Krista Vajanto & Maarten R. van Bommel

DYED TEXTILES FROM LATE IRON AGE FINLAND

Abstract

This article discusses Late Iron Age archaeological dyed woollen textile fragments found in Finland from inhumation burials. The aim is to shed light on the used dyestuffs and local dyeing traditions. Most of the Iron Age fragments were woven 2/2 twill with Sz-plied in the warp and unplied z-spun yarns in the weft, which thus defines their local origin. One fragment from the very end of the local Late Iron Age was woven using plain weave and half basket weave as well as dyed with non-local dyestuffs, suggesting an imported textile. The indigoid dye was present in most of the fragments, but anthraquinones alizarin and purpurin were also found. Several unidentified red and orange components, presumably anthraquinones, possibly indicate the use of an unknown and local dye resource.

Keywords: Late Iron Age, archaeological textiles, natural dyes, dye analyses, fibre analysis, dye references, indigoids, anthraquinones, apigenin, luteolin

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INTRODUCTION

The aim of this study is to shed light on the source and character of the dyes in the found artefacts and to trace local and imported dyestuffs in the Finnish archaeological material from the Late Iron Age (AD 800–1150/1300). Altogether 12 textile fragments with a clearly visible shade of colour were selected from five inhumation cemeteries (Fig. 1; Table 1). The samples were analysed using *High performance liquid chromatography* (HPLC) and a recently developed *Ultra High performance liquid chromatography* (UHPLC) method, both coupled to *Photo diode array detection* (PDA).

Earlier Finnish research has been done mainly thorough visual analysis to understand the structures and functions of the different textile materials to produce costume reconstructions (Lehtosalo-Hilander et al. 1982; Lehtosalo-Hilander 2001; Riikonen 2006). Fibre analysis has been done to some extent on Iron Age and Early Medieval material (Kirjavainen 2005a&b; Kirjavainen & Riikonen 2005; 2007; Vajanto 2013a). Less than two dozen fragments from the textile rich inhumation cemeteries of Luistari in Eura, Kaarina Kirkkomäki in Turku, Tuukkala in Mikkeli and Yliskylä in Perniö have been under dye analysis (Wikström et. al. 1972; Kirjavainen & Riikonen 2005; 2007; Vajanto 2014a). Since most of these fragments were analysed using *Thin layer chromatography* (TLC) which needed large sample sizes, this protective attitude is understandable: before the development of the HPLC analysis the sample sizes were relatively large and destroyed a significant part of the researched fragment.

The Iron Age burial offerings made of bronze and copper have played an essential role in the preservation of the Finnish organic archaeological material, including textile fragments. Unlike in southern Europe, in Finland the Iron Age ended relatively late, due to the slow spread of

Christianity to the north. Therefore, the burials were still done at a relatively late date in the non-Christian tradition, i.e. with grave goods. Burial habits also played an important role: during the Late Iron Age the inhumation burials become predominant while the earlier cremation burials become less frequent (Lehtosalo-Hilander 1984a: 279–84).

TEXTILE MATERIALS

Iron Age fragments from inhumation burials

The Late Iron Age textiles are mainly of wool, because the acidic soil has caused more intensive degradation of the plant fibre textiles. The men were buried with their armoury like spears and swords, while women were interred with their tool (spindles, sickles and shears, knives) and jewellery. The female costumes consisted of a dress or a peplos dress, a square cloak, an apron, leg wraps and an undergarment. Less is known about the men's garments, because the male burials consisted of smaller amount of protective bronze (Riikonen 2011a: 209). Probably the men wore knee pants, leg wraps, a square cloak and shirt or tunic (Lehtosalo-Hilander 2001: 77–81; Tomanterä 2006: 45–6).

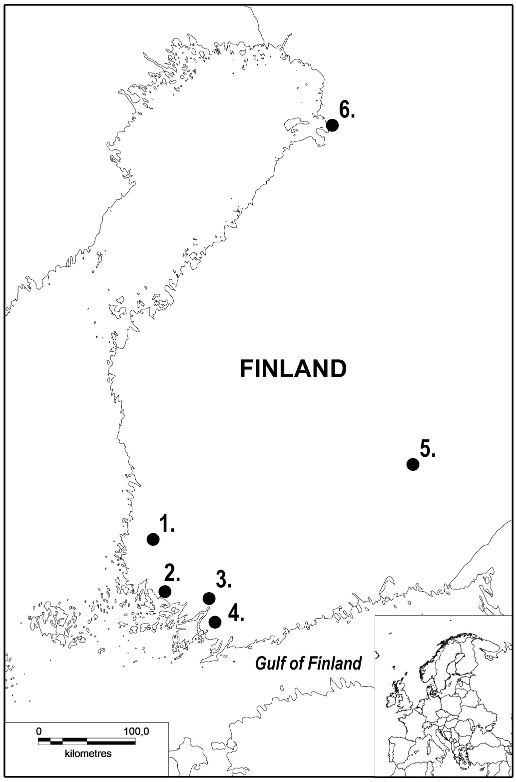

Fig. 1. Sites mentioned in the text: 1. Eura Luistari, 2. Turku Kaarina Kirkkomäki, 3. Halikko Rikala, 4. Perniö Yliskylä, 5. Mikkeli Tuukkala, 6. Oulu Cathedral. Illustration: K. Vajanto.

Cemetery of Luistari in Eura

Grave 95 of the inhumation cemetery of Luistari in Eura was excavated in 1969. This female burial contained round shoulder brooches, bronze chest chains and bronze finger rings. The forms of the finds date the burial to the Viking Age, i.e. AD 800–1050 according to local chronology (Kivikoski 1973; Lehtosalo-Hilander 1982: 292, 295, 402–3). Additional finds were a clay pot, two iron knives (one at the waist, one on the neck), bronze spirals from an apron and remains of fur (Lehtosalo-Hilander 1982: 111–3).

The female had two bronze arm rings in both hands, which had preserved the studied fragments KM 18000:2071 (1.5 × 2.5 cm) and KM 18000:2084 (7 × 10 cm). These might have originated from a single textile item due to the strong similarities in the structure. The Sz/z yarns have been spun of shiny, fine-looking wool. The thread count per centimetre is 10/10. The textile is 2/2 twill which is a very common textile structure amongst the Finnish Late Iron Age material. In the well-researched Luistari grave 56 the woollen undergarment was woven in tabby (Lehtosalo-Hilander et al. 1982: 25), but here the twill suggests that the fragments are not from an undergarment. Indeed, these fragments might be of a brooch fastened peplos-typed dress, which has often been found in prehistoric Finnish burials.

