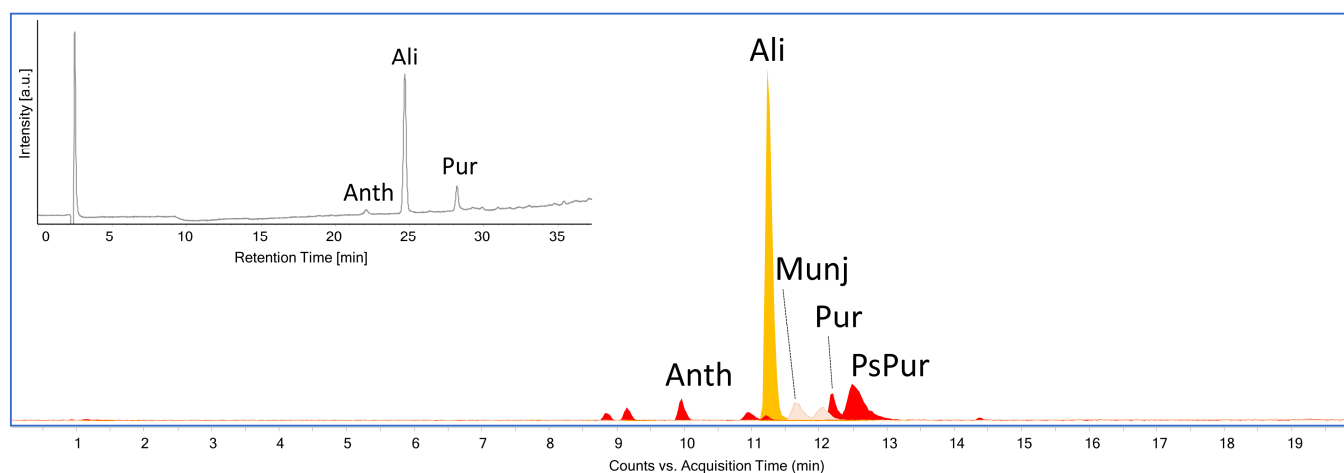


Figure 2. EIC HPLC-ESI-Q-ToF MS chromatograms of the EDTA/DMF extract of sample S1, acquired in negative mode; where Anth is anthragallol (EIC— m/z 255.029), Ali is alizarin (EIC— m/z 239.031), Munj is munjistin (EIC— m/z 283.002), Pur is purpurin (EIC— m/z 255.029), and PsPur is pseudopurpurin (EIC— m/z 299.019). In the inset, the HPLC-DAD chromatogram at 254 nm relative to sample S1 extract after HCl/MeOH + AcOEt is presented for comparison.

The second sample collected from the ship's tent (S2, Figure 3), characterized by a yellow/white color at the time of analysis, showed the presence of traces of alizarin and purpurin (only detected by HPLC-ESI-Q-ToF MS, thanks to its superior sensitivity with respect to HPLC-DAD), pointing again to the use of a madder-based dye.

Samples S3 (Figure 4) and S4 (Figure 5), collected from the embroidery, contained the anthraquinones anthragallol, alizarin, purpurin, and pseudopurpurin; rubiadin was only present in S3.

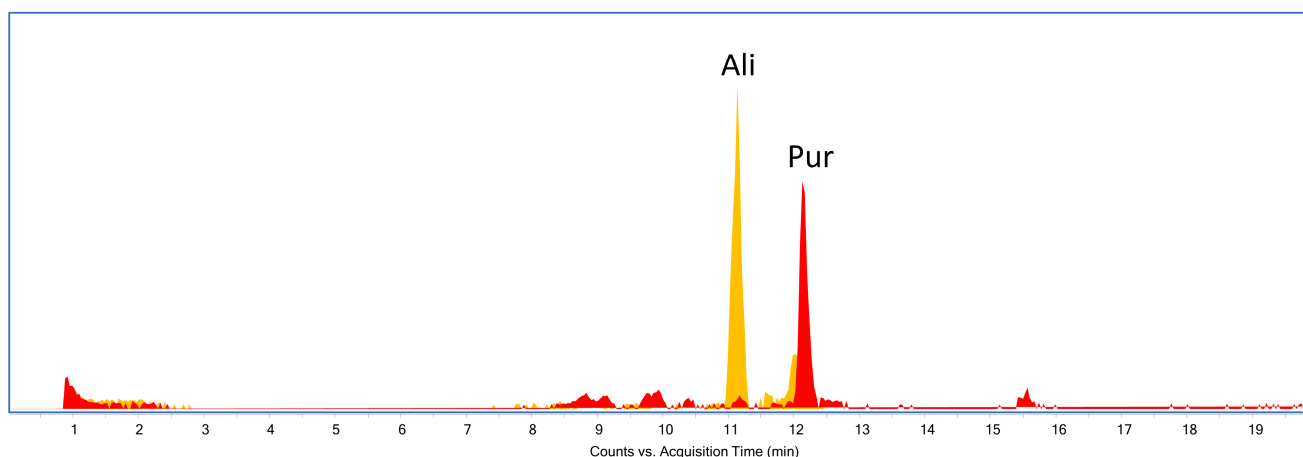


Figure 3. EIC HPLC-ESI-Q-ToF MS chromatograms of the extract of sample S2 extract after HCl/MeOH + AcOEt, acquired in negative mode; where Ali is alizarin (EIC— m/z 239.031), Pur is purpurin (EIC— m/z 255.029).

