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Changes in the production materials of Burmese textiles in the nineteenth century—dyes, mordants and fibres of Karen garments from the British Museum’s collection

Diego Tamburini^{1*} , Joanne Dyer¹, Caroline Cartwright¹ and Alexandra Green²

Abstract

The materials of six Karen textiles from the British Museum’s collection were investigated with the main aim of studying the production changes over the course of the nineteenth century and how these changes related to local and colonial trade networks. The textiles span chronologically from the 1830s to the early 1900s according to their attribution dates, and include traditional garments such as tunics and skirts, in addition to representing a broad colour palette and different dyeing and weaving techniques. The investigation was conducted non-invasively by using digital microscopy, broadband multispectral imaging (MSI) and fibre optic reflectance spectroscopy (FORS). The results guided a sampling campaign during which samples were taken and investigated by optical microscopy (OM), scanning electron microscopy energy dispersive X-ray spectrometry (SEM–EDX) and high-performance liquid chromatography coupled to diode array detector and tandem mass spectrometry (HPLC–DAD–MS/MS). Local natural dyes, such as red and yellow from *Morinda citrifolia*, lac dye (*Kerria lacca*), gamboge (*Garcinia* sp.), turmeric (*Curcuma longa*), indigo and tannins were found in the older textiles, supporting a possible early production, whereas mixtures of natural and synthetic dyes, including synthetic alizarin (CI 58000), rhodamine B (CI 45170), orange I (CI 14600), diamond green B (CI 42000), diamond green G (CI 42040), auramine O (CI 41000), naphthol yellow S (CI 10316), chrysamine G (CI 22250), crystal violet (CI 42555) and Pigment Red 53:1 (CI 15585:1) were identified in the later textiles. Observations on mordants revealed the use of aluminium, tin and chrome, whereas fibre characterisation highlighted the use not only of cotton (*Gossypium* sp.) but also of felted wool imported from Europe and Chinese silk dyed with the Chinese cork tree (*Phellodendron chinense*) yellow dye. The results draw an interesting picture of the introduction of new dyeing materials and techniques in Myanmar over the nineteenth century, and how these impacted the production of traditional Karen textiles.

Keywords Karen, Myanmar, Burmese textiles, Dyes, Fibres, Mordants, Imaging, SEM–EDX, HPLC, FORS

Introduction

The label “Karen” covers a large range of different peoples, often subjectively associated, who live in Shan, Kayin and Kayah States, along the Tenasserim coast in lower Myanmar, and in the Irrawaddy, Sittaing, and Salween delta regions, as well as in Thailand. Also called the Karenic family, the Karen in Myanmar include the Pwo, Sgaw and Karenni groups. Many of the current classifications only came into existence in the nineteenth

*Correspondence:

Diego Tamburini
DTamburini@britishmuseum.org

¹ Department of Scientific Research, The British Museum, Great Russell Street, London WC1B 3DG, UK

² Department of Asia, The British Museum, Great Russell Street, London WC1B 3DG, UK



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century as a result of colonial descriptions, the imposition of the British Census, missionary work, and scholarly analyses [1]. They do not necessarily reflect how the local people viewed themselves within their own cultural frameworks. The conflicts in the twentieth and twenty-first centuries have severely disrupted the production and preservation of Karen cultural objects, with the exception of textiles that are still widely produced and preserved [2]. Textiles form such an important part of a complex representation of identity construction and deconstruction that their study is an important avenue of historical investigation. Analysis of dyes, fibres, weaving and dyeing techniques and patterning has the potential to reveal the trade and cultural networks within which the Karen peoples operated during the nineteenth century, and by extrapolation, in earlier times. Historic textile collections are thus indispensable to understand changes to Karen material cultures. Dye and fibre innovations were incorporated into clothing quite rapidly, and diffused along the market system that created a network of exchange points across urban and rural areas [3]. This is an understudied research field, which places this study as an important starting point to explore how the alterations to textiles provide information about fashion and market changes as Myanmar became connected with the British empire.

Customarily, Karen textiles are made by women on backstrap (body-tension) looms and are most commonly warp-faced tabby (plain) weaves. Warps sometimes end in tassels or pompoms, while continuous and discontinuous supplementary weft yarns are used to develop complex patterning. Warp ikat is another regular feature, especially on women's skirts, where the mythological python design is a mark of a woman's weaving ability. Embroidery is another standard feature, often including

the use of Job's tears seeds (*Coix lacryma-jobi*), as is the incorporation of appliquéd pieces of red cloth [1]. These features are all represented in the six textiles chosen for this study (Figs. 1 and 2).

Early Karen textiles were made of cotton (*Gossypium* sp.) and occasionally silk, although hemp (*Cannabis sativa*) and wool were sometimes used as well. According to historical and ethnographical studies, the predominant natural dyes in Myanmar were mostly sourced from local plants. The most common red dye was extracted from the root bark of the *Morinda citrifolia* plant [4, 5]. Lac dye, obtained from the *Kerria lacca* insect, was also a natural source of red colour [6]. Natural yellow dyes used by the Karen peoples include turmeric (*Curcuma longa*), gamboge (*Garcinia* spp.), jackfruit (*Artocarpus heterophyllus*) wood and bark, mango (*Mangifera indica*) leaves and cockspur thorn (*Maclura cochinchinensis*) [5, 7]. Blue was usually obtained from indigo-producing plants, such as *Indigofera tinctoria*, that were grown and traded in many parts of Myanmar [8]. Mordanting and fixing methods were often used in natural dyeing practices to enhance the colour and fastness of certain dyes. In addition to alum and ferrous sulphate, tannins extracted from a wide variety of plants, fruits and seeds, were used both as mordants and to obtain dark colours [9]. Other mordanting methods include the addition of earth or ash to the dye bath, the use of aluminium-rich leaves (*Symplocos* sp.), the practice of burying the cotton yarns underground or their treatment with oil before dyeing [1]. This information on the materials used in Karen textiles has seldom been confirmed scientifically [6].

With the industrialisation of textile-making in Europe, an abundance of new materials became available in the second half of the nineteenth century. Synthetic dyes



Fig. 1 T1: tunic made in ca. 1830–1840 (accession number As1979,Q.101; length 77 cm; width 69 cm); T3: tunic made before 1863 (accession number As. +.6880; length 85 cm, width 69 cm); T5: tunic made in the late 19th–early twentieth century (accession number As1966,01.481; length 75 cm, width 84 cm). © The Trustees of the British Museum



Fig. 2 T2: skirt-cloth made before 1870 (accession number As.7765; length 114 cm, width 71 cm); T4: skirt-cloth made before 1886, probably 1870s to early 1880s (accession number As1919,0717.200; length 89 cm, width 73 cm); T6: woman's head cloth made in ca. 1890s (accession number As1901,0318.217; length 140 cm, width 30 cm). © The Trustees of the British Museum

represent one of the most important categories of such materials [10], and their introduction from Europe to Asia is an understudied topic [6, 11–15]. Myanmar was subject to considerable Western influence from the early nineteenth century and, as the East India Company became the main supplier of trade goods to the area following colonisation, these new products became available in Myanmar. Considerable transformations occurred in this time period, and evidence exists that the scientific advances and technological developments taking place in Europe impacted Myanmar's textiles and other art forms [16]. As the year of discovery of most synthetic dyes is documented, these molecules can be used as dating tools to establish the earliest possible date of production of textiles, thus potentially yielding important information to interpret the chronological changes in Karen textile practice. Moreover, little in-depth scientific work on Myanmar textiles has been undertaken to date. A study of six Chin and Karen textiles dated to the period 1890–1930 is the only scientific report on this topic, showing that synthetic red dyes, such as fuchsin, synthetic alizarin and rhodamine B, were in use at that time [6].

This study significantly expands this research topic by presenting the results from six Karen textiles with the aim of investigating the changes in textile production

that occurred over the course of the nineteenth century, as well as identifying light-sensitive dyes to develop appropriate display conditions. The scientific investigation applies a multi-analytical protocol that has shown great suitability for textile research in a museum setting. In fact, recent studies have shown the benefits of applying non-invasive methods, such as digital microscopy, broadband multispectral imaging (MSI) and fibre optic reflectance spectroscopy (FORS) to the study of dyes in historic and archaeological textiles [17–27]. Fluorescence spectroscopy [28, 29] and hyperspectral imaging [30, 31] are also applicable. These non-invasive methods show some limitations, especially in the presence of yellow dyes and mixtures of colourants [25, 32]. However, even when they do not provide conclusive results, useful information can still be gathered to guide strategic sampling with the aim of limiting the number of samples to be taken [33], which is often of extreme importance in a museum context. The access to samples provides the opportunity to apply powerful analytical and imaging techniques, from scanning electron microscopy coupled with energy dispersive X-ray spectrometry (SEM–EDX) for the characterisation of fibres and mordants [34–37], to spectroscopic approaches such as infrared and Raman spectroscopies [38], including surface-enhanced Raman spectroscopy (SERS) [39–41],

and, ultimately, high pressure liquid chromatography coupled to diode array detector and mass spectrometry (HPLC–DAD–MS) [6, 42–52]. Spectral and molecular databases of dye molecules are of particular importance in historic textile studies [42, 46, 47, 53, 54], especially when the presence of both natural and synthetic dyes is suspected [14, 30]. Additionally, the advantages of high resolution and accuracy mass spectrometric methods have been discussed in terms of enhanced structural elucidation and characterisation of dyes and their degradation products [55–58].

Materials and methods

The textiles

Chosen for their temporal spread across the early colonial period and the advent of synthetic dyes and fibres, the selection of these six textiles should not be considered representative of Karen textile production overall, particularly in view of the geographic spread of Karen peoples and their varying levels of access to new materials. Nevertheless, the textiles include tunics, skirts, and a waistcloth of Pwo, Sgaw and Paku production, in addition to representing a broad colour palette and a variety of weaving and dyeing techniques (tabby weave, embroidery, supplementary wefts, ikat, etc.), thus constituting a representative selection. Images of the textiles are provided in Figs. 1 and 2. Additional images and details and sampling locations are provided in Additional file 1: Figures S1–S6.

Accession number As1979,Q.101 (Textile 1–T1)

Collected in the Tenasserim region of lower Myanmar for its extensive use of two species of Job's tears (*Coix* sp.) seeds and presented to the Botanical Department of the British Museum in 1844, this tunic shirt (*hse*) is a beautifully produced part of a Pwo Karen woman's outfit made of two strips of dark blue tabby weave cotton cloth joined along the lengths but leaving spaces for the head and arms. The base is left plain in the upper third and is heavily embellished over the remainder with red, yellow and light green embroidery threads in stem and running stitches, as well as stars and zigzags formed from *Coix* seeds. The five rows of patterning of the lower third are interspersed with appliquéd red cotton bands that extend beyond the borders of the tunic as a thick 'fringe'.

Accession number As.7765 (Textile 2–T2)

Produced by a Sgaw Karen woman before 1870, this skirt-cloth (*ni*) is formed of two mirror-image pieces of warp-faced cotton cloth joined together. The upper and lower edges display four sections of horizontal warp stripes in red, blue, yellow and white. These are followed by

short vertical bands of stripes alternating with sections of zigzags woven with a discontinuous supplementary weft technique in red, yellow, white, green and dark blue-black. The four red and white warp ikat panels of the central section alternate with five sections of warp stripes and discontinuous supplementary weft patterns in geometric shapes. The ikat sections, as seen in this textile, are usually composed of repeated rhomboid, diamond or rectangular patterns, representing a design called "python skin" commonly found among the Sgaw Karen and particularly on skirt-cloths.

Accession number As, + 0.6880 (Textile 3–T3)

Likely to be a Pwo Karen woman's shirt made before 1863, this rich example displays dense patterning with two types of *Coix* seeds, including around the arm and neck holes. The seeds are interspersed with embroidered zigzags, lines and geometric shapes in coloured yarns, using chain and cross stitching. There are narrow red felted wool stripes lining the arm and neck holes and three broad ones around the lower half of the garment. The base textile of the jacket is blue cotton decorated with red, green, orange and white cotton embroidery threads.

Accession number As1919,0717.200 (Textile 4–T4)

A Sgaw Karen piece made before 1886, this warp-faced cotton skirt-cloth comprises numerous warp stripes of varying widths in blue, red, orange and green yarns, as well as four wide "python skin" ikat panels and four narrow undulating lines in blue and white warp ikat.

Accession number As1966,01.481 (Textile 5–T5)

This tunic was probably produced by a Pwo Karen woman. The designs differ from other Pwo examples, such as those of T1 and T3, suggesting that it comes from a different area, or a new fashion associated with a later date. This is partially corroborated by the fact that the reduced number of seeds and extensive, circular embroidery were features of tunics produced by Karens in Thailand in the twentieth century [1].

The tunic is made of black cotton embroidered with yellow, red, green and orange or beige cross, stem and chain stitches, as well as Job's tears seeds of the elongated type, to produce numerous circles set into sections separated by narrow vertical stripes. Embroidery and seeds also mark the edges of the holes for the neck and arms, and around the lower edge is a loosely woven red band that extends beyond the width of the shirt. It is flanked by bands of seeds arranged vertically and in a triangular pattern.