Cemetery of Yliskylä in Perniö

Some of the best preserved fragments of a Late Iron Age female costume were excavated from the inhumation cemetery of Yliskylä in Perniö in 1893 (Appelgren-Kivalo 1907: 28–58). There are remains of square cloaks, dresses and aprons. All the material is very fragmentary, but some pieces are exceptionally large: a preserved apron has a few centimetres wide, bronze spiral ornamented selvage with a length of almost 70 centimetres; a square cloak has bronze ornamented edges with a length of c 100 centimetres (Appelgren-Kivalo

No.	Find	Function	Structure	Yarns	Date (AD)
1.	Eura Luistari KM 18 000:2071	Peplos dress? Cloak?	2/2 twill	Sz/z	10–11th century
2.	Eura Luistari KM 18 000:2084	Peplos dress? Cloak?	2/2 twill	Sz/z	10–11th century
3.	Halikko Rikala KM 12 690:168	Cloak?	2/2 twill	Sz/z	11–12th century
4.	Mikkeli Tuukkala KM 9770:5	Cloak?	1/1 tabby + half basket weave	s/z	13/14th century
5.	Perniö Yliskylä KM 2912:53	Dress	2/2 twill	Sz/z	11th century
6.	Turku Kaarina Kirkkomäki KM 12687:H1:20	Cloak	2/2 twill	Sz/z	11th century
7.	Turku Kaarina Kirkkomäki KM 27025:H27:168	Cloak II	2/2 twill	Sz/z	11th century
8.	Turku Kaarina Kirkkomäki KM 27025:H27:203	Dress II	2/2 twill	Sz/z	11th century
9.	Turku Kaarina Kirkkomäki KM 27025:H27:230b	Belt of apron I	Finger-woven braid	z	11th century
10.	Turku Kaarina Kirkkomäki KM 27025:H27:235	Binding band of leg wraps	Braided band	z	11th century
11.	Turku Kaarina Kirkkomäki KM 27025:H27:237	Belt	Tablet woven band	Sz	11th century
12.	Turku Kaarina Kirkkomäki KM 27025:H27:239	Belt of apron II	Finger-woven braid	Sz	11th century

Table 1. Overview of the samples.

1907: plates VIII&XI).

In 1925, a reconstruction of the Perniö costume was made based on the Yliskylä materials, but by combining information from several graves. The first attempt to analyse the dyes was done in 1972 at the VTT Technical Research Centre Finland. The level of dye analysis was not very evolved at that time but indigotin was found in two fragments from graves 2 and 6 (Wikström et al. 1972).

Fragment KM 2912:53 is from grave 1 and probably belongs to a dress, which has been visually estimated to be reddish (Lehtosalo-Hilander 1984b). This fragment is 2/2 twill and woven using Sz/z yarns. The dress was made of a square fabric with shoulder seams and sewn tubular form; there is a seam in front of the dress (Appelgren-Kivalo 1907: 33–4, plate VII). The wool is shiny and the yarns very evenly spun.

Cemetery of Kaarina Kirkkomäki in Turku

The Kirkkomäki inhumation cemetery in Turku (formerly part of Kaarina parish) contains several inhumation burials. Grave 1 was excavated in 1950 (Riikonen 1990; 2003; 2006) and is dated to the early 11th century AD. This burial contained several blue textiles, including a peplos dress, an apron and a headdress (Riikonen 1990). In 2004, a sample from the square cloak of grave 1 was analysed. The warp yarn contained red tannins (Kirjavainen & Riikonen 2005: 41; Walton 2004).

Kirkkomäki grave 27 is somewhat younger than grave 1 (Kirjavainen & Riikonen 2005; 2007; Asplund & Riikonen 2007: 32), both made for females. Grave 27 was excavated in 1991, but mainly investigated in a laboratory after the field work and after being frozen for several years (Riikonen 2011b). The remains of an undergarment, two dresses, two square cloaks, two aprons, a headdress and leg wraps were found (Kirjavainen & Riikonen 2005; 2007). There was a tablet woven band at the waist area, bearing bronze bear tooth imitating pendants (Asplund 2005: 15; Riikonen 2005: 50–2). The pattern of this belt is very complicated and has been woven with blue and reddish yarns (Penna-Haverinen 2009a&b). The aprons had finger-woven, diagonal braided sashes in the upper corners (Kirjavainen & Riikonen 2007: 136, 139).

The dyestuffs of some Kirkkomäki grave 27 fragments were analysed using TLC (Walton 2001a). Indigotin and red tannins were found in the square cloak II. Dress II and square cloak II contained red-brown tannins. The dark warp yarns of the finger-woven sashes of aprons I and II contained red brown tannins, while red tannin and a trace of alizarin was found in the braided band of the leg wraps (Walton 2001a; Kirjavainen & Riikonen 2007: 135–7). The light coloured warp of the finger-woven sash of apron I contained indigotin. Two samples of the tablet woven belt both contained indigotin, but in different amounts (Penna-Haverinen 2009a: 66). The source of red tannins remained undefined, and because of this, the left over material of TLC was taken to re-analysis with the UHPLC.

Cemetery of Rikala in Halikko

Fragment KM 12690:168 (3.5 x 4.5 cm) was found in female's grave 12 of the inhumation cemetery of Rikala in Halikko. The site is one of the richest Late Iron Age inhumation cemeteries in Finland. It was excavated in 1950–51 and 1953, but already before that by non-archaeologists (Mäntylä-Asplund 2011: 223). Because of this activity, the original burial context of the grave 12 was disturbed and the original position of this fragment is unknown.

The fragment is 2/2 twill and woven using 10/8 yarns per centimetre. It might be of a square cloak (Riikonen 2007: 19–20). There is an uneven striped structure created by two different weft yarns and the phenomenon is visible also in X-ray pictures (Riikonen 2007: 19). The light-coloured weft yarns (weft 1, sample 3c) have a smaller spin angle and are half of the diameter of the thicker and more tightly spun, darker weft yarns (weft 2, sample 3d). Two samples were taken from warp yarns from two different places.

Cemetery of Tuukkala in Mikkeli

The cemetery of Tuukkala in Mikkeli was found in 1886 (Heikel 1886). This area with an estimated 90–100 graves has been excavated several times. The recent research dates the site to the transition of the 13th and 14th centuries AD (Mikkola 2009: 184). According to the local chronology this was the transition era of Late Iron Age and Early Medieval Period, but the burials were still done in the Iron Age style with grave offerings. Fragment KM 9770:5 (c 30 x 30) is from a male grave, found in 1933. Additional finds of the burial are a fragment made with the *nålbinding* technique, a knife, a fire steel striker, a belt with a bronze buckle and two other wool fabrics. The textile fragment has four stripes in the middle area woven with reddish, blue and white wefts (Fig. 2). There are no traces of felting.

