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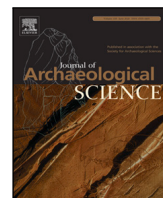
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## Research papers



# The oldest dress of the Netherlands? Recovering a now-vanished, colour pattern from an early iron age fabric in an elite burial

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## ABSTRACT

The Uden–Slabroek cemetery yielded one of the richest Early Iron Age burials found in the Netherlands: an inhumation grave of a person wearing elaborate bronze and iron ornaments (ca. 8th century BC). Exceptionally mineralised fragments of wool textiles were found inside the corrosion layer of the bronze anklets and bracelets. Advanced multidisciplinary analysis of these textiles identified them as the remains of the oldest garment of the Netherlands, made from a bright red and blue wool twill fabric woven in a shepherd's check pattern. Advanced mass spectrometry analysis provided direct identification of dyes, including cochineal — the earliest evidence of a red dye for the entire region (especially the precious insect dye variant). Virtual synchrotron-based 3D analysis of the fragments documented the textile weave types and the morphometry of surface and buried layers. The correlation of the two approaches makes it possible to recover an entire, now-vanished, colour pattern from highly altered fabrics.

## 1. Introduction

Over the past twenty years, new methodologies have emerged that allow a deeper understanding of ancient organic systems and their context. High-resolution mass detection of organic molecules and the development of new imaging modalities are among these major developments. In this paper, we report on the interdisciplinary study of some of the oldest known textiles in the Netherlands. They were discovered in an Early Iron Age elite burial excavated on Slabroekse Heide near Uden (North-Brabant) and date from the 8th century BC (Fig. 1). We combined state-of-the-art molecular dye identification with recently developed high-resolution X-ray synchrotron-based micro-computed tomography imaging. In this instance, it is the oldest reported use of a shepherd's check pattern in the Netherlands, woven from bright red and blue threads. The red threads were dyed using a crimson-dyeing scale insects dye, the earliest known use of this dye in the Netherlands. The results demonstrate the strengths and potential for knowledge gain of combining 3D imaging techniques with molecular identification and provide clues regarding the manufacturing processes of these early textiles and the possible provenance of the raw materials used to produce them.

## 1.1. Archaeological context

The textiles discussed in this paper are among the oldest known in the Netherlands. They were discovered in an Early Iron Age elite burial excavated on the Slabroekse Heide near Uden in the southern Netherlands (ca. 8th century BC). The burial consisted of the inhumation grave of an individual buried in a small wooden chamber. This person was adorned with an array of bronze and iron ornaments (Fig. 1a). Remarkably, textiles survived in the corrosion layer of the bronze bracelets and anklets, even though the harsh sandy soils had caused the skeleton to completely decay until only a shadow remained in the soil (Fig. 1b–d). During excavation, all the metal jewellery was block-lifted and moved to the restoration laboratories of Restaura (Haelen, the Netherlands). Here, they were meticulously excavated, recorded, and consolidated (polyvinyl acetate – PVAc 10%) with great care towards future analysis (Kempkens and Lupak, 2021).

Initial technical analysis of the textiles to identify the raw material and document thread diameter, fine structures of the weave and details of patterns was carried out in 2015 with a digital microscope (DinoLite,

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**Fig. 1.** The grave goods from the Uden-Slabroekse Heide elite burial and textile remains during excavation. (a) The grave goods from the elite burial, including bronze anklets and bracelets, bronze hair rings, bronze and iron pins, iron toiletry set, an amber bead and a selection of textiles. Source: J. van Donkersgoed. Textile remains on and under the bronze objects from the inhumation grave during excavation at Restaura: Find no. 58: right anklet (b), find no. 70: right bracelet (c), and find no. 72: left anklet (d). Image credits: Restaura.

AM73915MZT4) at 30× to 250× magnification (Grömer, 2015). This revealed the presence of two types of wool textiles (Van der Vaart-Verschoof, 2017; van der Vaart-Verschoof, 2017). Weave A was found on both bracelets and anklets, directly on the surface of the bronze ornaments. It consisted of a 2/2 twill made of single threads spun in z-twist with an average thickness of 0.5–0.8 mm and a thread count of 9–11 threads per cm. Due to the distribution of the textiles on the ornaments, we know that the garment had long sleeves and that the bottom was open like a skirt or a dress. Parallels from the period and pictorial sources led to the garment being interpreted as the probable remains of a long-sleeved, ankle-length dress (although a long skirt and long-sleeved top is also possible). Weave B was only found on the bracelets, located only on top of Weave A. It consisted of a finer twill with 0.3–0.4 mm S2z-ply yarn and 16–18 threads per cm, and is likely to have been either a veil, scarf or perhaps a shroud (Grömer, 2016; Schumann and van der Vaart-Verschoof, 2017; Van der Vaart-Verschoof et al., 2021).

Visually, a check pattern woven in two colours could be discerned in the fragments of Weave A. This observation prompted a detailed campaign of chemical dye analysis and 3D imaging of the textiles.