Accession number As1901,0318.217 (Textile 6–T6)

Collected for the Paris world exposition of 1900, this piece, along with many other textiles from Myanmar, was donated to the British Museum in 1901 by the India section committee. Although initially identified as a Paku Karen man's waist cloth, this is more likely to be a woman's headcloth (*hko peu ki*). Made of white cotton warp-faced cloth, each end is elaborately embellished with pink, dark red, green, and dark green felted wool geometric designs in continuous and discontinuous supplementary weft techniques. Warp-twined cotton fringes are interspersed with wool-wrapped fringes that end in pom-poms. Carefully kept as heirloom objects, such headcloths were placed on the head of a bride at her wedding [1].

Scientific methods

The initial stage of the investigation included visual examination of the textiles using digital microscopy to investigate the weaving of the textiles and other details with the aim of identifying colour nuances, possible mixtures of dyes and fading. These observations were followed using broadband MSI and FORS, for the preliminary identification of some of the colourants as well as the mapping of their distribution. The information obtained by MSI and FORS guided strategic sampling during which samples were taken from most colours of the textiles. The non-invasive investigation enabled areas dyed with the same colours to be identified and mapped, so that the representativeness of the samples was ensured [26]. A list of the samples taken is provided in Table 1. Access to loose threads as well as to the inside and back of the textiles facilitated the sampling (Additional file 1: Figures S1–S6). The samples were investigated by optical microscopy (OM) under visible and ultraviolet (UV) light and SEM–EDX for fibre identification, evaluation of possible degradation of the fibres as well as analysis of the elemental composition. Finally, the samples underwent extraction of the dye molecules from the fibres followed by HPLC–DAD–MS/MS analysis for the identification of the dyes at the molecular level. Some of the samples also underwent micro-Raman analysis to characterise inorganic colourants [59].

Digital microscopy (DM)

A Keyence VHX-5000 digital microscope was used to record magnified images (20–200x). The microscope is equipped with a lens VH-Z 20R, an automated stage VHX-S 550E and LED reflected illumination.

Broadband Multispectral Imaging (MSI)

All images were taken using a modified Nikon D7000 camera body with a maximum resolution of 4928 × 3264

(16.2 megapixels). The modification consists of the removal of the inbuilt UV-IR blocking filter, to exploit the full sensitivity of the CMOS sensor (c. 300–1000 nm). The lens used is a Canon EF 50 mm f/1.8II. The camera is operated in fully manual mode. A reference grey scale, comprising a set of Lambertian black, grey and white tiles, is placed in the same plane as the object under investigation. In each case the object is illuminated by two radiation sources symmetrically positioned at approximately 45° with respect to the focal axis of the camera and at about the same height. A filter, or combination of filters, is placed in front of the camera lens in order to select the wavelength range of interest. Combinations of radiation sources and filter(s) enable six sets of images to be acquired: visible-reflected (VIS), ultraviolet-induced visible luminescence (UVL), infrared-reflected (IRR), ultraviolet-reflected (UVR), visible-induced visible luminescence (VIVL) and multiband-reflected (MBR) images. Details are reported in [25, 26].

All images are acquired as RAW images and transformed into 4928 × 3264 pixel resolution images in 16-bit TIF (tagged image file) format, and by turning off all enhancements (e.g. recovery, fill light, blacks, contrast, brightness, clarity, vibrance, saturation, as well as setting the tone-curve to linear). This procedure can be carried out using the camera software or external programs such as Adobe Photoshop. Post-processing procedures for the standardisation and calibration of the VIS, IRR, UVL and UVR images and the creation of infrared-reflected false colour (IRRFC) and ultraviolet-reflected false colour (UVRFC) images are then carried out using “BM_workspace”, a plug-in for Nip2, the open-source graphical user-interface of VIPS, a free image processing software. For details for the post-processing of these images, see the manual on multispectral imaging techniques [60].

Fibre Optic Reflectance Spectroscopy (FORS)

Fibre optic reflectance spectra were recorded with an Avantes (Apeldoorn, The Netherlands) AvaSpec-ULS2048XL-USB2 spectrophotometer equipped with an AvaLight-HAL-S-IND tungsten halogen light source. The detector and light source were connected with a fibre optic bundle to an FCR-7UV200-2–1.5 × 100 probe. In this configuration, light was sent and retrieved by the bundle set at approximately 45° from the surface normal, thus excluding specular reflectance. The spectral range of the detector was 200 - 1160 nm; nevertheless, due to poor blank correction on both the extremes of the range, only the range between 350 and 900 nm was considered; as per the features of the monochromator (slit width 50 μm, grating of UA type with 300 lines/mm) and of the detector (2048 pixels), the best spectra

Table 1 List and description of the samples taken from the textiles under investigations and summary of the results.





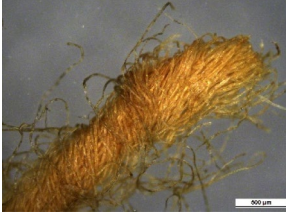
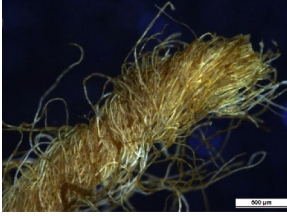

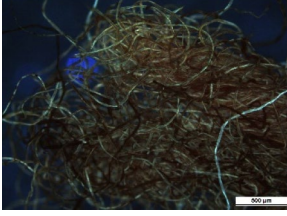

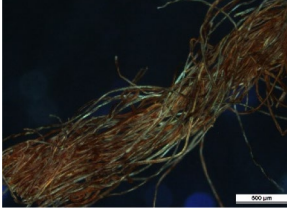
Sample	Colour	Description	Vis-OM	UV-OM	Dyes	Fibres	Mordants
As1979,Q,101 (Textile 1–T1)							
T1_1	Blue/black base	Sample taken from the front of the upper external part of the tunic			Indigo + tannins	Cotton	
T1_2	Light green embroidery	Sample taken from the embroidery thread of the geometrical decoration			Indigo + tannins	Cotton	
T1_3	Yellow embroidery	Sample taken from the embroidery thread of the geometrical decoration			Gamboge (<i>Garcinia</i> sp.)	Cotton	Al
T1_4	Red embroidery	Sample taken from the embroidery thread of the geometrical decoration			<i>Morinda citrifolia</i> (red)	Cotton	Al
T1_5	Red appliquéd band	Sample taken from the edge of the first horizontal band			<i>Morinda citrifolia</i> (red)	Cotton	Al
T1_6	Off-white thread attaching seed	Sample taken from thread attaching <i>Coix</i> seed			Undyed	Cotton	
T1_7	Blue/black base	Sample taken from the front of the upper internal part of the tunic					
As.7765 (Textile 2 – T2)							

Table 1 (continued)

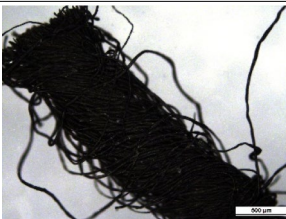


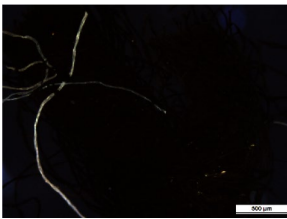
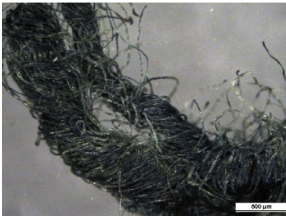
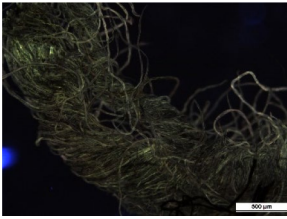


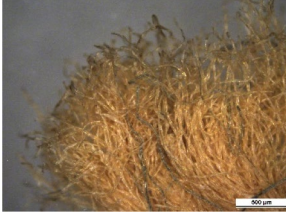





Sample	Colour	Description	Vis-OM	UV-OM	Dyes	Fibres	Mordants
T2_1	Blue/black weft	Sample taken from loose dark blue weft visible in the opening of the skirt			Indigo	Cotton	
T2_2	Blue/black band (warp)	Sample taken from warp used for the upper decorated dark blue band			Indigo	Cotton	
T2_3	Grey/blue-ish band (warp)	Sample taken from warp used for the blueish thin bands			Indigo	Cotton	
T2_4	Green/blue band (warp)	Sample taken from warp used for the greenish thin bands			Indigo + <i>Morinda citrifolia</i> (yellow)	Cotton	
T2_5	Yellow thin band (warp)	Sample taken from warp used for the yellow thin bands			Turmeric (<i>Curcuma</i> sp.)	Cotton	
T2_6	Yellow thick band (warp)	Sample taken from warp used for the yellow thicker bands			Turmeric (<i>Curcuma</i> sp.)	Cotton	
T2_7	Red band (warp)	Sample taken from warp used for the red bands			<i>Morinda citrifolia</i> (red)	Cotton	

Table 1 (continued)

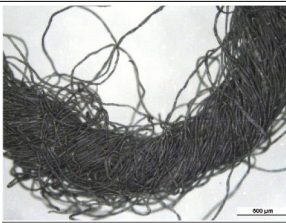


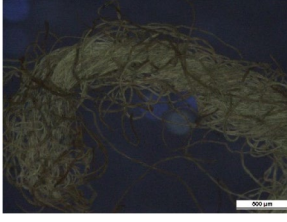
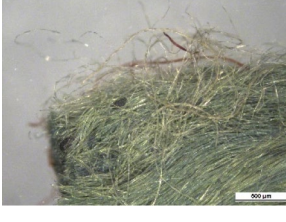
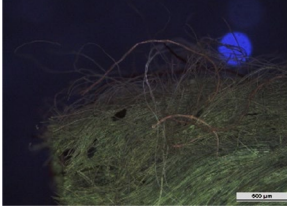




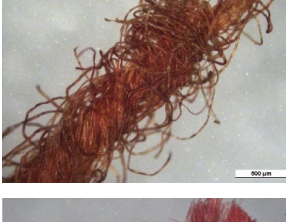
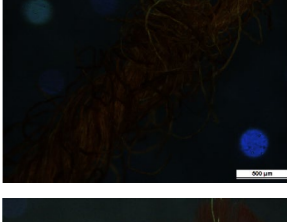
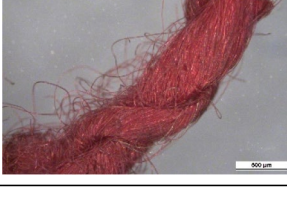
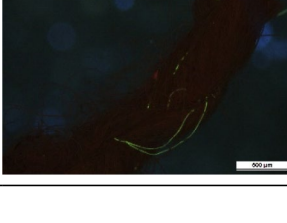
Sample	Colour	Description	Vis-OM	UV-OM	Dyes	Fibres	Mordants
T2_8	Blue/black supplementary weft	Sample taken from supplementary weft used for geometrical decoration			Indigo	Cotton	
T2_9	Light blue supplementary weft	Sample taken from supplementary weft used for geometrical decoration			Indigo + <i>Morinda citrifolia</i> (yellow)	Cotton	
T2_10	Green supplementary weft	Sample taken from supplementary weft used for geometrical decoration			Indigo + <i>Morinda citrifolia</i> (yellow)	Silk	
T2_11	Yellow supplementary weft	Sample taken from supplementary weft used for geometrical decoration			Turmeric (<i>Curcuma</i> sp.)	Cotton	
T2_12	Yellow supplementary weft (fluorescent)	Sample taken from supplementary weft used for geometrical decoration			Turmeric (<i>Curcuma</i> sp.) + Chinese cork tree (<i>Phellodendron chinense</i>)	Silk	
T2_13	Red supplementary weft	Sample taken from supplementary weft used for geometrical decoration			<i>Morinda citrifolia</i> (red)	Cotton	
T2_14	Pink supplementary weft	Sample taken from supplementary weft used for geometrical decoration			Lac dye (<i>Kerria lacca</i>)	Silk	

Table 1 (continued)





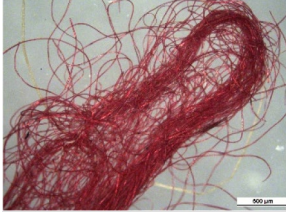
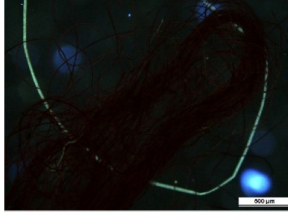
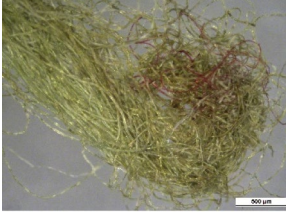
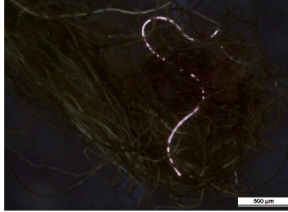
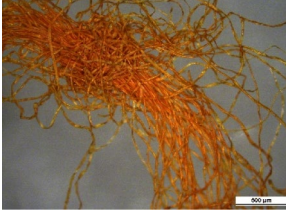
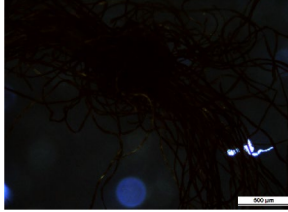
Sample	Colour	Description	Vis-OM	UV-OM	Dyes	Fibres	Mordants
T2_15	White band (warp)	Sample taken from warp used for the white bands			Undyed	Cotton	
T2_16	Blue/black	Sample taken from warp used for the upper decorated dark blue band (internal part of the skirt)					
As, +.6880 (Textile 3–T3)							
T3_1	Blue base	Sample taken from the front of the upper part of the tunic			Indigo	Cotton	
T3_2	Red appliqué band	Sample taken from the red appliqué band (first one from top)			Lac dye (<i>Kerria lacca</i>) + cochineal (probably <i>Dactylopius coccus</i>) + young fustic (<i>Cotinus coggygria</i>)	Felted wool	Sn
T3_3	Red embroidery	Sample taken from the embroidery thread used for the geometrical decoration			Lac dye (<i>Kerria lacca</i>)	Cotton	
T3_4	Green embroidery	Sample taken from the embroidery thread used for the geometrical decoration			Indigo + Chrome yellow/orange	Cotton	
T3_5	Orange embroidery	Sample taken from the embroidery thread used for the geometrical decoration			Lac dye (<i>Kerria lacca</i>) + Chrome yellow/orange	Cotton	
T3_6	White embroidery	Sample taken from the embroidery thread used for the geometrical decoration			Undyed	Cotton	