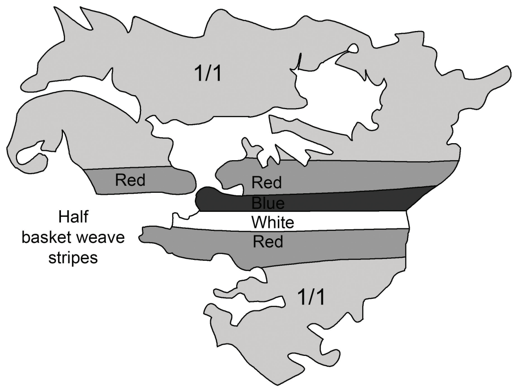

Fig. 2. Schematic map on Mikkeli Tuukkala fragment KM 9770:5. Illustration: K. Vajanto.

The striped area has been woven with half basket weave (extended plain weave) using thick and fluffy, z-twisted yarns. Otherwise the fragment is 1/1 plain weave (tabby), which has been woven with tightly spun s-yarns in warp and z-yarns in weft. The thread count is 10/10 per centimetre. The structure can be woven using a straight draft on four shafts; in which the heddling is done in the order of 1+2+3+4. When weaving, the shafts 1+3 and 2+4 are lifted in turn for a 1/1 plain weave. For the half basket weave, the shafts 1+2 and 3+4 are lifted in turn (Robinson & Marks 1973: 91–3). The striped Tuukkala fragment has been suggested to be imported (Riikonen 2007: 20) and indeed it has parallels in English 14th-century textiles (Crowfoot et al. 2006: 52–5, 64–5, plates 6–8: ray textiles) as well as in finds of Novgorod in Russia (Nahlik 1963: 232).

DYESTUFF AND FIBRE ANALYSES

Basics on the natural dyes

For natural dyes, three different dyeing procedures can be used depending on the dye molecule itself. The easiest way is the *direct dyeing* method that is used when dyeing with tannins, safflower (*Carthamus tinctorius* L.) and lichens (Cardon 2007: 4). In this method, the dye is extracted from the dye plant in (warm) water, filtered to remove the plant material and next the textile fibres or yarns or a whole textile is brought into the dye bath that is often boiled during the dyeing. The dyeing occurs via hydrophobic interaction, in which the colour producing chromophores prefer to seek the fibres rather than stay in the dye bath.

The most used dyeing method is *mordant dyeing.* In this process, the textile material is first inserted in a bath with metal salts like aluminium, iron, tin, chrome and copper (Dean 1999: 38–9). Then the metal-salt-mordanted textile material is put into the dye bath that contains dye compounds from plants. The dye compounds form a bond with the metal, which serves as a bridge between the textile fibre and the dye. The wash and light fastness of material dyed by this method is generally much better compared to direct dyes; the majority of the natural dyes are mordant dyes (Cardon 2007: 4–6).

The most complicated dyeing technique is *vat dyeing*. This is done with indigo plants and Tyrian purple. The main components of these dyes are not soluble in water; therefore, they have to be reduced into their water soluble leuco-form. Traditionally this was done using fermentation but nowadays reducing chemicals are used. The textile material is inserted into the dye bath after this reduction step and the leuco dyes are bound to the textile fibres. Once this textile is exposed to air, the dye is oxidised and forms a very stable dye bound to the textile fibre (Schweppe 1993: 282–318; Cardon 2007: 4, 337–8, 559–662).

Methods of dye analyses

Accurate dye analysis can be only done by taking samples from the objects of interest, thereby disturbing their integrity. *Thin layer chromatography* (TLC) is still used sometimes for dye analyses because its cost effective and relatively easy protocol. The disadvantage of this method is that it needs large samples. *High performance liquid chromatography* (HPLC) analysis coupled with *Photo diode array detection* (PDA) is a very strong identification tool that requires only a very small sample, for example a few millimetres of yarn (Fig. 3). Therefore, HPLC could be described as a micro-destructive method. Recently, an advanced technique, *Ultra high performance liquid chromatography* (UHPLC-PDA), was introduced for the analysis of natural colorants. Due to an improved separation of the components and a better resolution of the method, UHPLC-PDA is more sensitive than HPLC-PDA for which the sample size could be reduced even further (Serrano et al. 2013).

The dyestuffs of samples 1–4 were analysed using HPLC-PDA, but samples 5–12 were analysed with UHPLC-PDA. The references were analysed in the same way as the archaeological samples (Proano Gaibor 2011). Prior to analysis, the organic colorants were extracted from the textile fibres and brought into solution using a two-step extraction procedure. In the first step, an organic solvent, dimethyl sulfoxide (DMSO), was used to extract vat dyes and direct dyes by heating the sample at 80 °C for 10 minutes. Next, the DMSO fraction was separated from the sample and a second extraction was performed on the remaining sample using a solution of methanolic hydrochloric acid (HCl). This sample was heated at 100 °C for 10 minutes. During this step, also the mordant dyes can be extracted from the textile fibre. After the second extraction, the HCl was removed by evaporation under a nitrogen flow and the sample was dissolved using the DMSO from the first extraction step. After careful centrifugation to remove any remaining particles, an aliquot of the sample was introduced into the HPLC or UHPLC system.

During the (U)HPLC analysis, the organic colorants were separated from each other and their ultraviolet-visible spectra (UV-VIS) were recorded by PDA. Compounds were identified

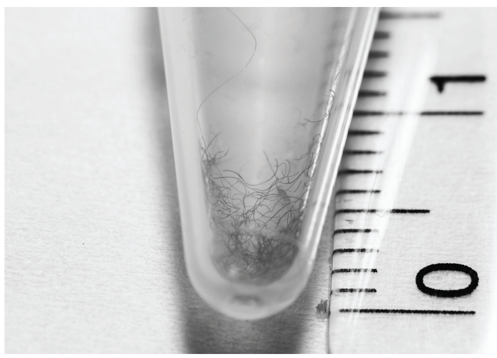

Fig. 3. Sample size of Mikkeli Tuukkala fragment KM 9770:5 red yarn was quite small. Nevertheless, several different dye compounds were detected. Photo: K. Vajanto.

by comparison of these UV-VIS spectra and their respective retention times with known reference material, which data is stored in a (U) HPLC-library. Unfortunately, identification is not always possible, due to the low concentration in the sample or a lack of reference material. However, from the UV-VIS spectra the colour of the unknown compound can be deduced unless the unknown compound is a degradation product, which has undergone a change of colour (Hofmann-de-Keijzer et al. 2013: 137–40).