## 1.2. Background on methodology

The development of methods dedicated to the molecular analysis of ancient organic systems has led to significant progress in recent years. A striking example is the development of analytical instruments and methods that provide direct species identification, phylogenetic and genetic evolution information from ultra-low traces of ancient DNA (Peris et al., 2020). Proteomics has been another area of intense development and has allowed independent identification of species, with a current deep interest in the identification of biomarkers of entire ecosystems found in diluted forms in soils (Hendy, 2021; Kjær et al., 2022; Manning et al., 2009). Isotopic studies of textiles are increasingly being used to

distinguish between local and traded textiles, and to determine their place of origin. Early attempts to isotopically trace textiles attached to metal objects were successful, but only if the textile material was in a minimally altered state of preservation (e.g. Voldtofte Bergfjord et al., 2012). The characterisation of dye sources in archaeological textiles has been tackled in the past by chromatographic and spectroscopic techniques (namely, HPLC-PDA), allowing the detection of the main molecular markers even in very complex matrices, although often requiring extensive sample preparation. Few cases of analyses undertaken on textiles from the 1st millennium BC have been described: Joosten et al. (2006) detected indigoids, flavonoids, lichen dye markers and anthraquinones (carminic and kermesic acids) in samples via HPLC-PDA from archaeological textile fragments found in the Hallstatt salt mine in Upper Austria (1400–400 BC) (Grömer et al., 2013). By applying a double step extraction pre-treatment, Vanden Berghe et al. (2009) detected flavonoids, indigotin and plant anthraquinones (alizarin and purpurin) in peat bog textiles from Denmark and Southern Norway (500 BC–400 AD); similar textiles have also been analysed by Surowiec et al. (2006) in 2006, although from much more recent archaeological sites (16th–17th century). Still, the use of the spectrophotometric UV-visible detection limited the identification of the dye source in terms of sensitivity and selectivity: matrix effects leading to high background signals often hindered the detection of minor components. In this regard, unprecedented identification of historical dye sources could be achieved through significantly increased resolution in mass spectrometry (Jia et al., 2014) and the development of chromatographic methods increasingly capable of detecting a variety of organic molecules in a single run (Degano, 2020; Degano and La Nasa, 2016).

Beyond conventional textile characterisation methods, such as optical microscopy and, more occasionally, scanning electron microscopy (SEM) for fibre identification, the advent of digital microscopy has brought a significant improvement, overcoming the limitations of depth of field and enabling digital reconstruction of the depth profile of

the samples surface. However, some morphometric parameters are generally impossible to obtain non-invasively. In parallel, a range of (spectral) imaging techniques have emerged, from multispectral imaging to new synchrotron-based X-ray techniques, which have enabled 2D or 3D visualisation of organic remains and a better understanding of long-term preservation pathways. While micro-computed tomography ( $\mu$ CT) has been primarily dedicated to the study of mineral systems (Sanchez et al., 2016), recent developments in optical Raman (and modalities such as SERS and TERS), focal plane array-based FT-IR and UV/visible-based multispectral approaches have provided unprecedented access to 2D organic speciation. Second harmonic generation microscopy has allowed the mapping of cellulose in ancient textile fibres and has demonstrated highly heterogeneous preservation states at the macrofibrillar level (Reynaud et al., 2020). Recently, X-ray Raman imaging has emerged as an entirely new way to assess the organic speciation of ancient systems non-invasively in 3D (Georgiou et al., 2019). Both routes of methodological development are impacting many areas of archaeology, from understanding past human manufacturing practices to the history of the interaction between humans and their social and environmental context.

The main mechanism leading to the preservation of textiles (for example, those from graves) in a form that can be interpreted is referred to by archaeologists as mineralisation. It is a fossilisation mechanism involving biocidal activity, permineralisation and authigenic growth of neoformed phases from dissolution and transport of metal cations from nearby artefacts (Reynaud et al., 2020; Biek, 1963). Despite their sometimes very degraded condition, the study of mineralised textiles still attached to metal objects has provided essential information in terms of “micro-stratigraphy”. Thanks to the context (position on the metal artefact, relation to the body, etc.) in which an object is found, it is possible to understand the function of the textiles (e.g. part of a garment, wrapping of an object in a tomb, textile lining of a scabbard). The clothing and burial practices, contexts of production and technical uses of past societies are thus highlighted, even when the textiles are very significantly altered (Davis and Harris, 2023; Gleba and Mannering, 2012; Solazzo et al., 2014; Iacconi et al., 2023, 2024). But their scientific study is particularly difficult in highly mineralised fragments or in the case of superimposed layers. Detailed study of these multiple layers without additional technical equipment is generally not possible without destroying the superimposed layers. Due to the scarcity of textiles from burial contexts in Iron Age Europe, a sensible and careful sampling strategy must be an integral part of the research approach. It has recently been demonstrated that synchrotron-based X-ray microtomography (SR- $\mu$ CT) allowed the technical analysis of textile samples in such cases by providing access to the microscopic characteristics of their internal morphology non-invasively, without damaging their physical integrity (Iacconi et al., 2023, 2024).