Table 1 (continued)

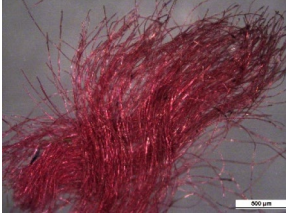

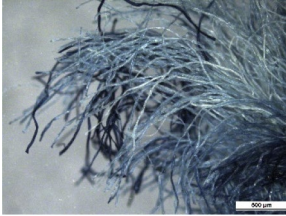
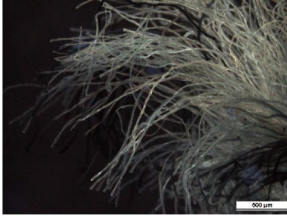

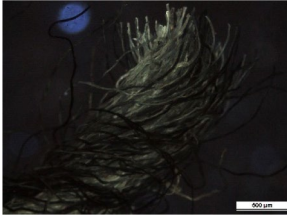
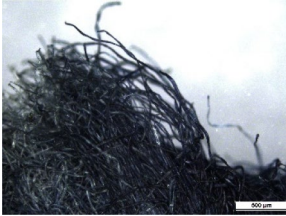
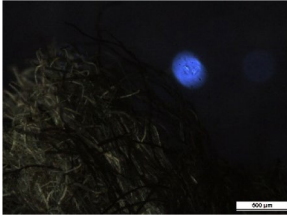

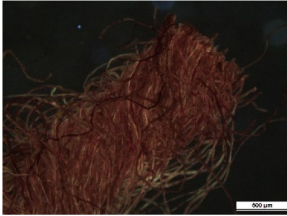
Sample	Colour	Description	Vis-OM	UV-OM	Dyes	Fibres	Mordants
T3_7	Pink stitch	Sample taken from the stitches used to apply the red bands around the arms			Cochineal (<i>probably Dactylopius coccus</i>)	Cotton	
T3_8	White part of appliqué band	Sample taken from the undyed area of the red appliqué band (third one from top)			Undyed	Felted wool	
T3_9	Thread attaching seed	Sample taken from thread attaching <i>Coix</i> seed			Undyed	Cotton	
As1919,0717.200 (Textile 4–T4)							
T4_1	Blue ikat	Sample taken from the middle ikat panel (first one from bottom)			Indigo	Cotton	
T4_2	Blue weft	Sample taken from blue weft			Indigo	Cotton	
T4_3	Blue warp (band)	Sample taken from warp used for the blue bands			Indigo	Cotton	
T4_4	Red warp (band)	Sample taken from warp used for the red bands			<i>Morinda citrifolia</i> (red)	Cotton	

Table 1 (continued)

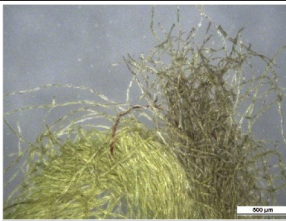
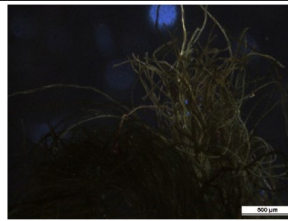

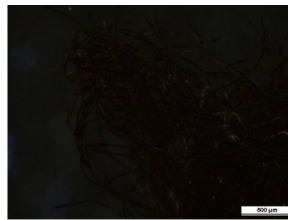
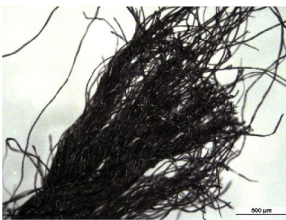
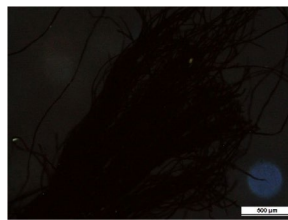

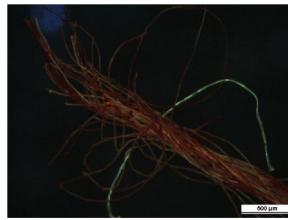
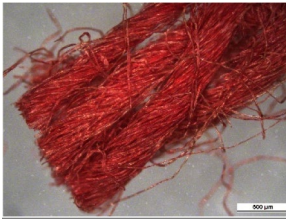
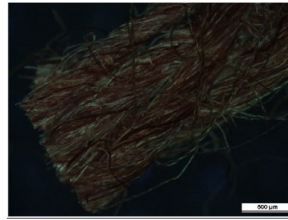
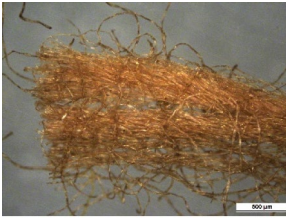

Sample	Colour	Description	Vis-OM	UV-OM	Dyes	Fibres	Mordants
T4_5	Green warp (band)	Sample taken from warp used for the green bands			Indigo + Chrome yellow/orange	Cotton	
T4_6	Orange warp (band)	Sample taken from warp used for the orange bands			<i>Morinda citrifolia</i> (red) + Chrome yellow/orange	Cotton	
T4_7	White ikat	Sample taken from undyed part of the ikat panel (first one from bottom)			Undyed	Cotton	
As1966,01.481 (Textile 5—T5)							
T5_1	Black base	Sample taken from the base of the tunic (external part)			Synthetic black (diamine black family?) + Chrysamine G (CI 22250, synthesised in 1884)	Cotton	
T5_2	Red appliquéd band	Sample taken from the red band in the lower part of the tunic			Synthetic alizarin (CI 58000, synthesised in 1868) + PR53:1 (CI 15585:1, synthesised in 1902)	Cotton	Al
T5_3	Red embroidery	Sample taken from the embroidery thread used for the circular geometrical decoration			Synthetic alizarin (CI 58000, synthesised in 1868) + PR53:1 (CI 15585:1, synthesised in 1902)	Cotton	
T5_4	Beige/orange embroidery	Sample taken from the embroidery thread used for the circular geometrical decoration			Synthetic orange (Congo red class?)	Cotton	

Table 1 (continued)


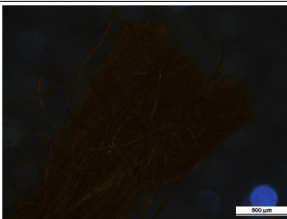
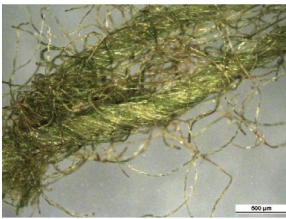
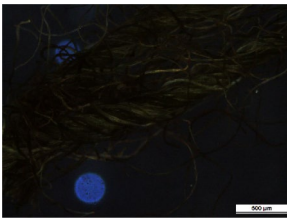

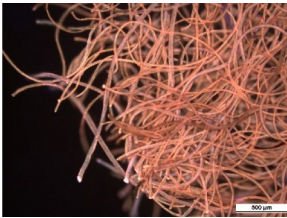
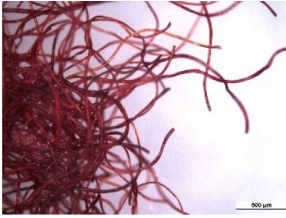
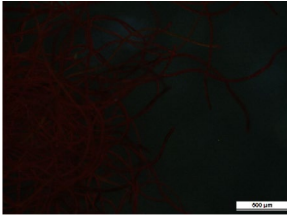
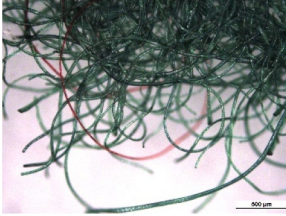
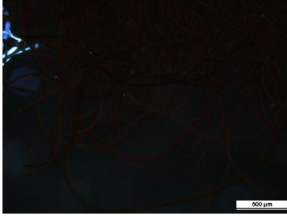

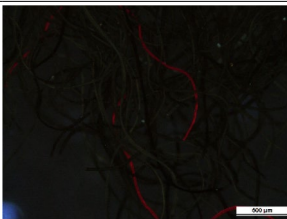

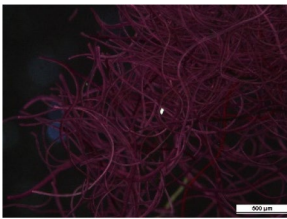

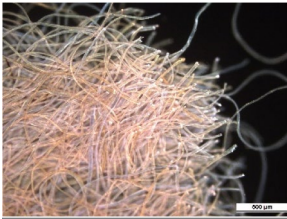
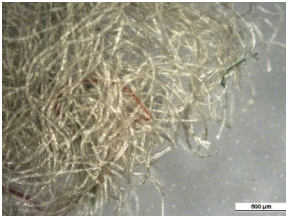
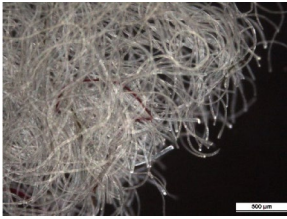
Sample	Colour	Description	Vis-OM	UV-OM	Dyes	Fibres	Mordants
T5_5	Yellow embroidery	Sample taken from the embroidery thread used for the circular geometrical decoration			Chrome yellow (PbCrO ₄)	Cotton	
T5_6	Green embroidery	Sample taken from the embroidery thread used for the circular geometrical decoration			Indigo + Chrome yellow (PbCrO ₄)	Cotton	
T5_7	Light green embroidery	Sample taken from the embroidery thread used for the vertical decoration on the left-hand side			Turmeric (<i>Curcuma</i> sp.) + diamond green B (CI 42000, synthesised in 1877)	Cotton	
T5_8	Off-white thread attaching seed	Sample taken from thread attaching <i>Coix</i> seed			Undyed	Cotton	
As1901,0318.217 (Textile 6–T6)							
T6_1	Bright pink supplementary weft	Sample taken from supplementary weft used for geometrical decoration			Rhodamine B (CI 45170, synthesised in 1887) + Auramine O (CI 41000, synthesised in 1883)	Felted wool	
T6_2	Dark red supplementary weft	Sample taken from supplementary weft used for geometrical decoration			Orange I (CI 14600, synthesised in 1876)	Felted wool	Cr
T6_3	Green supplementary weft	Sample taken from supplementary weft used for geometrical decoration			Diamond green G (CI 42040–1879) + Naphthol yellow S (CI 10316–1879)	Felted wool	

Table 1 (continued)

Sample	Colour	Description	Vis-OM	UV-OM	Dyes	Fibres	Mordants
T6_4	Dark green supplementary weft	Sample taken from supplementary weft used for geometrical decoration			Nitrobenzene/phenol class?	Felted wool	Cr
T6_5	Dark blue supplementary weft	Sample taken from supplementary weft used for geometrical decoration			Crystal violet (Cl 42555, synthesised in 1883)	Felted wool	
T6_6	Salmon pink pompom	Sample taken from pompom			Rhodamine B (Cl 45170, synthesised in 1887) + Auramine O (Cl 41000, synthesised in 1883)	Felted wool	
T6_7	Light green pompom	Sample taken from pompom			Nitrobenzene/phenol class?	Felted wool	
T6_8	White base	Sample taken from the base of the textile base fabric			Undyed	Cotton	
T6_9	Off-white fringe	Sample taken from the tip of one of the fringes			Undyed	Cotton	

Micrographs obtained by optical microscopy under visible (Vis-OM) and ultraviolet (UV-OM) light are included

resolution was 2.4 nm calculated as full width at half maximum (FWHM). Spectra were referenced against the WS-2 reference tile provided by Avantes. The diameter of the investigated area on the sample was approximately 1 mm, obtained by setting the distance between probe and sample at 1 mm. The instrumental parameters were as follows: 100 ms integration time, 5 scans for a total acquisition time of 0.5 s for each spectrum. The whole system was managed by the software AvaSoft 8 for Windows.

Optical microscopy (OM) under visible and UV light

The samples were placed on a glass slide and photographed using a Leica DM 4000 M microscope equipped with a Leica EL 6000 UV light source.

Scanning Electron Microscopy—Energy Dispersive X-ray Spectroscopy (SEM–EDX)

Sample were placed uncoated on an adhesive carbon disc mounted onto an aluminium SEM stub. Examination was carried out with a variable pressure VP SEM (Hitachi

S-3700N) using the backscatter electron (BSE) detector mostly at 16 kV. The working distance was 10 mm. The SEM chamber was only partially evacuated (mostly 40 Pa). For fibre identification, an in-house reference collection of known fibres, both in the form of thin sections and raw fibres collected from accredited sources of silk, wool and cotton, was used in combination with comparisons with literature data [37, 61–63].