Dyed references

Due to the northern latitude, the vegetation in Finland differs from that in the southern areas of Europe and the dye plant sources have been different. Thus, several references were dyed using the local and traditionally known dye plants (Vajanto 2014b). The recipes were found in Finnish dyeing books (Kontturi 1945; Hassi 1978) as well as from folklore collected by the Finnish Literature Society. According to these, the red was dyed with bedstraws or using fermented tannins. The yellow shades came from local wild plants. The blue was either woad (*Isatis tinctoria* L.) or anthocyanin dye produced from purple flowers with clubmoss (*Lycopodium* species) as a mordant (Vajanto 2013b), a recipe that was in earlier research (Hirviluoto 1999) assumed to describe woad dyeing.

Most of the references were pre-mordanted with alum and cream of tartar and boiled with plant material: the roots of northern bedstraw (*Galium boreale* L.) and Lady's bedstraw (*Galium verum* L.), the bark of common alder (*Alnus glutinosa* L.), branches of crowberry (*Empetrum nigra* L.), bog rosemary (*Rhododendron tomentosum*) and heather (*Calluna vulgaris* L.), spring leaves of silver birch (*Betula bendula* L.) and mushroom surprise webcap (C*ortinarius semisanguineus* Gillet). A bladder wrack (*Fucus vesiculosus* L.) bath was first fermented, and then the yarn was added and boiled with added alum. The rock tripe (*Lasallia pustulata* L.) reference was dyed in a fermented urine vat. Fermentation baths were made of water, wood ash lye and roots of common tormentil (*Potentilla erecta* L.) or the inner bark of silver birch (*Betula pendula* L.) or the bark of alder buckthorn (*Rhamnus frangula* L.) (Vajanto 2013c; 2014b).

Wool types

Wool yarn can contain fibres from underwool, hairs from the outer coat or be homogenous depending on many parameters like the sheep breed, sheep's age and sex. Wool can be categorised by the often-used Ryder's grouping, which has been seen to follow the evolution of sheep and fleece. The wool types are named as *Mouflon type, Hairy* (H), *Hairy medium* (HM), *Generalised medium* (GM), *Medium* (M), *Fine/Generalised Medium* (F/GM), *Semi Fine* (SF) and *Fine* (F) (Ryder 1974; 1984; 1987; Walton-Rogers 2004: 83). This is a practical tool to see similarities and differences amongst the studied wool material. However, the Ryder's grouping does not take into the consideration the spinner's selections set for the yarn quality. This activity has probably changed the original fleece's range of fibre diameter in the finished yarn. A recently presented alternative classification is based on the textile industry (Rast-Eicher 2008; Rast-Eicher & Bender Jørgensen 2013: 1226).

The Ryder's grouping is made by measuring the diameter of 100 single fibres (Ryder 2000: 4). Samples with a length of 1–2 millimetres were cut from yarns and placed on a slide with a drop of distilled water. The fibre diameters were measured with a Leica DMLS (DFC 420) transmitted light microscope using the Leica LAS Core V 3.6 program. The fibres' medulla and pigmentation were also observed.

Most fragments were organic, but the Luistari

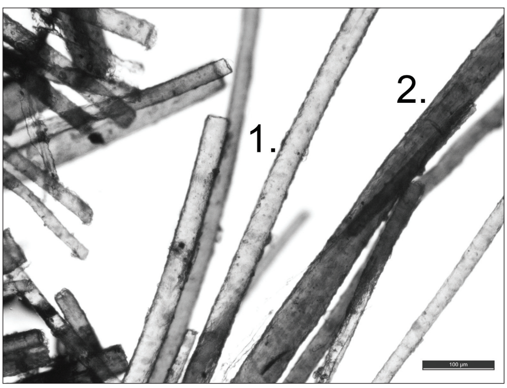

Fig. 4. Colourful, but degraded wool fibres from Eura Luistari KM 18000:2071 weft: 1. light brownish, 2. dark blue. Scale bar 100 µm. Photo: K. Vajanto.

Sample	Wool type	Count of fibres (100%)	Medullated (100%)	Pigmented (100%)	Mean (µm)	Mode (µm)	Variance	Standard deviation
1a.	HM	119	11	0	27.11	20	125	11.19
1b.	HM	87	18	0	30.45	26	167	12.92
2a.	HM/GM	114	2	0	30.7	24	96	9.81
2b.	HM	61	2	0	23.70	20	46	6.77
3a.	HM/GM	25	20	0	37.36	52	233	15.27
3b.	HM/GM	50	6	0	27.84	30	47	6.84
3c.	HM	87	3	0	27.43	20	120	10.96
3d.	HM	50	2	0	26.26	18	149	12.22
4a.	M?	5	0	0	30.8	28	16	3.97
4b.	GM/M	55	16	0	38.55	32	238	15.43
4c.	GM/M	77	0	0	27.25	26	33	5.75
5.	H?, visual analysis only							
6.–12. Discussed by Kirjavainen & Riikonen (2005; 2007): Kaarina Kirkkomäki grave 27 material contained H, HM and GM wool.								

Table 2. Wool types of the researched samples.

and Rikala samples were partly mineralised and so degraded, that proper fibres for the analysis were very difficult to find (Fig. 4). In these cases, only 50 fibres were measured. It has been pointed out that this number of measurements differs statistically very little from the recommended 100 measurements (Kirjavainen 2005a&b; Kirjavainen & Riikonen 2007: 135). In addition, the Tuukkala sample 4a was very small, only a few fibres altogether. Only some fibres were measured and the remaining fibres were sent to HPLC-PDA dye analysis. However, by visual observation, it could be seen the yarn's fibre material seemed to be very homogenous throughout. The Yliskylä sample was left out of fibre analysis due to the small sample size, but in visual observations it contained very fine underwool with some quite coarse hairs.

RESULTS

Fibres

The samples from Kirkkomäki, Luistari and Rikala contained *Hairy* and *Hairy medium* wool (Table 2), accompanied with *Generalised medium* and *Hairy medium/Generalised medium* intermediate wool type. The yarns in Tuukkala KM 9770:5 fragment contain *Medium/Generalised medium* and *Medium* wool.