## 2. Materials and methods

### 2.1. Textile samples from Uden-Slabroekse Heide

The wool textiles studied were found in the corrosion crust of the bronze bracelets and anklets found at Uden-Slabroekse Heide. Before sampling, the textile fragments were carefully examined and areas of interest were selected from microscopic observations (digital microscopy and hyperspectral imaging). Fragments from the right and left anklets were chosen because their colour differences were the most distinctive. These ten textile samples, ranging in size from 13 mm  $\times$  10 mm (smallest) to 17 mm  $\times$  12 mm (largest), were studied with SEM-EDX and SR- $\mu$ CT. From these textile samples, threads (0.5–5.0 mm long) were extracted and analysed with UHPLC-PDA-HRMS. An additional large textile fragment (90 mm  $\times$  18 mm) was examined with SR- $\mu$ CT.

### 2.2. Dye extraction and analysis by UHPLC-PDA-HRMS

The dye extraction was performed with a double step procedure, entailing first a non-acidic treatment for retrieving possible glycosides and labile dye-molecules (dimethyl sulfoxide, methanol and water extraction solvent, heated for 5 min at 80 °C), followed by an acidic one (2:1:1 mixture of 35% hydrochloric acid and methanol, heated in a heating block at 100 °C for 5 min), chosen to reduce as much as possible any interferences due to the complex matrix of the target samples. Two consecutive blanks were run and checked between the analyses of each of the archaeological samples. Analysis of the organic colourants was carried out with an ultra-high performance liquid chromatograph with a UV-visible photodiode array detector and a heated electrospray ionisation (HESI) – high resolution mass spectrometer (Orbitrap Q-Exactive focus, Thermo). The separation took place on a BEH-RP18 column and was achieved with a gradient of methanol and water, using formic acid as mobile phase modifier. The mass spectrometer operated in the 140–1700 m/z range in a single run (resolution 35,000; positive and negative polarity mode). Details are provided in Supplementary Texts S1.3 and S1.4.

### 2.3. Synchrotron X-ray microtomography

Synchrotron X-ray microtomography (SR- $\mu$ CT) was performed at the PSICHE beamline of the SOLEIL synchrotron facility (Saint-Aubin, France). The samples were studied in a  $\mu$ CT configuration identical to that used in Iacconi et al. (2023), ‘pink’ beam: 40–100 keV; peak intensity at ca. 65 keV:  $2.7 \times 10^{11}$  ph/mm<sup>2</sup>/s; effective voxel size: (5.7  $\mu$ m)<sup>3</sup>, for the large sample and peak intensity at ca. 60 keV:  $2.7 \times 10^{11}$  ph/mm<sup>2</sup>/s; effective voxel sizes: (5.1  $\mu$ m)<sup>3</sup> and (3.1  $\mu$ m)<sup>3</sup> for the smaller samples. Details are provided in Supplementary Text S1.5.