The EDX spectra were collected using an Oxford Instruments AZtec EDX spectrometer with a 0 ± 20 keV spectral range, 150 s live time, and 2048 channels. AZtec-Energy analysis software (Oxford Instruments) was used to process the data.

High pressure liquid chromatography – diode array detector – electrospray ionisation – quadrupole – time of flight (HPLC–DAD–ESI–Q–ToF)

The dye extraction was performed using a method published in [37], which briefly consists of a double mild extraction procedure, using dimethylsulphoxide (DMSO) first, and secondly a mixture of methanol/acetone/water/0.5 M oxalic acid 30:30:40:1 (v/v/v/v).

The instrumentation consisted of a 1260 Infinity HPLC (Agilent Technologies), coupled to a 1100 DAD detector (Hewlett-Packard) and to a Quadrupole-Time of Flight tandem mass spectrometer 6530 Infinity Q-ToF detector (Agilent Technologies) by a Jet Stream ESI interface (Agilent Technologies). Separation was achieved using a Zorbax Extend-C18 column (2.1 mm \times 50 mm, 1.8 μ m particle size) with a 0.4 mL/min flow rate and 40 °C column temperature, and a gradient of water with 0.1% formic acid (eluent A) and acetonitrile with 0.1% formic acid (eluent B). The elution gradient was programmed as follows: initial conditions 95% A, followed by a linear gradient to 100% B in 10 min, and held for 2 min. Re-equilibration time for each analysis was 10 min. 5 μ L injection volume was adopted for MS experiments and 10 μ L for MS/MS experiments.

The DAD detector (cell volume 50 μ L) scanned in the range 190–700 nm with 2 nm resolution. The ESI operating conditions were drying gas (N_2 , purity >98%) temperature 350 °C and 10 L/min flow; capillary voltage 4.0 kV; nebulizer gas pressure 40 psig; sheath gas (N_2 , purity >98%) temperature 375 °C and flow 11 L/min. High resolution MS and MS/MS spectra were acquired in both negative and positive mode in the range 100–1700 m/z. The fragmentor was kept at 100 V, nozzle voltage 1000 V, skimmer 65 V, octapole RF 750 V. For the MS/MS experiments, different voltages (from 10 to 50 V) in the collision cell were tested for Collision Induced Dissociation (CID), in order to maximize the information obtained from the fragmentation of the single molecules. The collision gas was N_2 (purity 99.999%). The data were

collected by targeted MS/MS acquisition with an MS scan rate of 1.0 spectra/sec and a MS/MS scan rate of 1.0 spectra/sec. Auto-calibration was performed daily using Agilent tuning mix HP0321 (Agilent Technologies) prepared in 90% water-10% acetonitrile.

MassHunter Workstation Software was used to carry out diode array detector and mass spectrometer control, data acquisition, and data analysis.

Reference samples of dyes were used for identification. For natural dyes, the reader is referred to [25, 38, 46], in which the relevant reference samples are discussed. For synthetic dyes, relevant reference samples were available as both powders and dyed wool samples from the CHARISMA project (2009–2014) funded by the European Union FP 7 Research Infrastructures programme (CHARISMA Grant Agreement no. 228330). Additional samples of historic dyed fabrics were available from copies of two historic books: “A Manual of Dyeing: For the Use of Practical Dyers, Manufacturers, Students, and All Interested in the Art of Dyeing (Specimen of dyed fabrics)” by Knecht, E., C. Rawson, and R. Loewenthal published in London in 1893 [38] and “The Coal Tar Colours of Farbwerke Vorm. Meister Lucius & Brüning, Hoechst on the Maine, Germany—A General Part” by Farbwerke Vorm Meister Lucius & Brüning published in 1896. Some literature data on these samples are available [42, 64, 65].

Results and discussion

Broadband multispectral imaging (MSI)

As described in recent publications [19, 25, 26], MSI is a powerful tool for mapping the distribution of colouring materials on textiles and, in some cases, for obtaining a reliable preliminary indication of the dyes used. The full set of images obtained for the six textiles is reported in Additional file 1 (Figures S7–S12) and the most interesting observations are described below.

All the dyed components in T1 are infrared transparent to some degree, appearing shades of red, orange and yellow in the IRRFC image (Fig. 3 and Additional file 1: Figure S7). Additionally, the very dark blue base of the tunic appears white in the MBR image, suggesting the presence of indigo [26]. Under UV the dyed areas absorb with no characteristic features either in reflectance, false colour or luminescence images, preventing preliminary considerations on the red and yellow areas from being obtained. The *Coix* seeds, however, show variable UV-induced visible luminescence properties, with some seeds appearing light blue and other seeds appearing yellowish in the UVL image. Three types of seeds are used in this textile, i.e., short and long elongated ones as well as thicker skittle-shaped ones (details in Additional file 1: Figure S13), but the differences in the UVL images

appear unrelated to the type of seed. High magnification images by digital microscopy enabled a correlation to be established between the preservation state of the surface of the seeds and the colour of the luminescence, which shifts from bluish to yellowish with increasing degradation of the surface (Fig. 3 and Additional file 1: Figure S13). The luminescence properties of the seeds are linked to the physicochemical composition of their surface, which is mostly a mixture of polysaccharides and proteins [66]. In general, proteinaceous materials exhibit bluish luminescence under UV illumination, whereas lignocellulosic materials appear yellow-beige or slightly pink [26]. Hence, the change in colour under UV might be linked to a depletion in proteins on the surface of the most degraded seeds. However, dedicated work would be needed to further explore this point.

T2 exhibits intricate decoration patterns, whose details are further elucidated under different wavelengths of light (Additional file 1: Figure S8). As shown in Fig. 4, two of the vertical stripes in the decorated horizontal bands exhibit a bright yellow luminescence in the UVL image, revealing that the dye used in these threads is different from that used in the other yellow areas, such as the yellow stripes flanking the ikat, which exhibit a weaker, duller yellow luminescence. The difference between these two dye sources would have been extremely difficult to spot with the naked eye, highlighting the importance of MSI in this type of investigation. Differences were observed in the UVRFC image between the dark red areas and the areas of pink embroidery, which appeared yellow and green respectively, pointing to the use of a different dye source. Additionally, the blue and green areas appear different shades of red and pink in the IRRFC image, an indication of the likely presence of indigo, which is confirmed by the bright white appearance of these areas in the MBR image [26]. This effect is particularly visible in the background of the central band (Fig. 4 and Additional file 1: Figure S8).

The most striking feature of T3 are the bright red stripes applied horizontally and lining the arm holes and collar of the garment. These are strongly luminescent and appear bright red/orange in the UVL image (Additional file 1: Figure S9). The garment also features embellishment with *Coix* seeds, which behave similarly under UV to that observed for T1, as well as embroidered details in green, orange and red threads. The latter areas absorb UV radiation and appear dark, whereas the green areas show a weak red emission in the UVL image, which is unusual for natural dyes. The presence of indigo is again indicated in the blue base textile of the shirt by its red appearance in the IRRFC and white appearance in the MBR images, respectively. Similar observations are drawn for T4 (Additional file 1: Figure S10). Interestingly, in the blue

ikat areas, the stripes appear green in the UVRFC image rather than the deeper green/black response of the dark blue areas, an effect related to the lower concentration of the dye in these areas [67]. The bright orange and lime green coloured warp threads exhibit similar behaviour to those observed in T3, suggesting similar colourants may have been used in these areas.

The black base textile of T5 partially absorbs IR radiation (Fig. 5e and Additional file 1: Figure S11) and appears dark in the MBR image, in contrast to indigo-containing areas, which typically appear white or grey [25, 26]. The deep red colour exhibited in the IRRFC image, however, still suggests that this material is partially IR transparent. It is the comparison between the MBR, IRR and IRRFC images that allows the important characterisation of the black dye as a non-indigo containing material to be made. As with previous textiles, the red colour absorbs UV radiation, and appears yellow in IRRFC and green in UVRFC, general features that make it difficult to restrict the pool of possible candidates for this colourant. Features of interest in the UVL image include the diverse luminescence behaviour exhibited by the *Coix* seeds, as seen in T1 and T3. Additionally, whilst the areas of orange/beige circular embroidery exhibit orange luminescence, a distinctive bright yellow luminescence is observed in two of the three vertical cross decorations, suggesting that different dyes were used in these otherwise similar areas. Interestingly, the bright yellow areas of circular embroidery completely absorb UV radiation, which is unusual for natural yellow dyes and may be indicative of a synthetic source of the colourant. These areas of yellow also appear a distinctive red colour in the UVRFC image. Finally, the green areas also display a weak red luminescence in the UVL image, as observed for T3 and T4. They appear pink in the IRRFC and white in MBR images, suggesting the presence of indigo.

The use of synthetic dyes in T6 was highly suspected, particularly in areas of bright colour. Amongst these, the bright pink and salmon pink areas are highly fluorescent under UV light (Fig. 6 and Additional file 1: Figure S12), with bright orange luminescence reminiscent of the behaviour of the xanthene class of synthetic dyes, such as rhodamine or erythrosine. Other areas did not exhibit luminescence or other features that would be useful for a preliminary identification. The light and dark green (almost black) colours, however, behave very differently. Although both absorb in the UV range, the dark green partially absorbs in the IR range as well, whereas the light green does not. This would suggest that indigo is unlikely to be present in the dark green areas. The small areas of bright blue are also infrared transparent and appear bright red and green in the IRRFC and UVRFC images, respectively. Although indigo cannot be excluded

in these areas, the hue makes it more in-keeping with a synthetic colourant.

Fibre Optic Reflectance Spectroscopy (FORS)

Overall, MSI enabled preliminary indications of the nature of some dyes to be obtained as well as a sense of which areas might be dyed with similar or different dyes, even when they exhibit very similar colourations under visible light. FORS was therefore used to confirm these observations, by exploiting the possibility of acquiring spectra from multiple areas within the same textile in a short amount of time. All spectra are reported in Additional file 1 (Figures S14–S19) and representative spectra and results are discussed below.

The dark blue background of T1, the blue warps and wefts of T2, the deep blue background of T3 and the blue areas of T4 show reflectance spectra with a steep inflection centred at ca. 700–720 nm, some of which are represented in Fig. 7a. This characteristic feature enables the presence of indigo to be confirmed in these areas [68]. However, some differences are present. In particular, for T1 the reflectance curve, rather than levelling out above 750 nm, as is typical for indigo, continues to increase steeply and with a slightly different gradient, probably indicating the presence of tannins, for which similar observations are reported [19]. The blue of T3 shows an absorption feature between 800 and 900 nm, which is not straightforward to explain, but is likely to be related to the concentration of indigo, and has been observed in other indigo-dyed textiles [30].

The black background of T5 is confirmed not to contain indigo, but the reflectance spectrum, which exhibits a gentle increase above 700 nm, is common to

many dark colours, which, although dark in the visible region, tend to transparency in the infrared (Fig. 7a), thus not enabling further hypothesis to be made on the nature of the dye(s) used. By contrast, the dark blue colour of T6 produces a spectrum with a broad absorption between 530 and 590 nm, which points towards the possible presence of methyl/crystal violet formulations [54, 69].

The green areas of T2, T3, T4 and T5 show the typical reflectance curve of indigo mixed with a yellow colourant, as inferred by the absorption features exhibited at ca. 420–440 nm (Fig. 7b). Very little can be speculated from FORS data on the source of yellow dyes [32]. However, the spectra show that the yellow colourant in T2 is probably different from the one in T3 and T4. By contrast, the green areas of T3 and T4 produce identical spectra, suggesting that similar colourants were used. The yellow component of the green areas of T5 is different and does not show any similarities with yellow natural dyes [25]. The inflection point at ca. 520 nm and the broad and shallow absorption feature between 420 and 480 nm point towards the use of a different type of yellow colourant. The spectrum obtained from the bright green areas of T6 shows two main absorption areas, a broader one centred between ca. 420 and 460 nm and a second one centred at ca. 635 nm with a shoulder at ca. 580 nm (Fig. 7b), which is consistent with data reported on triarylmethane green dyes [54]. By contrast, the dark green areas of T6 produce a spectrum that is difficult to interpret, with less resolved absorption features and an inflection point at ca. 750 nm, which does not enable further considerations to be formulated. Similarly, the spectra obtained from the very light green colours of T1 and T6 are not informative, most probably due to the very low concentration of the dyes in these areas.