Dyes

The results of the dye analyses are presented in Table 3. If possible, the warp and weft yarns were analysed separately, but the plied warps were not opened for separate analysis. The indigo dye was present in most of the samples (Table 3). This dyestuff was often found together with isatin and indirubin; the latter is a red isomer of indigotin. A trace of red purpurin dyestuff was found in the warp yarn of the Kirkkomäki sample 6b square cloak using UHPLC analysis. Sample 4a from Tuukkala contained both red dyestuffs alizarin and purpurin, but in addition, there was also a trace of indigotin present in this sample. Sample 4c from the tabby area of the Tuukkala fragment contained the yellow dyestuffs apigenin and luteolin. In addition, several unknown compounds were detected (Van Bommel 2013a&b).

DISCUSSION

Wool

Hairy, Hairy medium, *Generalised medium* and *Hairy medium/Generalised medium* intermediate wool types are predominant in the Finnish Late Iron Age material (Ryder 1978; Kirjavainen 2005a&b; Vajanto 2013a). In earlier research, the *Hairy, Hairy medium* and *Generalised medium* wool types have been defined in the Kirkkomäki grave 27 samples (Kirjavainen & Riikonen 2005: 36–7). According to these samples, the Iron Age sheep in Finland had predominantly a double coated fleece; thus exceptional fibre distributions might refer to an imported textile or a textile made for a very special purpose by carefully selected fibres. This suggests that the Tuukkala textile is imported or a special item. All the wool

No.	Sample	Indigoid dyestuffs	Red anthraquinones	Yellow and colourless components	Unknown red and orange components	Other remarks
1a.	Eura Luistari KM 18000:2071 warp	Indigotin, indirubin, isatin	–	Chrysoeriol? Unknown yellow and colourless compounds	–	–
1b.	Eura Luistari KM 18000:2071 weft	Indigotin, indirubin, isatin	–	Chrysoeriol? Unknown yellow and colourless compounds	–	–
2a.	Eura Luistari KM 18000:2084 warp	Indigotin, indirubin, isatin	–	Chrysoeriol? Unknown yellow and colourless compounds	–	–
2b.	Eura Luistari KM 18000: 2084 weft	Indigotin	–	Chrysoeriol? Unknown yellow and colourless compounds	–	–
3a.	Halikko Rikala KM 12690:168a warp 1	Indigotin (trace)	–	Woad flavonoid	–	–
3b.	Halikko Rikala KM 12690:168b warp 2	Indigotin (trace)	–	Maclurin equivalent? Unknown yellow and colourless compounds	–	–
3c.	Halikko Rikala KM 12690:168c weft 1	Indigotin (trace)	–	Unknown yellow and colourless compounds	–	–
3d.	Halikko Rikala KM 12690:168c weft 2	Indigotin (trace)	–	Unknown yellow and colourless compounds	–	–
4a.	Mikkeli Tuukkala KM 9770:5 red weft	Indigotin (trace)	Alizarin, purpurin, rubiadin, unknown anthraquinone	–	–	–
4b.	Mikkeli Tuukkala KM 9770:5 beige weft	–	–	Unknown yellow and colourless compounds	–	Possibly undyed
4c.	Mikkeli Tuukkala KM 9770:5 brown warp and weft from tabby	–	Alizarin (trace), purpurin (trace), unknown anthraquinone	Luteolin (trace), apigenin (trace), unknown flavonoid	–	–
5. (*)	Perniö Yliskylä KM 2912:53 warp	Indigotin, indirubin, isatin equivalent, isatin	–	–	Red (498 nm/ 18.22 min), red-orange	Very rich in blue dye
6a. (*)	Turku Kaarina Kirkkomäki KM 12687:H1:20 warp	Isatin (trace)	–	–	Orange (385nm/ 15.96min)	–
6b. (*)	Turku Kaarina Kirkkomäki KM 12687:H1:20 weft	Indigotin, Indirubin, Isatin (trace)	Purpurin	-	Red 468 nm/16.11 min), red-orange	Unknown red or blue components (traces)
7. (*)	Turku Kaarina Kirkkomäki KM 27025:H27:168 warp	indigotin, indirubin, isatin	–	–	Red-orange (429 nm/ 19.60min)	–
8a. (*)	Turku Kaarina Kirkkomäki KM 27025:H27:203 warp	Indigotin, indirubin (trace), isatin (trace)	–	–	Red-orange (429/19.60 min) and red	–
8b. (*)	Turku Kaarina Kirkkomäki KM 27025:H27:203 weft	Indigotin (trace), indirubin (trace), isatin	–	–	Red-oranges (429nm/ 19.60 min), reds and oranges	Unknown reddish component from woad?
9. (*)	Turku Kaarina Kirkkomäki KM 27025:H27:230b warp	Isatin?	-	Traces of unknown yellow components	Red-orange (429nm/ 19.60 min)	–
10. (*)	Turku Kaarina Kirkkomäki KM 27025:H27:235 warp	–	–	Unknown yellow components	Red-orange (429nm/ 19.60 min)	–
11. (*)	Turku Kaarina Kirkkomäki KM 27025:H27:237 warp	–	–	Unknown yellow components	Red-orange (429nm/ 19.60 min)	–
12a. (*)	Turku Kaarina Kirkkomäki KM 27025:H27:239 warp, light yarn	Indigotin, indirubin (trace), isatin	–	–	–	–
12b. (*)	Turku Kaarina Kirkkomäki KM 27025:H27:239 warp, dark yarn	Indigotin	–	–	Red-oranges	Diamond green B, unknown synthetic red

Table 3. The HPLC-PDA and UHPLC-PDA (*) results. The KM-number refers to the archaeological collections of the National Museum of Finland.

samples seemed to contain unpigmented wool. However, since all of the fibres were more or less colourful due to dyeing, defining the wool's natural colour was difficult.

Indigoid dyestuffs and their sources

Indigotin is one of most stable natural dyes and the only natural dye that can give a stable blue colour. Because of this good permanence, it is no wonder that indigotin was found in most of the analysed samples (see Table 3). The presence of indigotin, isatin and indirubin indicates the use of woad or indigo (*Indigofera tinctoria* L.), which are the two indigoid plant species mainly used in Europe. However, it is impossible to identify the particular indigoid plant via dye analysis as all indigoid plants contain the same principle components.

An especially skilled dyer has been dyeing the Yliskylä yarn, which contained a very rich amount of dyestuff (Table 3, sample 5). In the Kirkkomäki sample 8b, the ratio between indigotin and indirubin was abnormal: it differed from the sample 8a that got the normal ratio (Table 3). This might refer to two different indigo dyeing recipes. Unfortunately, it is unknown, which of many indigo dyeing recipes was used in Iron Age Finland, but the folklore survey data from the early 20th century contains knowledge of woad balls and vat dyeing method with old urine.[1] An explanation for the different ratios might be that one vat was prepared using frost bitten and thus indigo-poor woad leaves during early summer (or late autumn) and the second vat was made with indigo-rich leaves during midsummer.