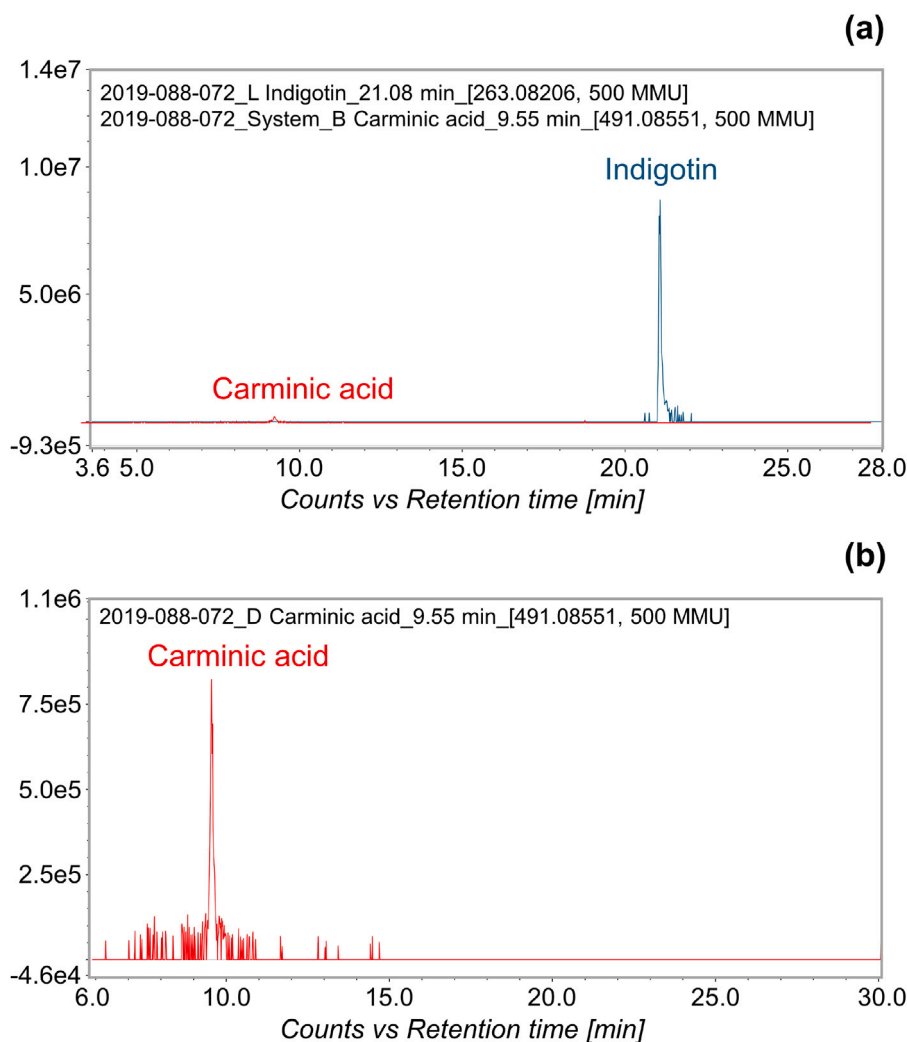
### 2.4. Additional methods

Digital microscopy was performed using an Hirox instrument. Details regarding the use of video-spectral comparison (VSC) and scanning electron microscopy coupled to energy dispersive X-ray spectrometry (SEM-EDX) are provided in Supplementary Texts S1.1 and S1.2, respectively.

## 3. Results

In order to identify the dyestuff used to create the bicolour pattern visually observed and to non-destructively visualise the different layers of the textiles from the Uden-Slabroek elite burial, textile samples were studied through a combination of dye identification, SR- $\mu$ CT imaging and electron microscopy. The textiles appear to be in an intermediate state of mineralisation. They clearly contain copper-based minerals, the presence of which has been confirmed by our various analytical approaches. However, it has been shown that highly mineralised textiles can retain well-preserved organic domains (Reynaud et al., 2020). Consequently, the distinction between more and less mineralised textiles appears more complex than generally reported. Multispectral imaging using video-spectral comparison allowed for a better distinction between threads that appeared lighter (type L) or darker (type D) in colour, on which a minimal sampling strategy was established for further dye analysis (see Supplementary Figures S3.1 and S3.2). Darkness and lightness were chosen relative to what was visible in one fragment because their hue differs from fragment to fragment. This finding raised the question of the possible use of different dyes for the two types of threads. Analysis of the textiles revealed the presence of two different weaves on top of each other on the bracelets and established that one of these (found at both bracelets and anklets) was woven in a bicolour check pattern.





**Fig. 2.** Identification of carminic acid and indigotin in the extract of textile fragments VNR 72 A, B from the left ankle of Uden-Slabroekse Heide (Early Iron Age, ca. 8th BC): (a) Extracted ion chromatogram corresponding to the  $[M - H]^-$  molecular ion of carminic acid ( $t_R$  9.55 min) and the  $[M + H]^+$  molecular ion of indigotin in the extract of type L thread. (b) Extracted ion chromatogram corresponding to the  $[M - H]^-$  molecular ion of carminic acid in the extract of type D thread.

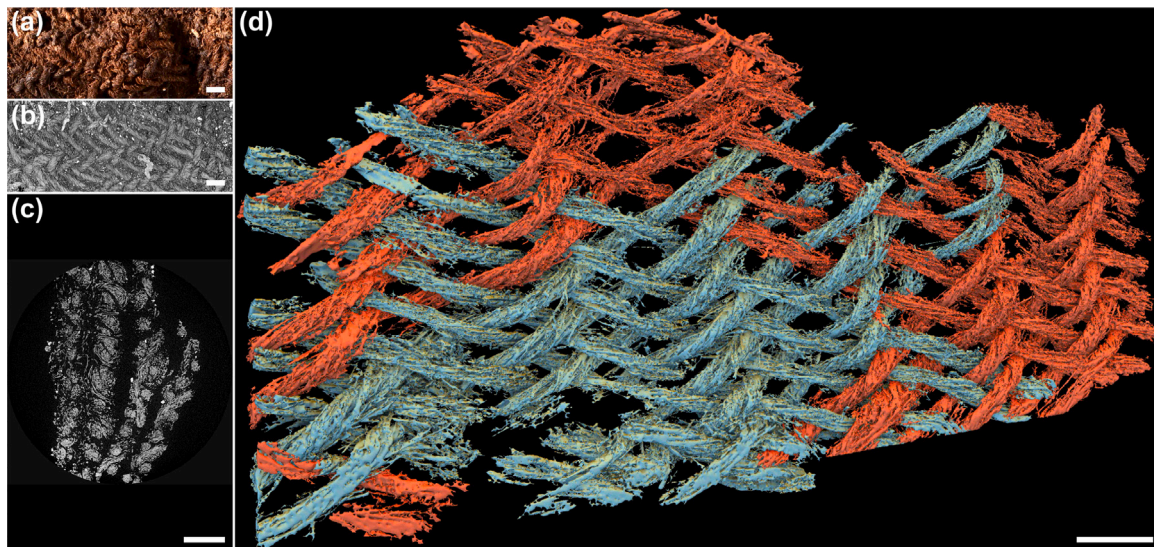
### 3.1. Dye analysis

Ultra-high performance liquid chromatography – photodiode array – high-resolution mass spectrometry (UHPLC-PDA-HRMS) was performed using a protocol designed to collect high resolution mass spectra from archaeological fragments (resolution 35,000) by sampling only 0.5 mm of thread from the heavily altered textile fragments.