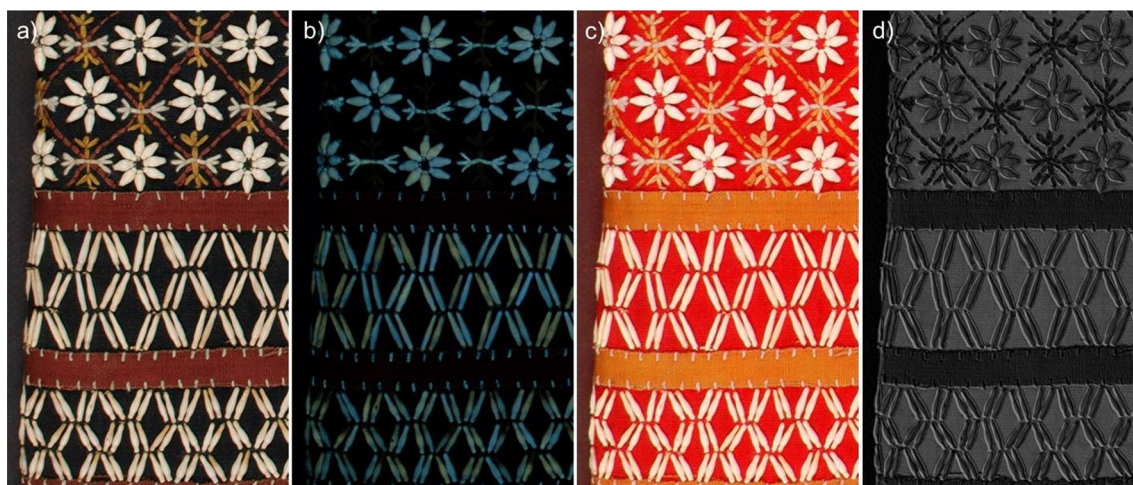


Fig. 3 a VIS, b UVL, c IRRFC, d MBR images of a detail of Textile 1 (accession number As1979,Q.101). © The Trustees of the British Museum

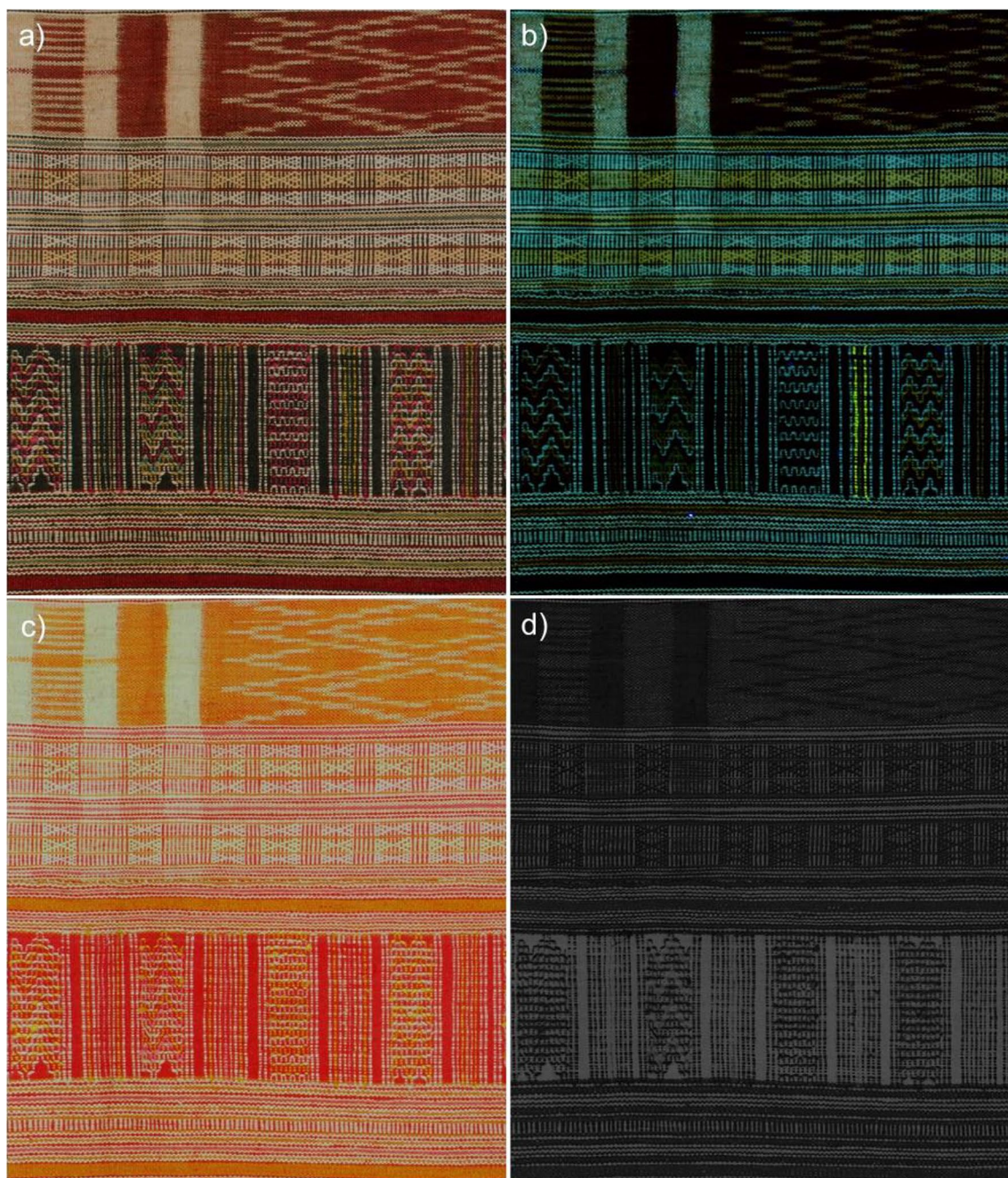


Fig. 4 a VIS, b UVL, c IRRFC, d MBR images of a detail of Textile 2 (accession number As.7765). © The Trustees of the British Museum

Although, as noted, the FORS spectra for the yellow areas are not conclusive, there is sufficient evidence to suggest that the yellow dyes in T1 and T2 are probably different and that two different yellow dyes are likely to be present in T2, as suggested by the slightly different absorption bands at ca. 420–440 nm (Fig. 7c), and as inferred by MSI. It is worth noting that some of the yellow spectra show an inflection around 700 nm. This

is a contribution from the indigo present underneath or next to the yellow threads targeted by FORS. The spectrum obtained for the yellow areas of T5 show the same characteristics described for the green areas of the same textile. T5 also includes some beige/orange embroidered areas, for which a spectrum showing a possible mixture of yellow and red dyes is obtained, as suggested by the double absorption features at ca. 430 and 515 nm.

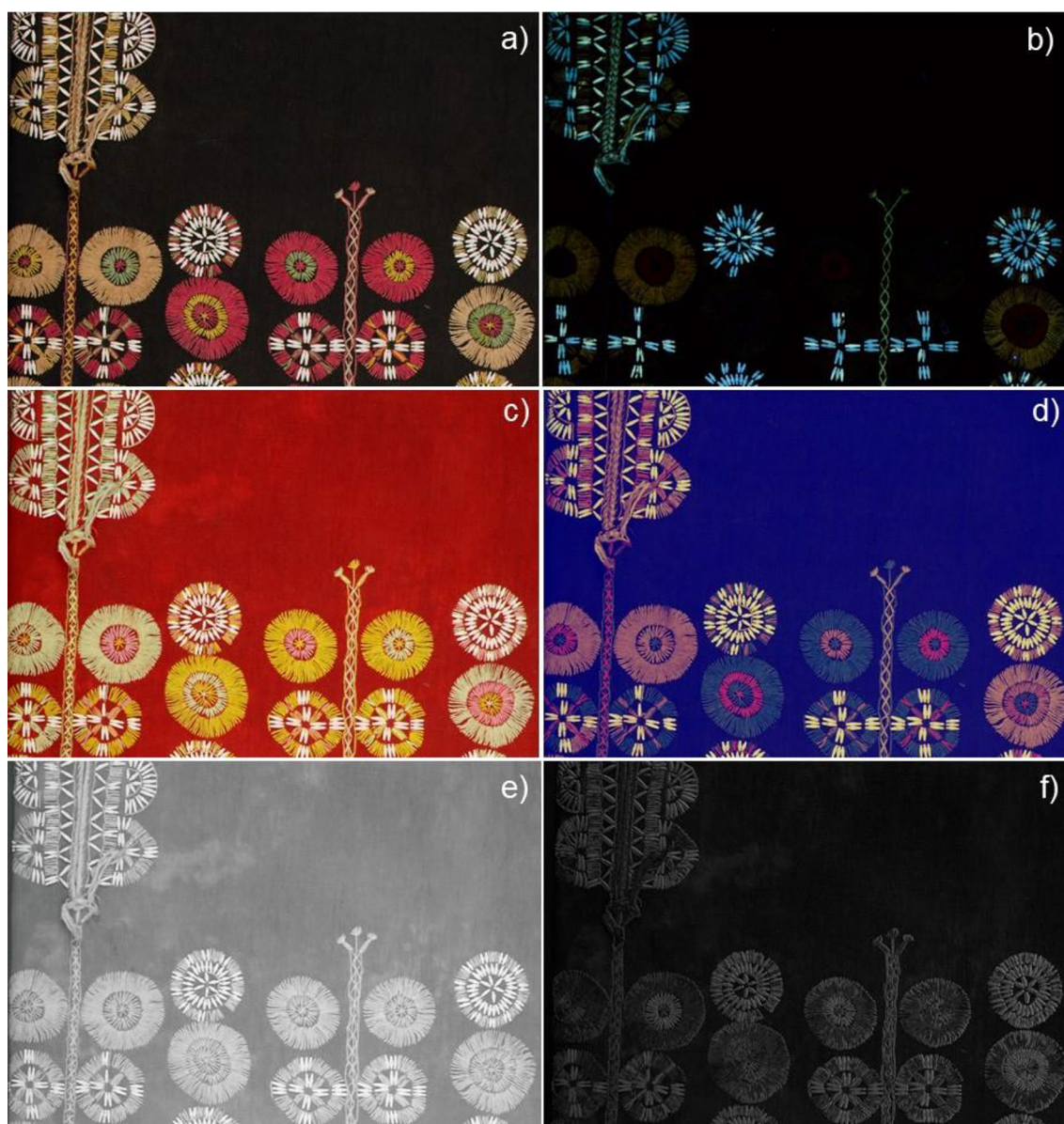


Fig. 5 a VIS, b UVL, c IRRFC, d UVRFC, e IRR and f MBR images of a detail of Textile 5 (accession number As1966,01.481). © The Trustees of the British Museum

The bright orange areas of T3 and T4 yield a spectrum with an inflection point at ca. 560 nm, not particularly indicative of a specific dye or dye class.

All the red areas in T1, T2 and T4 produce the same spectrum (Fig. 7d), which is unstructured and shows an inflection point at ca. 560 nm. This is not specific enough to indicate a particular dye. By contrast, the bright pink areas of T2 and the dark red areas of T3 produce structured spectra with two distinct absorption bands at ca. 525 and 565 nm, which are typical of insect-based red dyes [70]. T3 also includes bright orange/red wool felted

bands, whose spectrum shows less evident structure. Nevertheless, three small absorption features around 485, 520 and 555 nm are noted, and may suggest that a mixture of dyes is present. The spectra for the red areas of T5 show even less defined features that do not allow the possible dyes sources to be narrowed down. The dark red of T6 exhibits a very sharp sigmoidal curve with an inflection point at ca. 620 nm, which is uncommon for natural red dyes and points towards the possible presence of a synthetic colourant. The bright pink areas of T6 produce a characteristic spectrum with an absorption

band at 555 nm and a shoulder at 520 nm followed by a steep increase in reflection and a gradual decrease above 610 nm. These features are found in synthetic xanthenes dyes [54].

High pressure liquid chromatography – diode array detector – electrospray ionisation – quadrupole – time of flight (HPLC–DAD–ESI–Q–ToF)

Guided by the information gathered non-invasively, especially regarding areas that are likely to be coloured with similar dyes, 45 samples were taken as representative of the various colours found in the 6 textiles. The results obtained by HPLC analysis as well as the information obtained about the fibres and mordants used are summarised in Table 1.

The results show that the dark blue colour of T1 was obtained by using a source of indigo and a source of hydrolysable tannins, as indicated by the detection of indigotin, indirubin and ellagic acid (Fig. 8a). The exact plant sources are not identifiable. The light green

embroidery threads were also dyed with indigo and tannins, constituting an example of very different colour shades obtainable with the same materials. The red embroidery threads and the horizontal bands were dyed with the red dye extracted from *Morinda citrifolia*. Morindone (1,2,5-trihydroxy-6-methyl-anthraquinone) is a molecular marker for this dye. However, morindin (morindone primveroside) was also detected together with several additional anthraquinones present as minor components, as partially discussed in other studies [6, 38, 71] (Fig. 8b). The yellow embroidery threads were found to be dyed with gamboge (*Garcinia* sp.). Morelloflavone, its sulphonated derivative, morellic acid and isomorellic acid were the main components detected (Fig. 8c). Many additional molecules were present as minor components, representative of both the colourant and resinous fractions of gamboge [46]. The full characterisation of these molecules is beyond the scope of this article. The results reveal a palette of natural dyes in agreement with local plant sources, with the finding of gamboge being particularly interesting, as this dye is often mentioned as an early source of yellow for Southeast Asian textiles [5, 7], but this is the first scientific evidence of its actual use on a historical textile at the best of our knowledge.