In Early Modern Finland, woollen folk textiles were dyed with either woad or tropical indigo to produce evenly blue or patterned, ikat-dyed *flammu* yarns (Lehtinen & Sihvo 2005: 24–5). The striped *flammu* yarns were dyed by tying the skeins around a special wooden stick, a *lamupuu*, with indentations (Vuorela 1977: 496–7). No evidence of this craft is found in the Iron Age blue textiles. These were probably dyed either as yarns or as whole garments; the undyed parts in the plied yarns in Yliskylä and Luistari textiles suggest that.

In Finland woad grows on the southern coast as a wild plant, mostly in the maritime environment on sandy shores on bladder wrack banks (Fig. 5) and was it even cultivated in small quantities in peasant gardens (Linnilä et al. 2002: III, 63–4). The oldest Finnish archaeological seeds of woad have been found in the Early Iron Age Ketohaka 1 settlement in Salo (Aalto 1982: 141). The site was inhabited from the Pre-Roman Iron Age to the beginning of the Viking Age, i.e. 500 BC – AD 800 (Schauman-Lönnqvist et al. 1986: 91–4). However, the seeds do not indicate necessarily presence of the craft of woad dyeing. In southern Europe and amongst the Arabs woad was known as a medical and antiseptic plant (Balfour-Paul 2011: 218–9). In addition, it was used as body paint by the Gaul warriors and women (Plin. NH, XXII: 2).

Woad was cultivated in Medieval Europe in large quantities (Cardon 2007: 376–8), but the history of blue goes far back in time: blue colorant probably from woad has been found in textile materials from Neolithic Adouste cave in France and from Bronze and Iron Age salt mines in Austria (Cardon 2007: 374–5; Hofmann-de-Keijzer et al. 2013: 135–62) as well as from textiles of a Celtic princely burial of Eberdingen-Hochdorf (Banck-Burghess 2012: 145). Blue garments were known in Scandinavia already during the Early Iron Age, when the Lønne Hede girl was buried (Bender-Jørgensen & Walton 1986; Mannering et al. 2012: 110). The Swedish Högöm male burial from the Migration Period (AD 400–600) contained textiles dyed with the insect dye Polish cochineal (*Porphyrophora polonica*), weld (*Reseda luteola* L.), Dyer's madder and indigotin (Nockert 1991: 72–5). In addition, seeds of woad were found in the famous Norwegian Oseberg ship burial of Queen Åsa, dated to the early 9th century AD (Christensen et al. 1992: 222).

Tropical indigo pigment was known as a painting pigment in the Roman world already in the 1st century BC (Vitr. VII: 14). It was also known Italy in the in the 12th century AD, when it began its competition with woad as the main source of blue dye (Cardon 2007: 364). The import of the tropical indigo pigment was strictly forbidden in many countries from the 16th to the late 18th centuries AD, but the regulations were also broken (Cardon 2007: 376–7; Balfour-Paul 2011: 55–7) and eventually woad was replaced by indigo. However, there were routes from Scandinavia to the Black Sea and Byzantine Empire already during the Viking Ages. It is not impossible, that tropical indigo was a trade article

Fig. 5. Woad growing at seashore in Espoo, Finland. Photo: K. Vajanto.

already during that time at the Vikings' Eastern Route (Vajanto 2014a: 97). As an easily transportable, high-price product it might have been an ideal trade item (Peets 1998: 306).

Red anthraquinone dyes of plants

Dyer's madder contains many different anthraquinones, of which alizarin and pseudo-purpurin are the main compounds. The pseudo-purpurin is converted into purpurin during the HCl extraction, but this does not alter the identification of the dye plants. The Finnish bedstraws have mostly pseudo-purpurin and only a little alizarin (Proano Gaibor 2011), while in Dyer's madder the ratio is the opposite (Cardon 2007: 112, 127). Accordingly, the purpurin in Kirkkomäki sample 6b might be from bedstraws (Table 3). In addition to these plant sources, the mushrooms of the genus *Cortinarius* contain other antraquinones, mainly emodin and dermocybine.

According to folklore, the Finns dyed red with northern and Lady's bedstraw. The yarns were mordanted in a fermented clubmoss bath[2] or predyed with birch leaves (Linnilä et al. 2002: II, 261). All bedstraws, even smaller species, were collected to the dye bath, sometimes with roots of common tormentil.[3] However, it is possible that the folk beliefs held that the Lady's bedstraw was the most valuable. It was believed to be good for milk and cheese production as well as a remedy for female diseases, epilepsy and heart problems. The scenting yellow flowers were laid to coffins below the deceased (Linnilä et al. 2002: II, 263). Possibly the peasant Finns were aware of the Dyer's madder, since the Finnish word for bedstraw is *matara*. This is close to the English word *madder* (i.e. Dyer's madder) and its variants in several other languages (Chenciner 2000: 21). Moreover, the red dye was greatly valued and a specialised tool, *matarakokka*, a bedstraw hoe, was developed to collect the narrow bedstraw roots[4] to achieve the bright red dye from local sources.

Sample 4a from Tuukkala contained both alizarin and purpurin, suggesting the use of Dyer's madder (Table 3). The sample also contained a trace of indigotin, a possible cross-contamination of the blue stripe next to the red stripe; it could that the small blue dyed fibres have contaminated the red yarns. The ratio between alizarin and purpurin did not correspond with the ratio normally found in Dyer's madder. This could indicate the degradation of the dyestuffs or a specific dyeing technique or a dye mixture made of Dyer's madder and bedstraws. In general, alizarin and purpurin are rare dyestuffs in the Finnish Iron Age material. Previously these have been found in Luistari KM 18000:1696 *nålbinding* fragment (Vanden Berghe 2012; van Bommel 2013a). In addition, alizarin and red tannin were found in a leg wrap band from Kirkkomäki grave 27 (Kirjavainen & Riikonen 2007: 135–7; Walton 2001a).

Dyer's madder is not native in Finland, so the Tuukkala textile, or the red weft yarn or the dye was probably imported. During the Middle Ages, Dyer's madder was the predominant source of red dye in Europe, while in France it had been cultivated at least from the 9th century AD (Chenciner 2000: 44). Dyer's madder and bedstraw dyestuffs have been detected in TLC analyses in Finnish Medieval archaeological textiles of local and foreign origin (Kirjavainen 2002: 348; 2004). Probably it was especially the professional dyers in urban centres, who used the Dyer's madder and woad in dyeing (Kirjavainen

2002: 348). Overall, the use of Dyer's madder and bedstraws seem to be more typical to the Finnish Medieval Period than the Late Iron Age.