The textile fragments from the left and right anklets have a complex check pattern of at least two coloured thread systems. An indigoid dye as indigo (unknown in the Iron Age Netherlands) or woad was identified in type L threads from the left ankle (sample 72; Fig. 2a), thanks to the detection of their major component indigotin ( $t_R$  21.0 min,  $[M + H]^+$   $m/z$  263.082). In type D threads from the left ankle (sample 72; Fig. 2b), the molecular marker of crimson-dyeing scale insects (as cochineal) was detected, namely carminic acid ( $t_R$  9.5 min,  $[M - H]^-$   $m/z$  491.085). No secondary component, which might allow for discrimination of the insect genus or species as dCI, present in Armenian (*Porphyrophora hamelii* L.) and American cochineal (*Dactylopius coccus*) (Serrano et al., 2011), and pPI, marker of Polish cochineal (*Porphyrophora polonica* L.) (Lech and Jarosz, 2016), could be determined above the instrumental limit of detection. The attribution of the molecular markers was based on the comparison with retention times of reference materials, and on the exact mass, lower than 5 ppm in all cases.

Similar results were obtained for the analysis of sample 58 (right ankle), where type D threads mainly contained the marker of crimson-dyeing scale insects (carminic acid,  $[M - H]^-$   $m/z$  491.085) with traces of indigotin, and type L threads contained indigotin as the main component ( $t_R$  22.04 min,  $[M + H]^+$   $m/z$  263.084) and traces of carminic acid. It is very probable that the threads were partially cross-contaminated during their long-term stay in the soil, hence the very minor presence of a red dye in type L threads and blue dye in type D threads. None of the analytical blanks run between the analysis of the archaeological textiles showed any of the above discussed chromatographic peaks.

Based on the dating and provenance of the textiles, the most probable candidates as dye sources are woad (*Isatis tinctoria*) (Hofmann-de Keijzer, 2016) for the light threads containing indigotin, and among the crimson-dyeing scale insects, Polish cochineal (*Porphyrophora polonica* L.) for the darker threads. The positive identification of crimson-dyeing scale insects as raw material is important because it is currently the earliest evidence of a red dye in the entire region. Early use of crimson-dyeing scale insects has been identified in several Iron Age sites in other regions, in particular in Hallstatt (Upper Austria; Grömer, 2016; Hofmann-de Keijzer, 2016). Moreover, carminic acid was detected in several textiles from the Near East and Central Asia, as in the Pazyryk carpet (5th–4th c. BC, Siberia), that is the earliest surviving knotted-pile carpet, found in Scythian Iron Age tombs discovered in the Altai Mountains (Böhmer and Thompson, 1991); in slightly later fragments,



**Fig. 3.** Reconstruction of the Iron Age shepherd's check textile from Uden-Slabroekse Heide (Early Dutch Iron Age, ca. 8th BC, find no. 58) combining data from mass spectrometry dye identification with SR- $\mu$ CT. (a) Macrophotography. (b) 3D reconstruction. (c) Virtual section showing the four superimposed textile layers. (d) 3D visualisation of the blue and red shepherd's check pattern visible on a hidden textile layer of find no. 58. All scale bars: 1 mm.

as described by Cardon (2007); and even in textiles from the desert of Taklamakan (half of the 1st millennium BC, China), as reported by Debaine-Francfort and Idriss (2001).

The presence of aluminium, copper and iron was established by SEM-EDX in both type L and D threads (see Supplementary Text S4). All the fibres contained similar significant amounts of aluminium, traces of iron and copper. While it is possible that most of this content came from the soil, it cannot be ruled out that some came from mordants used for dyeing. The question of which mordant was used for type D threads remains unanswered.

### 3.2. Virtual technical analysis

A selection of 11 samples was characterised using SR- $\mu$ CT according to the virtual examination method developed by Iacconi et al. (2023). For each sample, macroscopic to microscopic parameters were determined, in particular the thickness of each layer, the weave type, the thread count, and the respective diameters of the System 1 and the System 2 threads (Supplementary Table S2.1). Four samples had superimposed layers, up to four, which could be studied separately by virtual individuation.

We first studied the large textile fragment from the right ankle showing different visual hues and some threads dyed with different dyes (find no. 58; Fig. 3a). SR- $\mu$ CT revealed that the sample was made of four superimposed layers of textiles of similar thickness and overall technical characteristics (Fig. 3c; Supplementary Table S2.1). The individual textile layers were segmented, and a statistical analysis using gradient estimation-based segmentation was used to better differentiate the System 1 and System 2 threads (Iacconi et al., 2023). The thread diameters were determined based on sphere fitting, which does not depend on the selection of a cutting plane as in conventional microscopy (Iacconi et al., 2023).