For T2, all red areas were dyed with *Morinda* red. As discussed in the previous sections, some bright pink supplementary wefts were present in the geometric decoration. Laccic acids A, B, C, D and E as well as xantholaccic acids A and B were identified (Fig. 9a), revealing that lac dye, probably from the *Kerria lacca* insect, is the source of the pink colour [46], in agreement with the FORS data. Turmeric (*Curcuma longa*) was used in the yellow horizontal bands and most yellow supplementary wefts, as proved by the presence of curcumin, demethoxycurcumin and bisdemethoxycurcumin in the samples taken from these areas (Fig. 9b), and in agreement with the UV-induced visible luminescence observed in these areas [25]. The green and light blue supplementary wefts in the decoration pattern and the greenish/blueish thin bands were dyed with a yellow flavonoid dye mixed with indigo. Two glycosylated flavonoids were detected with $[M-H]^- = 563.1406$ and 593.1512 m/z respectively (Fig. 9c). The tandem mass spectra showed the typical fragmentation of C-glycosides [72, 73], suggesting that these two molecules are the 6,8-C-hexosyl-C-pentoside and 6,8-di-C-glucoside of a molecule with $[M-H]^- = 269.0455$ m/z (Additional file 1: Figure S20). These compounds have been observed in previous studies and have been tentatively related to the yellow dye extracted from the bark of the *Morinda citrifolia* plant, for which morindone would be the corresponding aglycone [38]. However, they also appear in the extracts of the leaves of *Viola yedoensis* [73], for which apigenin would

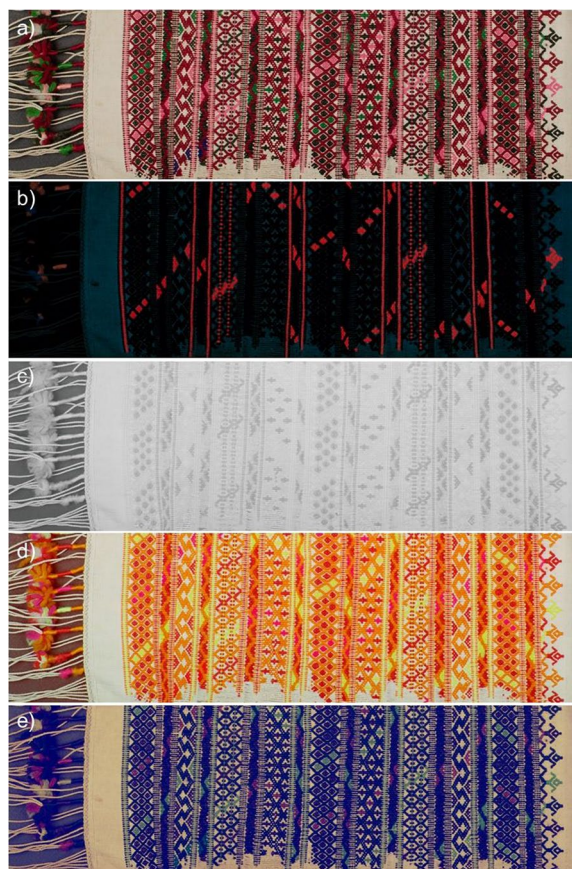


Fig. 6 a VIS, b UVL, c IRR, d IRRFC, e UVRFC images of a detail of Textile 6 (accession number As1901,0318.217). © The Trustees of the British Museum

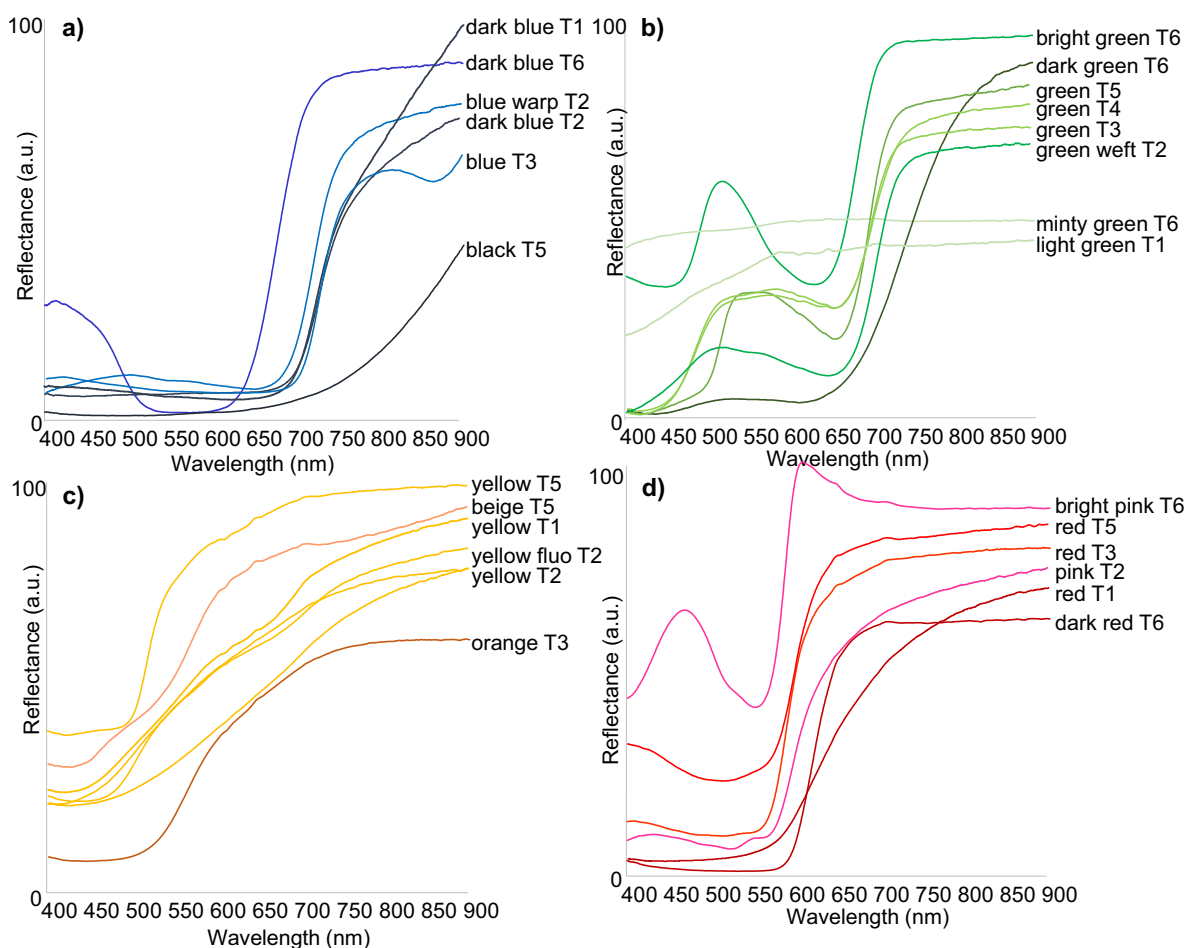


Fig. 7 Representative reflectance spectra obtained by FORS analysis of the **a** blue and black, **b** green, **c** orange and yellow, **d** red and pink areas of the textiles under investigation

be the corresponding aglycone. Additional molecules are reported in the extracts of *Viola yedoensis* that were not detected in these samples. Therefore, the use of *Morinda* yellow seems more likely in this case, especially considering the geographical origin. However, an ultimate identification would require reference samples of this dye, which are not currently available. MSI suggested that another type of yellow dye is present in the supplementary wefts used to create the vertical stripes in the decorative pattern, as suggested by a bright UV-induced visible luminescence exhibited (Fig. 4b). This was identified as the dye extracted from the bark of the Chinese cork tree (*Phellodendron chinense*), which is a typical Chinese dye [46] with the characteristic of emitting a bright yellow UV-induced luminescence [25]. Berberine, hydroxyberberine and jatrorrhizine were identified as the main alkaloids present (Fig. 9d). The absence of palmatine enables this dye to be differentiated from its Japanese relative extracted from the amur cork tree (*Phellodendron amurense*) [46, 74]. The Chinese cork tree dye was mixed with

turmeric. With indigo being the dye used in all dark blue, blue and green details, it is concluded that only natural dyes are present in this textile.

The base blue textile of T3 was confirmed to be dyed with indigo. The dark red embroidery threads were dyed with lac dye, whereas the analysis of the bright green and orange threads revealed the presence of indigo and lac dye respectively, but no trace of an organic yellow component. Further investigation using SEM–EDX and Raman spectroscopy revealed that the additional colourant present in these threads is chrome orange (basic lead chromate – Pb_2CrO_5). The details of this discovery are reported in a separate publication [59]. The bright red/orange felt bands were found to be dyed with a mixture of lac dye, cochineal (probably *Dactylopius coccus*) and young fustic (*Cotinus coggygria*), as inferred by the detection of laccaic acids, carminic acid, fustin, fisetin and sulfuretin [46] (Additional file 1: Figure S21). The felt was stitched with a pink cotton thread that was also dyed with cochineal. The results show the use of

dyes and other colourants not typical of Myanmar, such as cochineal, young fustic and chrome-based colourants, clearly suggesting the use of materials imported from Europe for the creation of this textile.

Indigo and *Morinda* red were identified as the dyes used for the blue and red areas of T4, respectively. The bright orange and green colours were again obtained with chrome orange overdyed with *Morinda* red and indigo, respectively [59], showing the use of typical Southeast Asian natural dyes as well as atypical dyeing methods.

The black base of T5 produced a complex mixture of molecules (Fig. 10a). The two molecules at high retention times were identified as the disazo yellow dye chrysamine G (direct yellow 1; CI 22250) and its degradation product or synthetic by-product without one of the diazo substituents. These molecules produced negative deprotonated ions $[M-H]^-$ at m/z 481.1154 and 317.0932 respectively, and positive protonated ions $[M+H]^+$ at m/z 483.1299 and 319.1077 respectively. The tandem mass spectrum of chrysamine G is reported in Additional file 1 (Figure S22). The other molecules in the chromatogram appeared to be related to a synthetic black dye. The most abundant molecule corresponded to $[M+H]^+ = 727.1742$ m/z and the corresponding tandem mass spectrum (Additional file 1: Figure S23) shows several fragment ions in common with other molecules identified in a sample present in the book “A Manual of dyeing” and referred to as Shade 19 [75]. This dye is described as an ingrain colour obtained by dyeing with diamine black R or B followed by diazotisation and on-fibre development with phenylene-diamine, resorcinol, or a mixture of resorcinol and β -naphthol [76]. It was difficult to hypothesise an exact molecular structure for these molecules, but a connection with the diamine black class is possible for this colour. Alizarin (CI 58000) was identified in all red areas together with flavopurpurin and anthrapurpurin, which are typically found as synthetic by-products of alizarin [6, 42] (Fig. 10b). Another red molecule was identified as the monoazo Pigment Red 53:1 (CI 15585:1), according to its $[M-H]^-$ at m/z 375.0212 and its tandem mass spectrum (Additional file 1: Figure S24) matching a reference sample of this dye.

The orange/beige dye used in the embroidery was also found to be synthetic. Several high molecular weight molecules were detected, mostly producing doubly charged negative ions (Fig. 10c). One of these molecules was identified as a degradation product of Congo red (CI 22120), according to its $[M-H]^-$ at m/z 675.1126, $[M-2H]^{2-}$ at m/z 337.0527 and MS fragmentation in agreement with literature data [55]. The tandem mass spectra of the other molecules (Additional file 1: Figure S25) share several fragment ions with this molecule, in particular m/z 427.0840, 363.1232, 247.0170 and

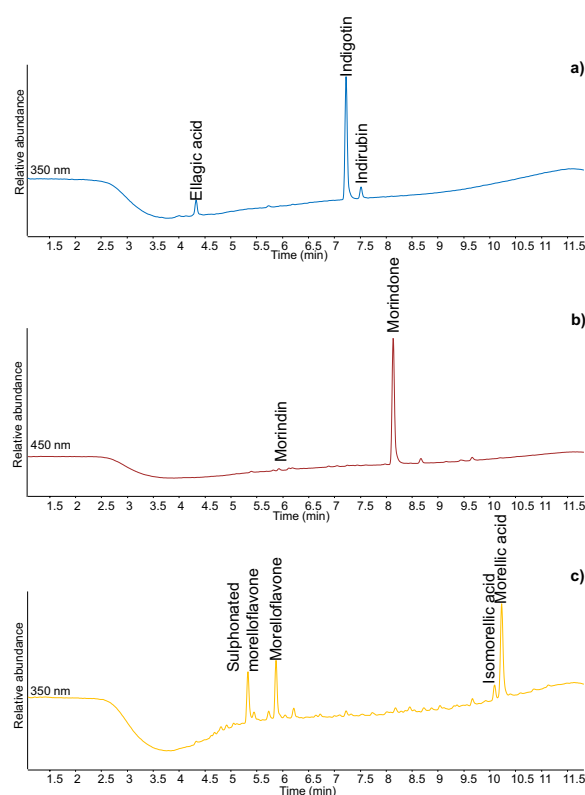


Fig. 8 DAD chromatograms obtained by HPLC-DAD-MS/MS analysis of **a** T1_1, **b** T1_4 and **c** T1_3. Refer to Table 1 for sample labels

182.0484, indicating that these compounds belong to the same molecular class with additional substituents on the structure. The ultimate identification was not obtained, but it appears reasonable to hypothesise that this dye belongs to the Congo red class and was probably developed with an ingrain process similarly to what described for the black colour of the same textile.

The yellow areas were dyed with chrome yellow ($PbCrO_4$) and the bright green areas were dyed with a mixture of chrome yellow and indigo [59]. MSI pointed out that the light green vertical embroidery is dyed differently from the other yellow threads. In fact, the natural yellow dye turmeric was found mixed with the synthetic dye diamond green B (CI 42000), identified by the presence of the intact molecule ($[M]^+ = m/z$ 329.2012) and its demethylated derivatives [14] (Fig. 10d).