Yellow dyestuffs from weeds and shrubs

Sample 4c was from the tabby area of the Tuukkala fragment (Table 3). The dye analysis detected luteolin and apigenin, but at a low concentration level. The specific markers for dyer's broom and sawwort were not detected, but this could due to the low concentration. Since no other samples from this textile contained these compounds, it is likely that these dyestuffs were intentionally used to dye this specific yarn to yellow. Luteolin and apigenin are present as the main components in weld (*Reseda luteola* L.), dyer's broom (*Genista tinctoria* L.), sawwort (*Serratula tinctoria* L.), dandelion (*Taraxacum officinale* L.) and yarrow (*Achillea millefolium* L.) (Hofmann-de-Keijzer et al. 2013: 153). These compounds have been detected in several peat bog Scandinavian materials dated to the Early Iron Age and Migration Period (Vanden Berghe et al. 2009; 2010), but not in the Finnish material.

Weld, dyer's broom and sawwort have been traditional dye plants in southern Europe and weld was even grown there for dyers (Cardon 2007: 169–80). In Finland, the cold winters prevent the cultivation of these biennial plants, of which the harvest is collected during the second year of growth. However, recently a tiny occurrence of sawwort was found in south-western Finland. It has been speculated whether this is group of plants is recent or even of archaeophytic origin, which would mean that it might have been introduced to Finland during the prehistoric time (Alho et al. 2012).

In Finland, yellow dyes traditionally came from local wild plants that were freely available according to local rights to utilise natural resources. However, not all plants were collected for dye baths. For example, dandelion was mainly known as an editable herb and medicinal plant (Linnilä et al. 2002: II, 252). Yarrow had the same aspects, but it was also known as a source of yellow dye, especially when boiled with wood ash lye (Linnilä et al. 2002: II, 206).

Samples from Luistari contained a low concentration of a luteolin equivalent that matched with chrysoeriol (Table 3, samples 1a, 1b, 2a, 2b). This is present as a minor component in several plants, in tansy (*Tanacetum vulgare* L.) for example. However, in all these plants the luteolin and apigenin are the main components (Schweppe 1993: 322, 352). Since luteolin was not found, it is not clear if this yellow component represents a dye. Chrysoeriol is the main component in Holy herbs (*Eriodictyon* species) of the *Boraginaceae* family (Schweppe 1993: 356), but these plants are native to North America and Mexico and are therefore excluded. The yellow component might be a degradation product or originate from the archaeological context of an unknown plant material.

All Rikala samples contained traces of indigotin, but also several unknown yellows and a woad related flavonoid in the weft 1 (Sample 3a, 3b, 3c, 3d, Table 3). This dye combination has possibly produced a greenish tint on the textile. In Finland, the traditional yellows and oranges with a good light fastness came from the spring leaves of silver birch, bog rosemary, crowberry and heather. These have rutin and quercetin as the main dye components (Proano Gaibor 2011). These dyestuffs are absent in all the analysed fragments, which raises the question whether these yellow yielding plants were used at all as a mordant dye in Iron Age Finland or perhaps the dyestuffs have just degraded completely.

Unknown colouring components

There are several cases in the European textile research, in which unknown dyestuffs have been detected. There are unknown yellow and red-orange components in the Hallstatt textiles and Scandinavian peat bog textiles (Bender-Jørgensen & Walton 1986; Walton 1988; Vanden Berghe et al. 2009: 1918; 2010; Hofmann-de-Keijzer et al. 2013: 160–1). An unidentified dye called yellow-X has been detected in TLC analyses in Scandinavian and Finnish textiles (Walton 1988; 2001b.) Several unknown yellow and colourless compounds in the Luistari and Rikala samples as well as the Tuukkala sample 4b might be present due to contamination of the buried or the burial environment (Table 3).

An unknown red component was found in the Kirkkomäki sample 6b at 16.11 minutes with an absorption maximum of 468 nm (Fig. 6; Table 3). The same component has been found in three Early Modern samples from Oulu Cathedral (Lipkin et.al. forthcoming). In these samples, several other unknown red and orange components were detected that are probably related to the component labelled 'Unknown-red-468nm'. This component could indicate the use of one or maybe several local dye plants. In addition, an unknown red or orange component was found in samples 7, 8a, 8b, 9a, 9b, 10, 11 eluting at 19.60 minutes, with an absorption maximum of 429 nm (Fig. 7; Table 3). This component showed a very typical spectrum, but could not be related to any dye class known. In addition, this component could indicate a local dye species.

Many unknown dyestuffs, presumably anthraquinones, correlated with the samples in which red tannins were found using TLC analyses (Walton 2001a; 2004). This situation suggests that the TLC analysis detected these same compounds, but it must be noted that with the (U)HPLC system used no specific markers could be found for red tannins. However, the UHPLC analysis was not able to solve the mystery of these dyestuffs despite several reference yarns. For example, the reference dyed with mushroom surprise webcap showed the presence of emodin and equivalents as the main components, but since these are lacking in the analysed samples this dye source can be excluded. However, there might still be mushrooms that do not produce emodin and are not present in the reference library. Late Iron Age flora was different from modern flora and today it is difficult to find certain old dye plants for reference dyeing, such as the common corn gromwell (*Lithospermum arvense* L.) that was typical in the Medieval environment (Lempiäinen 1999: 122). Thus, the tested references possibly do not reflect the total dye plant stock that was available in prehistoric Finland.

Contamination sources

An inhumation burial has many possible sources of dye contamination that may cause false-positive results. The results of the dye analyses represent the dyestuffs only in a very small area of the textile fragment. In the Tuukkala sample 4b no dye was detected (Table 3), suggesting that the yarn has possibly been truly undyed or the sample amount was insufficient. Even the same yarn, but different parts, can give different results. For example, the earlier TLC analysis (Walton 2001a) found a trace of alizarin from the Kirkkomäki leg wrap's band, but the UHPLC gave a negative result. In addition, in theory, the trace of alizarin as well as the negative result might both be a caused by the migration of the dyestuffs, although this is still to be proven.

The female in Kirkkomäki grave 1 was buried with newly cut barley in her hands and moss and fur layers under her (Riikonen 2003). Quite similar additions were found in the Kirkkomäki grave 27 which contained moss, fur and a wooden coffin (Kirjavainen & Riikonen 2005: 34; 2007: 135). All these grave goods could in a theory yield colouring compounds to burial textiles. The plant offerings and moss may contain flavonoids and tannins, and the furs might have been tanned with tree bark. Moreover, there have likely been plants growing above the burial for centuries. It is difficult to estimate the leak of colorants, their role in the soil and respectively in the archaeological textiles. Attempts at systematic macrofossil and pollen sampling both inside and outside the burial have been applied in textile research recently in Spain (Llergo et al. 2013) and that technique would also be useful for the Finnish research.