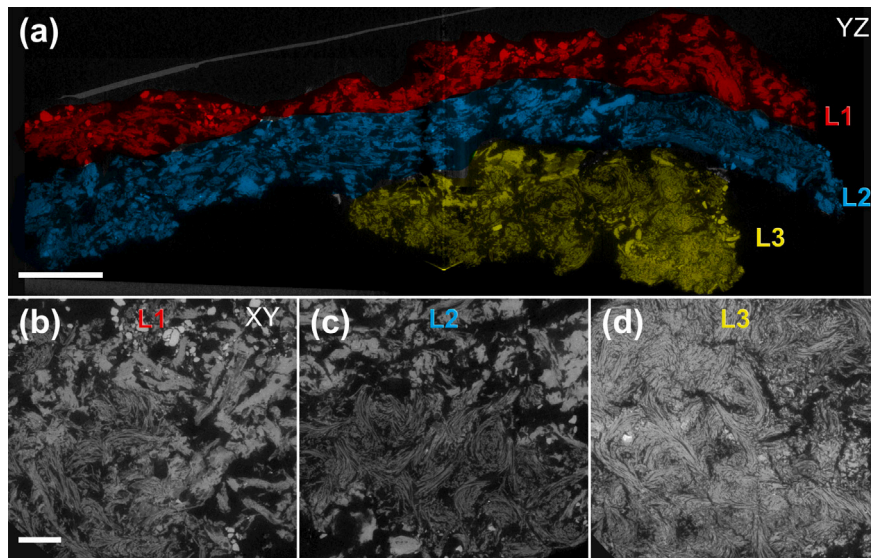
In general, the diameters of the threads used for all 11 samples are very fine, in comparison with textiles from the 1st millennium BC in Central and Northern Europe, and the textiles had to be particularly well finished (Grömer, 2016; Grömer et al., 2013; Gleba and Manning, 2012). It can be assumed that they come from at least three different fabrics: two different types of twill and one shepherd's check. While the surface textile layers could previously be identified visually, the buried layers could only be identified by our  $\mu$ CT-based approach (see Supplementary Text S2). Twill samples with superimposed layers could all be characterised non-invasively, all layers being identified as

2/2 twill z/z-yarns (see for instance Fig. 4). In this sample, the three layers show a very similar morphometry, consistent with the past use of a single folded textile.

### 3.3. Identification and study of a bicolour check pattern

We identified a bicolour check pattern, not only for the visibly accessible surface layer, but for each of the four layers (Fig. 3d). For layers studied solely by SR- $\mu$ CT (i.e. those not visible from the outside), the dyes themselves are not visible, and only characteristics such as thread diameter and twist angle can be used to differentiate between the two types of thread (D and L). Morphological differences such as diameter and twist value are used to match the right dye to the right thread system. Beyond the quantitative assessment of the technical parameters of the fabrics, SR- $\mu$ CT segmentation is guided by the visual perception of the 3D volumes, which helps to validate the results of the segmentation steps. We observed that indigo-dyed threads were less twisted, more compact (with less space between individual fibres) and slightly smaller in diameter. This may be linked to the observation that these threads contain more medullated fibres (kemp) than those dyed with cochineal. As kemp is thicker and coarser, it offers greater resistance to torsion. There are several versions of bicolour checks known in Central Europe and Northern Italy since the 9th century BC (Grömer and Gleba, 2020). In our sample, a pattern alternating eight red and eight blue threads in warp and weft was for the first time identified in the Netherlands, which is referred to as shepherd's check by modern textile technology and textile archaeology. In order to obtain a realistic 3D rendering of the textile with the colours at the time of burial, the colours measured by colorimetry on a modern dyed textile were applied to the virtual threads. As the surface layer is much more altered than the buried layers, the pattern is particularly evident when reconstructing these otherwise inaccessible layers.

A very interesting point is that some organic dyes tend to fade when exposed to sunlight and wear. Garments dyed only with woad, for example, are often 'brightened' after prolonged use by redyeing the entire garment. This strategy would not have been feasible for the Slabroek garment, due to the shepherd's check pattern. When dyeing with an anthraquinone colourant, such as crimson-dyeing scale insects as cochineal, the hydroxyl groups actively engage in attaching the dye to the wool fibres. The fibres are commonly pre-treated with the salt of a metal such as aluminium, iron, tin, or copper to increase colour saturation and its long-term stability. For the Slabroek garment whose



**Fig. 4.** Identification of three layers of twill from a bronze bracelet of Uden-Slabroekse Heide (Early Dutch Iron Age, ca. 8th BC) by SR- $\mu$ CT imaging. Sample 71C shows three superimposed layers of textile that can be studied separately by virtual individuation. (a) Virtual cross-section of the whole sample in the YZ plane. Three layers of textiles in total are observed. (b–d) Virtual sections of layers 1 (b), 2 (c) and 3 (d) in the XY plane. All scale bars: 1 mm.

light fastness may have been a sign of its value, the use of a mordant is likely. Due to the highly altered condition of the wool threads and the burial conditions, the composition of the possible mordant could not be identified by SEM-EDX.