For T6, the two shades of pink (bright and salmon) were obtained with a mixture of rhodamine B (CI 45170) and auramine O (CI 41000) (Fig. 11a). Rhodamine B ($[M]^+ = m/z$ 443.2323) was detected together with the deethylated derivatives [77]. Auramine O ($[M]^+ = m/z$ 268.1808) was detected together with the demethylated and bisdemethylated (two isomers) compounds.

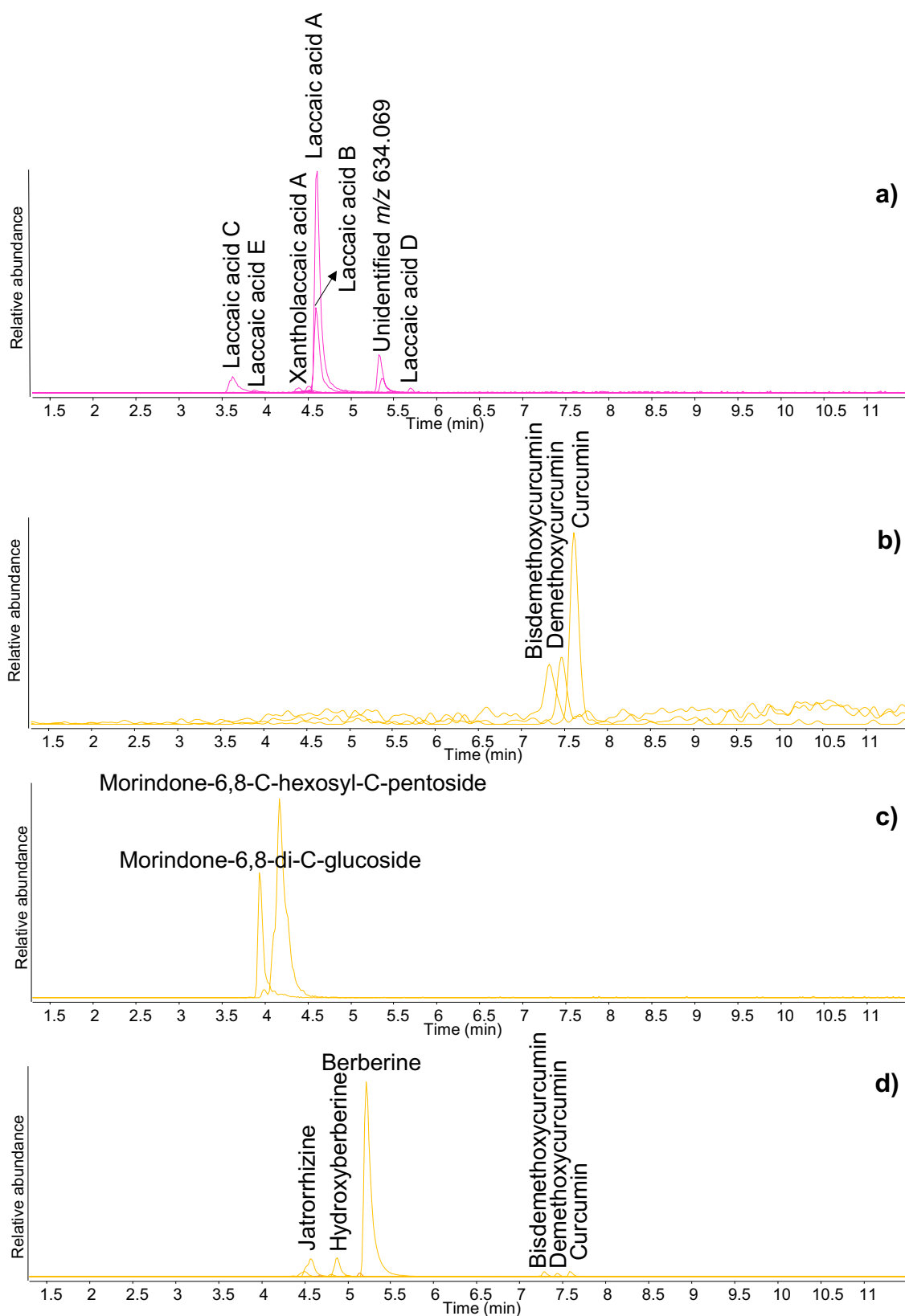


Fig. 9 Extracted ion chromatograms obtained by HPLC–DAD–MS/MS analysis of **a** T2_14, **b** T2_6, **c** T2_4 (all negative ionisation) and **d** T2_12 (positive ionisation). Refer to Table 1 for sample labels

Additionally, Michler's ketone ($[M]^+ = m/z$ 269.1648) and its corresponding demethylated homologues were detected as well as another series of non-fully identified compounds with $[M]^+ = m/z$ 254.1414 ($C_{16}H_{18}N_2O^+$), 240.1257 ($C_{15}H_{16}N_2O^+$) and 226.1102 ($C_{14}H_{14}N_2O^+$) still related to the auramine structure (Fig. 11a). Dark red was obtained by using orange I (CI 14600), identified as a single molecule with $[M-H]^-$ at m/z 327.0445 (Fig. 11b). The bright green areas contain a mixture of diamond green G (CI 42040) and naphthol yellow S (CI 10316). Diamond green G ($[M]^+ = m/z$ 385.2638) was identified in positive ionisation mode together with a series of demethylated and deethylated derivatives [78], whereas naphthol yellow S ($[M-H]^- = m/z$ 312.9772) was identified in negative ionisation mode (Fig. 11c). The dark blue/purple details contain crystal violet (CI 42555), identified as a series of N-methylated pararosanilines, including the N-hexamethylated compound ($[M]^+ = m/z$ 372.2434) (Fig. 11d). The identification of the dyes in the light and dark green areas was more challenging, due to the lack of matches with available reference samples. Some of the molecules appeared in both samples and seemed to be related to nitrobenzene/phenol/catechol structures (Additional file 1: Figure S26), but further elucidation was not obtained.

Scanning electron microscopy—energy dispersive X-ray spectroscopy (SEM–EDX)

SEM–EDX was applied with the aim to identify the fibres and, in some cases, evaluate their preservation state as well as to study the inorganic components possibly present, such as mordants and other additives.

T1 was confirmed to be entirely made of cotton (Fig. 12a), and the *Coix* seeds were also attached with undyed cotton threads. The fibres of the red horizontal band were found to be particularly rich in aluminium (Fig. 13a). Aluminium mordanting is needed to dye with *Morinda* red [71], but such a high level of aluminium is rarely detected on alum-treated fibres [37, 79]. This might point towards the use of a vegetable aluminium-rich mordant rather than a mineral one. For example, the leaves of the *Symplocos* sp. plant are reported as a traditional mordant specifically used for dyeing with *M. citrifolia* [4]. However, specific experiments would be needed to explore potential differences in aluminium concentration caused by the use of plant-based and mineral-based mordants. Lower but significant levels of aluminium were found in the other red and yellow areas. The significance of Al was evaluated based on its ratio over Si, as these two elements are commonly found in dirt and dust as well. An Al/Si ratio close to 1 can be taken as an indication of Al as a mordant [37, 79] (Fig. 13b), whereas, when Al is present as a contaminant, the Al/Si ratio is generally close to 0.5 [36] (Fig. 13c).

T2 was also confirmed to be made of cotton except for the bright pink and bright yellow supplementary wefts, which were made of silk (Fig. 12b). These threads were found to be dyed with lac dye and Chinese cork tree respectively, differently from the rest of the textile. The presence of silk and the Chinese cork tree dye supports a possible import of these threads from China. Indications of mordanting was more challenging for this textile, as the presence of Al was not obvious, although expected for dyes such as *Morinda* red and lac dye (Fig. 13c).

T3 was made of cotton threads, apart from the appliquéd red/orange bands, which were confirmed to be made of felted wool (Fig. 12c). EDX spectra of these red fibres revealed the presence of tin (Fig. 13d). Tin mordanting was a common practice in Europe and was used to confer brightness to red colours [9]. This, in addition to the identification of European dyes, further supports the import of this felted wool cloth from Europe. The orange and green embroidery threads showed the presence of inorganic particles rich in Pb and Cr (Figs. 12d and 13e), which was a first indication of the presence of the inorganic colourant chrome orange (Pb_2CrO_5) in these threads, as confirmed by Raman analysis [59].

T4 was entirely made of cotton. Although mordanting was expected in the red areas dyed with *Morinda* red, a clear indication was not obtained. The green and orange areas showed the presence of Pb and Cr, as discussed for T3 [59].

T5 was also entirely made of cotton, although the different quality of the threads was particularly evident. The black base textile was made with highly twisted and compressed cotton yarns with very flattened and broad fibres (Fig. 12e), whereas the embroidery stitches were hardly spun. Very fine cotton fibres were used for the loosely woven red horizontal band (Fig. 12f), probably an indication of the high quality of this textile. Al mordanting was also evident from the EDX spectra of these fibres. The bright yellow and bright green areas showed a fine dispersion of particles rich in Pb and Cr [59].

The base cloth of T6 was confirmed to be made of cotton and all the embroidery threads were made of wool. Chromium was detected in the EDX spectra of the dark red and dark green threads (Fig. 13f). Mordanting with chromium was a common method used in combination with synthetic dyes to obtain dark shades [76]. This mordant justifies the presence of the orange dye in the dark red threads, as this was darkened using chromium.

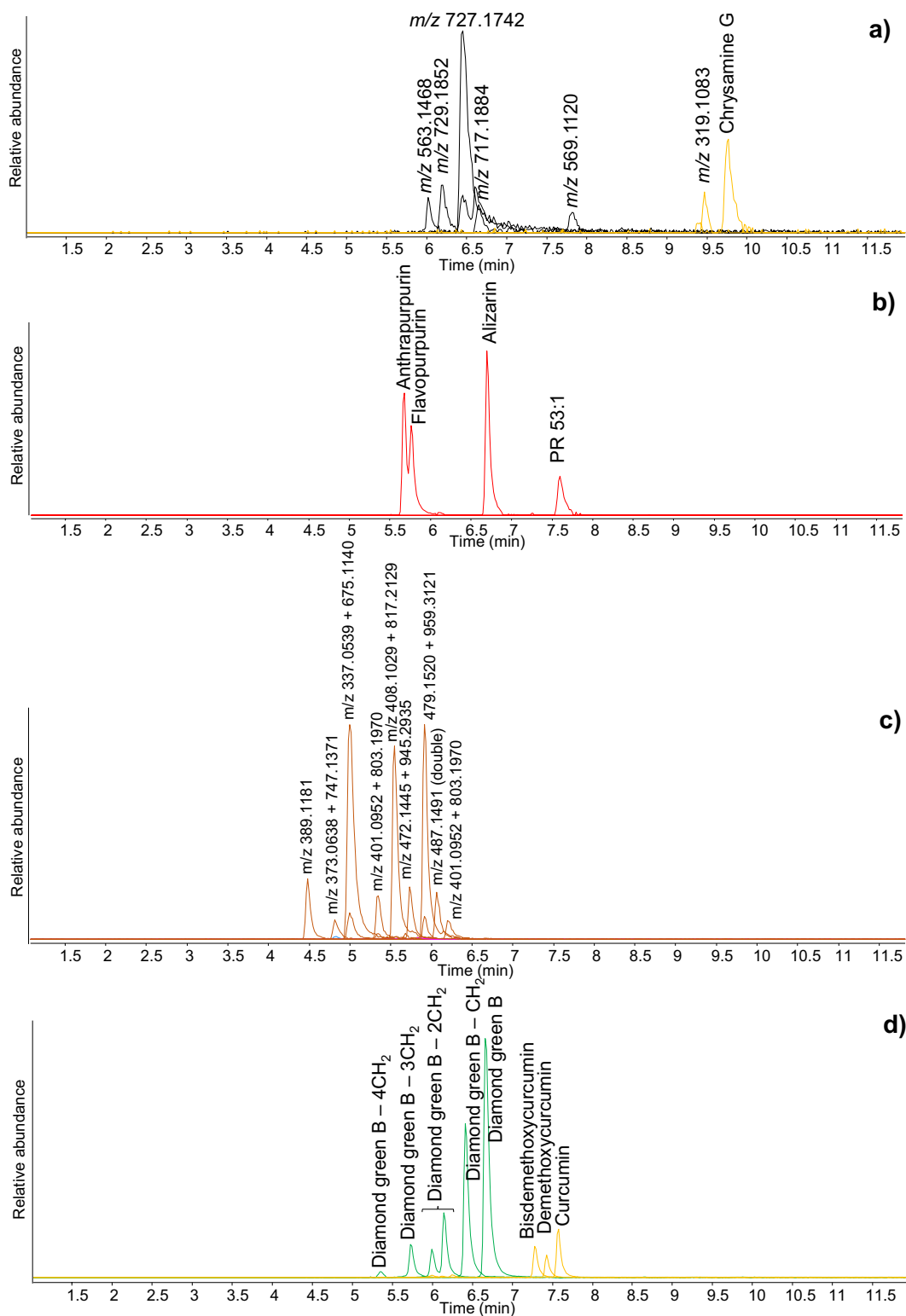


Fig. 10 Extracted ion chromatograms obtained by HPLC–DAD–MS/MS analysis of **a** T5_1 (positive ionisation), **b** T5_3 (negative ionisation), **c** T5_4 (negative ionisation) and **d** T5_7 (positive ionisation). Refer to Table 1 for sample labels