In Finland, there is frost in the ground four months in a year every winter. In the experiments done by the Finnish dyers and the author, it has been found, that some red dyestuffs turn brighter and pinker, if frozen first (Tetri 2008: 49). An Iron Age textile that has been buried for one thousand years has been subject to frost and frost-free periods so many times, that this might have affected somehow the organic dyes. Unfortunately, it is unknown, how these repetitive changes in the burial environment have affected the degradation rate of the dyestuffs.

In addition to organic compounds, a synthetic dyestuff Diamond green was found in the Kirkkomäki sample 12b (Table 3). This synthetic dye is also known as Malachite green, but unlike mineral malachite, this dye contains no copper. It was developed in 1877 by O. Fischer and is still produced (Schweppe 1993: 625). Because of this finding, sample 12b was examined with a scanning electron microscope (JEOL 5910 LV) coupled to an Energy Dispersive X-ray Spectrometer (SEM-EDX), which revealed partly degraded and slightly contaminated wool fibres. Therefore, the fibres were much older than the dyestuff detected. In this case, the indigoid dyestuffs and the unknown oranges are from the Iron Age. The synthetic dye was most probably a result of modern contamination.

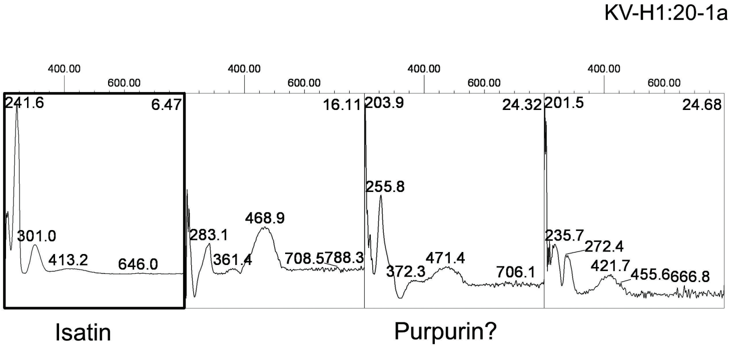

Fig. 6. Spectrum of sample 6b. Illustration: M. van Bommel.

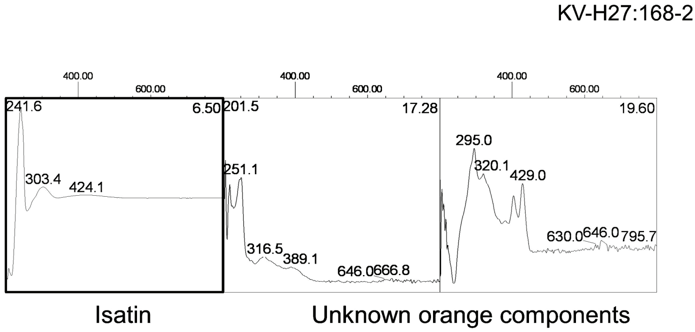

Fig. 7. Spectrum of sample 7. Illustration: M. van Bommel

It is difficult to know how the age of the textiles, the different excavation methods and the idle-time between the excavation and the dyes analysis affect the organic compounds. Possibly the excavation made in a laboratory environment, as well as the relatively rapidly carried out dye analyses, might detect the dyestuffs better. This could explain the several different dye compounds found in the Kirkkomäki grave 27 samples, which were excavated in the laboratory. Indigoid dyestuffs were the only findings in the Luistari, Rikala and Yliskylä fragments; these were also amongst the oldest textiles by date and excavated in the field. Perhaps the time spent in both the ground and the museum destroys dyestuffs; the mordant dyes faster than the indigoids.

CONCLUSIONS

A typical Finnish Iron Age textile type, 2/2 twill made of Sz/z yarns, as well as the period-typical double coated wool suggest strongly, that the textile fragments from Kirkkomäki, Rikala, Yliskylä and Luistari are of local origin and contain local dyestuffs but the source for red remained undefined. However, the Late Iron Age red dye came from different sources than the Finnish Medieval Period red: Dyer's madder and bedstraw reds predominated in the Medieval Finnish and the contemporary European material. The presence of purpurin in the Kirkkomäki sample 6b, KM 12687:H1:20 indicates that bedstraws were rarely used with the unknown red-yielding local plants.

The analyses made with HPLC-PDA and UHPLC-PDA gave interesting results without harming the original textile fragments remarkably. In all cases, the sample sizes were very small, less than one centimetre of yarn. Several different dye compounds were found, but indigoid dyestuffs were the most often detected. This does not indicate that blue garments would have been the Iron Age's main fashion; indigo pigment is just one of the most stabile natural dyes and thus likely has been preserved when other dyestuffs have vanished.

The presence of Dyer's madder and weld in the striped Tuukkala textile is an exceptional finding amongst the Finnish prehistoric textiles. These dye plants were not spread or have been cultivated in Finland. The unusual textile structure, exceptional fibre diameter distribution, and presence of non-local dyes suggest an imported textile product. This item represents dyeing craft of the international Medieval world rather than the local Iron Age dye traditions.

Some of the unknown red and red-orange dyestuffs correlated to the red tannins that were found in earlier TLC analyses (Walton 2001a; 2004). The unknown dyestuffs might refer to the use of still unknown local dye sources or an undefined natural mordant, degraded products of dyestuffs, or contamination of the burial environment or from the deceased. Currently, the reference material of the northern dye plants is quite wide, but still there might be plants, mushrooms or lichens that are not included to the database.

NOTES

[1] SKS / KRA. Pielavesi. Tiitinen, M. 9. 3186. 1937; SKS / KRA. Korpilahti. Hyvärinen. i, k, e: 2691–2692. 1945.
[2] SKS / KRA. Kirvu. Pärssinen. E 142: 14–16. 1938; SKS / KRA. Rautu. Snellman. E 134: 6–7. 1936.
[3] SKS / KRA. Kirvu. Pärssinen. E 142: 14–16. 1938; SKS / KRA. Inkeri. Mannonen. 12 d, hi, j, s: 4409. 1937.
[4] NBA / Ethnographic archive. Photograph of a bedstraw hoe from Karelia, Räisälä.

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NBA / Ethnographic archive. Photograph of a bedstraw hoe from Karelia, Räisälä. C12, L 2607, Dia 26075.

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