#### 4. Discussion

##### 4.1. The Slabroek textiles in context

The elite grave from Uden-Slabroek is unique in the Netherlands both in terms of grave goods and funerary rites, though the latter does show some connection with local elite burial practices (Bourgeois and Van der Vaart-Verschoof, 2021). The presence of textiles surviving in an Early Iron Age grave is almost as rare in this area. While Early Iron textiles have been found in other parts of Europe (Grömer, 2016, 2017), where preservation conditions are more favourable, there is only one contemporary textile find in the area: the textiles from the Chieftain's Grave of Oss (Van der Vaart-Verschoof, 2017; van der Vaart-Verschoof, 2017). Oss is located only 12km north of Slabroek. It is a cremation grave of a male interred with a rich set of grave goods: a Mindelheim type sword with gold-inlaid handle, tack for two draft horses and part of a yoke, a knife, an axe, dress pins, and more. The Chieftain's cremated remains and his grave goods were placed in a large bronze situla that served as an urn. Most of the grave goods were imported from the Hallstatt Culture of Central Europe (Van der Vaart-Verschoof, 2017; Schumann and van der Vaart-Verschoof, 2017). The burial yielded a surprising amount of textile fragments that survived both in the corrosion of the metal grave goods and as loose fragments. Analysis revealed a total of eight different weaves, of which some were used to wrap grave goods, while two high-quality textiles (possibly garments) were folded up together and deposited as grave goods (Grömer, 2017). The latter consisted primarily of an extremely fine and high-quality diamond twill, it is the result of the work of a highly skilled and experienced craftsman, and probably took several months to be made (Grömer, 2017).

Similar to the Slabroek textiles, in this case, the comparison of the virtual technical analyses carried out on the samples made it possible to highlight the superior quality of those found in Oss (Grömer, 2017). In short, it would appear that the textiles from Oss and Slabroek did not serve the same functions during the funerary rituals. In the case

of Slabroek, the textile remains were identified as coming from a long-sleeved dress worn by the deceased buried there. In contrast, the Oss textiles were primarily used to wrap grave goods such as the sword and some horse tack, while others could be identified as grave gifts in their own right. While the latter may represent a garment, it was not worn by the deceased as he was cremated, but rather placed in the bronze urn as a grave gift. While the quality of some of the textiles in the Oss elite burial may be superior to those of Slabroek, no patterns woven in different colours have yet been found, maybe primarily due to the heavy mineralisation of the Oss textiles. The Slabroek textile therefore forms the oldest evidence in the Netherlands for a bicolour textile, and the oldest chequered pattern in the area.

Bicoloured chequered textiles, however, have been found elsewhere in Europe from the Early Iron Age onwards, for example, in the salt mines of both Hallstatt and Dürnbürg — perhaps the most famous sites in terms of prehistoric textiles (Grömer, 2016; Grömer et al., 2013; Grömer and Gleba, 2020; Bichler et al., 2005). The earliest bicoloured checked fabrics in Europe were found at Sasso di Furbara in Central Italy and date from the 9th century BC; others are known from Vulci (8th c. BC) and Verucchio (ca. 700 BC) (Grömer and Gleba, 2020). Bicoloured chequered textiles identified as specific garments are rare and have only been recovered from later on in the Iron Age. Famous examples include the checked wool skirt and scarf worn, among other things, by the so-called Huldremose woman who was placed in a Danish bog in the 2nd century BC. Colour analysis has shown that these garments were originally blue and red (Giles, 2020). Another example is the Gerum cloak (ca. 360–100 BC), which was found in a Swedish bog in the 1920s, and is a true houndstooth with a tessellating shape woven of dark and light wool (Frei et al., 2009). Overall, the Slabroek dress appears as an unusually early example of a bicoloured checked garment.

##### 4.2. Innovative combination of morphological and spectroscopic data

Coupling broad band multispectral or hyperspectral imaging with dyes analysis has become part of the protocol to be applied in the examination of textile collections, as proven by the successful application of this strategy to Egyptian and Asian textiles at the British Museum (Dyer et al., 2018; Tamburini and Dyer, 2019; Tamburini et al., 2020). Nonetheless, combining molecular identification and imaging information remains a difficult undertaking. This is due to



analytical limitations that often prevent direct combination but also, more trivially, to interdisciplinary challenges. The study of faint organic signatures from highly altered systems of biological origin in complex soil environments is one of the key questions in this historical field. Their study is particularly challenging as organic systems gradually evolve into hybrid organic–inorganic complexes (e.g. adsorbed geopolymers) with intermediate products that are notoriously difficult to characterise with most analytical means, especially to combine chemical information and 3D distribution (Bernard and Papineau, 2014). Case studies are essential for defining new protocols and instruments to explore the potential of combining advanced molecular identification and 3D imaging. An emblematic case is the study of ancient traces of textiles found in temperate areas (Gleba and Manning, 2012; Biek, 1963). While textile production is attested since deep prehistoric times, enzymatically-induced hydrolysis and oxidation of macromolecules, and the leaching of labile hydrolysable compounds (keratin and keratin-associated proteins, cellulose and hemicellulose, fibroin and sericin, for keratinous (such as wool), cellulosic (such as bast) and silk respectively) leads rapidly to scission and dispersion in temperate soils of regular pH in weeks to months, or polymerisation towards the formation of geopolymers (Collinson, 2010; Arshad et al., 2014).