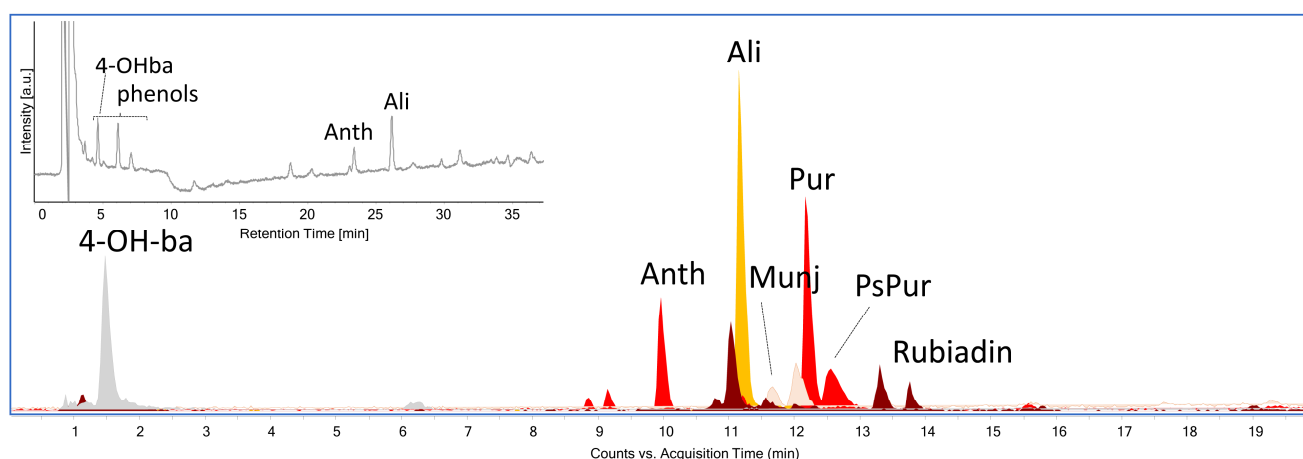


Figure 4. EIC HPLC-ESI-Q-ToF MS chromatograms of the extract of sample S3 extract after HCl/MeOH + AcOEt, acquired in negative mode; where 4-OH-ba is 4-hydroxybenzoic acid (EIC— m/z 137.024), Anth is anthragallol (EIC— m/z 255.029), Ali is alizarin (EIC— m/z 239.031), Munj is munjistin (EIC— m/z 283.002), Pur is purpurin (EIC— m/z 255.029), PsPur is pseudopurpurin (EIC— m/z 299.019), Rubiadin (EIC— m/z 253.051). In the inset, the corresponding HPLC-DAD chromatogram at 254 nm is presented for comparison.

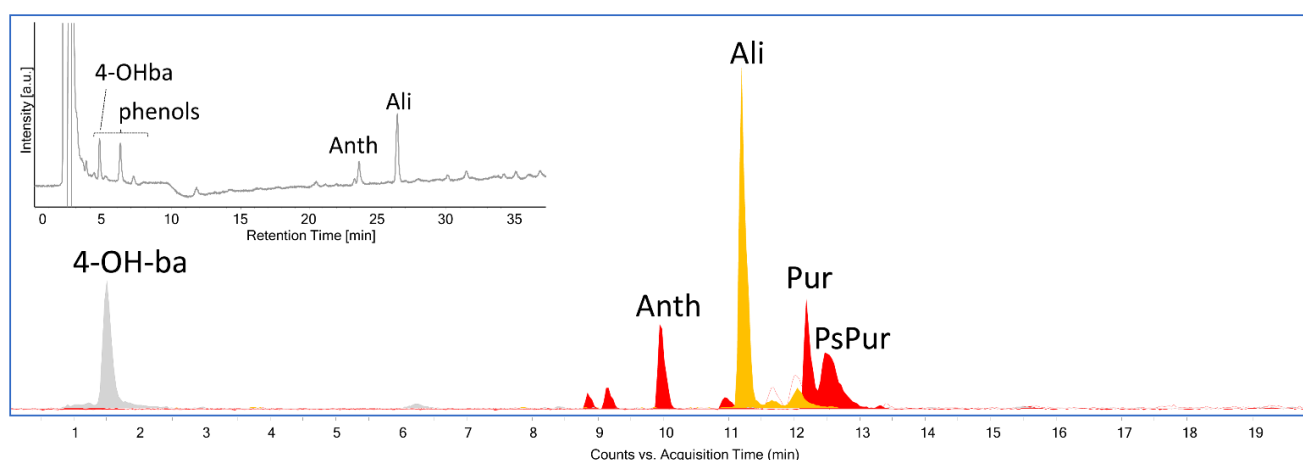


Figure 5. EIC HPLC-ESI-Q-ToF MS chromatograms of the extract of sample S4 extract after HCl/MeOH + AcOEt, acquired in negative mode; where 4-OH-ba is 4-hydroxybenzoic acid (EIC— m/z 137.024), Anth is anthragallol (EIC— m/z 255.029), Ali is alizarin (EIC— m/z 239.031), Pur is purpurin (EIC— m/z 255.029), PsPur is pseudopurpurin (EIC— m/z 299.019). In the inset, the corresponding HPLC-DAD chromatogram at 254 nm is presented for comparison.

In addition, the chromatograms feature 4-hydroxybenzoic acid, most probably due to degradation processes involving the fabric (silk) [34]. Peaks attributed to further unknown phenols based on their early retention times and maxima of absorption around 275 nm in the DAD spectra were also detected as highlighted in the insets in Figures 4 and 5, but were not confirmed by HPLC-ESI-Q-ToF MS analysis.

Table 2 summarizes the results obtained by both analytical systems (HPLC-DAD, HPLC-ESI-Q-ToF); it includes detected markers and identified dyes (associated with the presence of these compounds).

Table 2. Molecular markers detected along with identified materials in the analyzed extracts.

Sample	Molecular Markers Detected by HPLC-DAD	Molecular Markers Detected by HPLC-ESI-Q-ToF MS	Identified Materials
S1	alizarin, purpurin, anthragallol	alizarin, purpurin, pseudopurpurin, munjistin, anthragallol	madder-type dyestuff
S2	no peak identified	alizarin, purpurin	madder-type dyestuff
S3	4-hydroxybenzoic acid, phenols, anthragallol, alizarin	4-hydroxybenzoic acid, anthragallol, alizarin, purpurin, pseudopurpurin, rubiadin	madder-type dyestuff; degradation products of protein-based fibers
S4	4-hydroxybenzoic acid, phenols, anthragallol, alizarin	4-hydroxybenzoic acid, anthragallol, alizarin, purpurin, pseudopurpurin	madder-type dyestuff; degradation products of protein-based fibers

4. Conclusions

In this paper, an investigation of textile samples collected from Gokstad Viking ship's grave by high performance liquid chromatography (HPLC) with diode array (DAD) and high resolution mass spectrometric detector (ESI-Q-ToF MS) was performed. The use of ESI-Q-ToF MS as a detector was paramount in order to allow us to detect not only major but also minor compounds, and to confirm the presence of analytes in sample 2. The presence of molecular markers attributable to madder-type dyestuff, most probably from *Rubia tinctorum*, was determined in all four analyzed samples. A harsh sample treatment, followed by purification by liquid/liquid extraction with ethylacetate, was needed to retrieve significant amount of analytes from most samples (only sample S1 gave satisfactory results after treatment in mild conditions). This can be attributed to the extensive degradation of the dyestuffs or the use of specific dyeing recipes which prevented the extraction with chelating agents, requiring acidic conditions.

Madder-type dyestuffs are commonly found in graves from the Viking Age and medieval Scandinavia: a number of written sources mentioned the use and export of madder and surviving trace amounts of dyes interpreted as madder have been found in a number of Scandinavian graves dating to the Viking Age [1,5,35].

With regard to the embroidery, the two samples (S3 and S4) were collected from the threads forming a polychrome pattern [14], which looked slightly redder (S3) and more yellow (S4) respective to each other. Although both extracts had a very similar composition, consisting of alizarin, purpurin, pseudopurpurin, and anthragallol, the relative alizarin and anthragallol content with respect to all other anthraquinones is much higher in the golden sample (S4) than in the redder one (S3). Thus, the HPLC data confirmed the visual observation, being alizarin and anthragallol more yellow than the other components, featuring maxima of absorbance in the visible range around 430 and 410 nm, respectively, whereas purpurin and pseudopurpurin both have maxima of absorbance higher than 480 nm. Thus, the different amounts of the components of the dyeing material suggest that different recipes were indeed employed to obtain different shades of silk thread. Finally, protein-based fiber degradation products were identified in the silk samples, confirming the animal origin of the textiles [36].

In one sample collected from the tent (S1), madder-type markers were also identified, while in S2, characterized by a yellow/white color at the time of analysis, only traces of anthraquinones were detected related to the dyestuff. It is possible that, given the purpose

of this textile material, only a small amount of dyestuff was applied, or it severely faded during the use of the fabric. On the other hand, given the different results obtained for samples S1 and S2, the tent might have been colored in two different alternating shades.

In conclusion, the application of different extraction methods along with sensitive and selective analytical techniques allowed us to detect madder-type dyestuffs used to dye both a red-and-white/undyed striped woolen cloth used as tent or sail, and for coloring a delicate embroidery of silk and gold.

These new analyses provide a small but important new piece of information about the Viking Ship grave from Gokstad, allowing us to picture the original multicolored funerary goods.

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Conflicts of Interest: The authors declare no conflict of interest.

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