While the analysis of dyes in archaeology has evolved rapidly with increasingly sensitive and selective mass spectrometry detectors and more complete mass databases, the development of micro-computed tomography for such samples is very recent (Iacconi et al., 2023; Lipkin et al., 2023; Karjalainen et al., 2023). The latter can, to some extent, be seen as an extension of the use of CT imaging in palaeontology and natural history, where the method has developed considerably over the past two decades. As in palaeontology, CT in archaeology first developed for macroscopic observations, notably through the use of medical scanners to study human remains and other pluricentimetric archaeological artefacts. The move towards gathering archaeological information on a microscopic scale is more recent. This development means that we can go beyond the general description of shapes to take morphometric measurements, exploiting not only the qualitative potential of imagery, but also its quantitative potential. The latter takes advantage of the possibility of obtaining accurate measurements, less affected by the often arbitrary selection of cutting planes when preparing physical sections of samples for optical and electron microscopy. New biases then emerge (Iacconi et al., 2023), as the intrinsic complexity of a system as simple as a textile cannot be underestimated, nor can the effects of long-term alteration.

The coupling of spectroscopic and morphological levels of information raises a number of questions. On the one hand, the spectroscopic data concern the composition of the sample, i.e. its material composition. On the other hand, the imaging data concern the disposition of the same sample, i.e. its shape. In a sense, it is intriguing that we are combining detailed images resolved at the micro-scale (obtained non-invasively and non-destructively) with point information on the colour of the threads (obtained minimally invasively and micro-destructively), on the assumption that each thread must have been of a defined colour due to the dyeing process used. Another important aspect is that some of the textile layers studied are completely invisible to the naked eye and remain enclosed in the depth of a sample. They are therefore known only through virtual images, although they can still be studied. The visual display of the distribution of spectroscopic information could be an aid to visualisation for scientists, helping to reveal unexplained details beyond the regularity of the weave. This approach should not be expected to provide a vastly simplified overview of the pattern and chemical composition of textiles, but to contribute to a better understanding of the weaving process and the conservation of remains. In the future, the spatialisation of information, i.e. the combination of material and shape, opens the door to a better grasp of the historical trajectory of these samples.

## 5. Conclusions

The Slabroek textiles appear as an exceptional testimony of the use of a dye that required advanced technologies, including preliminary treatment by mordanting baths. A chequered pattern was created by alternating eight red and eight blue threads in both warp and weft. While this is not a particularly complicated pattern to create, the result would have been a striking, brightly coloured garment, befitting its high value and the elite status of the wearer. We have shown that millimetre-scale remains of archaeological textiles could be studied jointly in terms of shape and colour by combining high-resolution synchrotron-based X-ray microtomography with colour information derived from molecular dye identification. The unusual combination of these approaches provides an innovative direction for the study of highly altered bioarchaeological remains. The methods detected along with an indigo-based dye, the first known use of an insect red dye in this area, possibly entailing the joint use of a mordant, and a shepherd's check pattern that required considerable skill and understanding of three-dimensional shapes associated with highly developed textile weaving technologies. Insect dyes were precious because they were much rarer than plant dyes in prehistoric times (and a distant trade item). These findings are the result of very early skills developed in northern continental Europe in the Early Iron Age. They also provide a glimpse of the potential for new discoveries that will be enabled by new imaging techniques in archaeology capable of reading microscale layers hidden beneath the surface of artefacts, especially when coupled with the potential for identification of diagnostic biomarkers in the ultratrace state using advanced mass spectrometry capabilities. Our results probably herald many future discoveries that can be gained from these techniques and their unusual combination, which may continue to expand our knowledge of the onset dates of very early manufacturing processes and material circulation.

## CRedit authorship contribution statement

**Clémence Iacconi:** Writing – review & editing, Writing – original draft, Methodology, Investigation. **Art Proaño Gaibor:** Writing – review & editing, Methodology, Investigation. **Iaria Degano:** Writing – review & editing, Methodology, Investigation. **Sasja van der Vaart-Verschoof:** Writing – review & editing, Investigation. **Ineke Joosten:** Writing – original draft, Investigation. **Karina Grömer:** Writing – review & editing, Investigation. **Loïc Bertrand:** Writing – review & editing, Writing – original draft, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Data curation, Conceptualization.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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## Appendix A. Supplementary material

All data needed to evaluate the conclusions in the paper are present in the paper, the Supplementary Material and/or deposited in the Zenodo repository (doi: [10.5281/zenodo.14002962](https://doi.org/10.5281/zenodo.14002962)).

Supplementary material related to this article can be found online at <https://doi.org/10.1016/j.jas.2024.106109>.

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