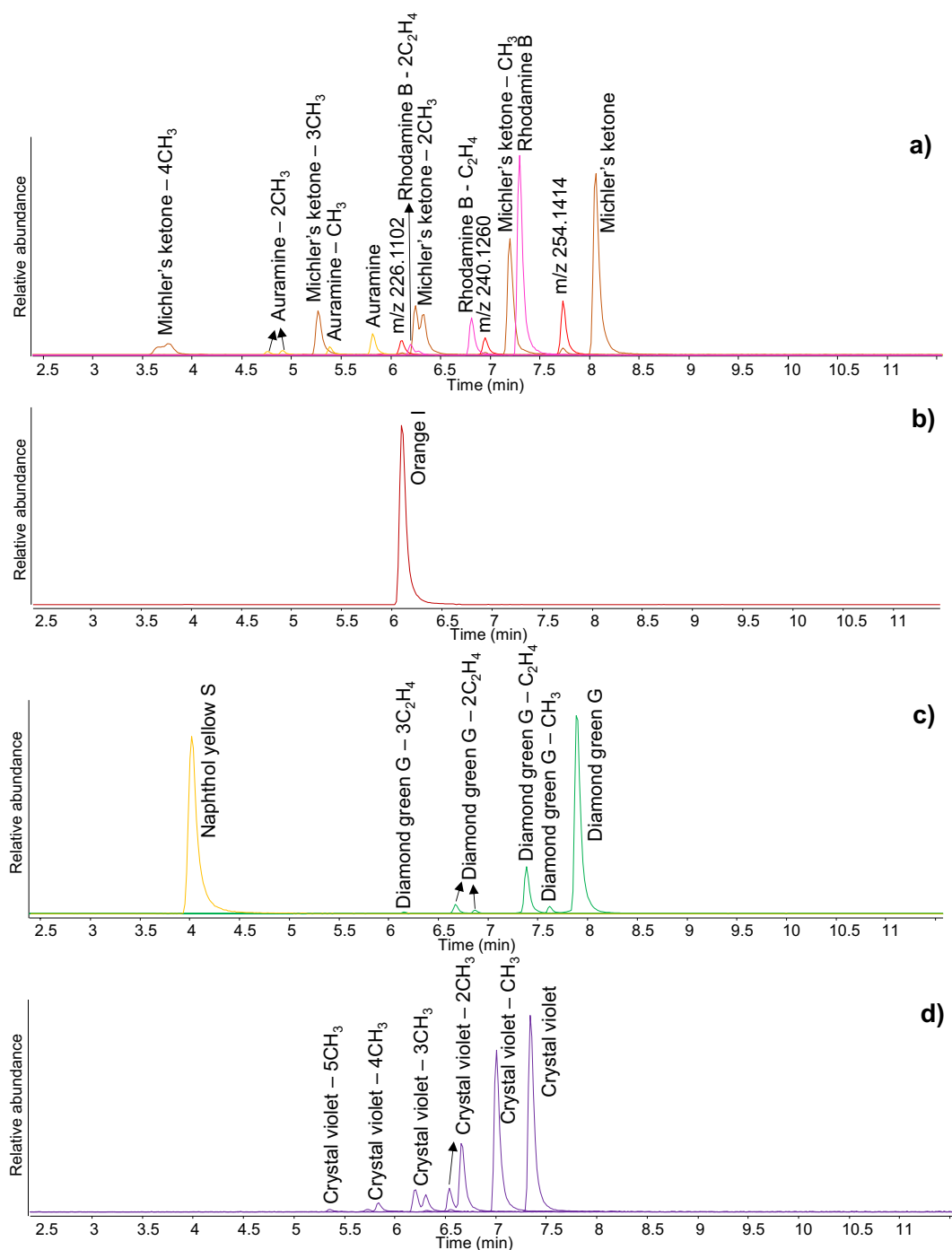


Fig. 11 Extracted ion chromatograms obtained by HPLC–DAD–MS/MS analysis of **a** T6_1 (positive ionisation), **b** T6_2 (negative ionisation), **c** T6_3 (negative ionisation for Naphthol yellow S and positive ionisation for diamond green G) and **d** T6_5 (positive ionisation). Refer to Table 1 for sample labels

Conclusions

This study provides a thorough identification of the dyes, fibres and mordants of six Karen textiles from the British Museum's collection, revealing a dynamic

textile-making scenario that evolved during the nineteenth century. The natural dyes, such as red and yellow from *Morinda citrifolia*, lac insect (*Kerria lacca*), gamboge (*Garcinia* sp.), turmeric (*Curcuma longa*), indigo

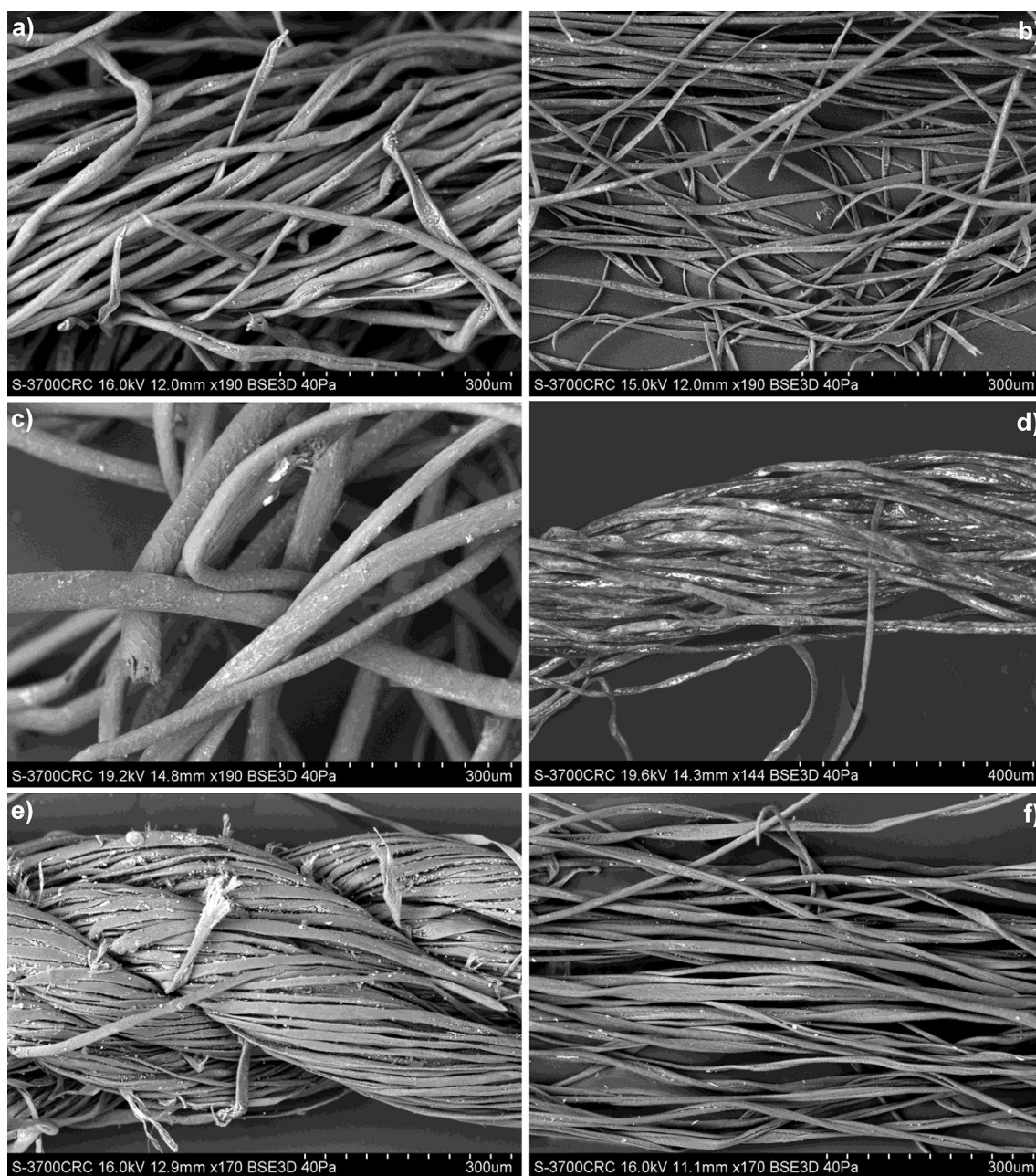


Fig. 12 VP SEM images of fibres from samples **a** T1_4, showing a well-preserved cotton thread, **b** T2_14, showing silk fibres, **c** T3_2, showing wool, **d** T3_5, showing the presence of inorganic particles on a cotton thread, **e** T5_1, showing a tightly spun cotton thread, and **f** T5_2, showing fine cotton fibres. © The Trustees of the British Museum

and tannins were found on the supposedly earlier textiles (T1 and T2), supporting their relatively early attribution dates. Scientific evidence for the use of gamboge in historic textiles is presented in this study for the first time. Although cotton was confirmed as the main fibre in these textiles, some silk threads dyed with the typical Chinese dye from the Chinese cork tree (*Phellodendron*

chinense) were also found on T2, confirming trade connections with China, which are many centuries old.

Despite the use of natural dyes in the production of T3 and T4, materials imported from Europe are evident in these textiles. Threads dyed with new modern methods, such as the use of chrome yellow and chrome orange, developed in 1820s [59], as well as tin mordanting on felt

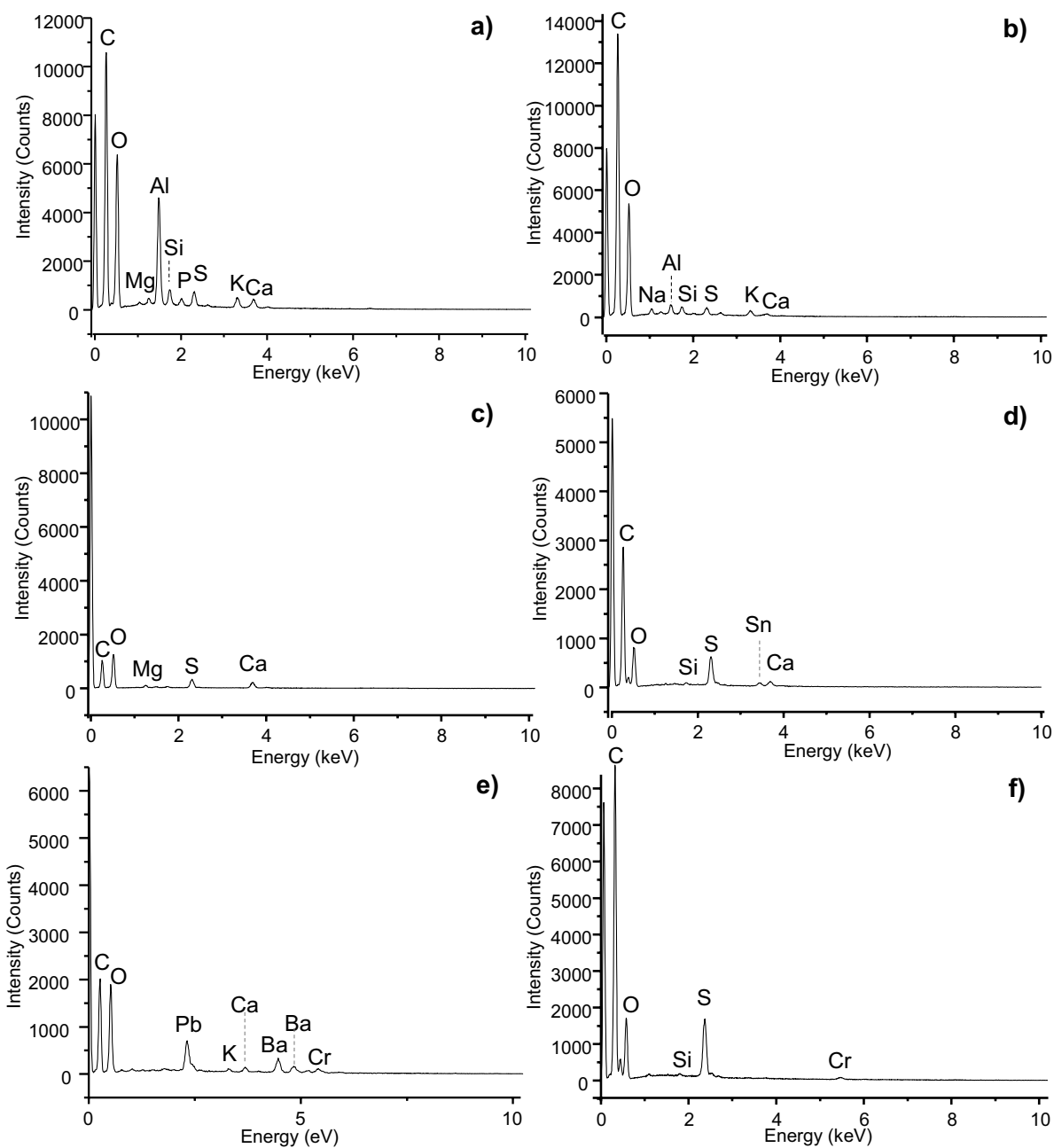


Fig. 13 EDX spectra of **a** red band of T1, **b** yellow thread of T1, **c** red left of T2, **d** red felted wool band of T3, **e** green thread of T3, **f** dark green supplementary weft of T6

and the use of typically European dyes, highlight substantial changes occurring in Karen textile production as early as the 1860s.

The detection of early synthetic dyes in T5 and T6 enables the refinement of their production dates to be attempted. In particular, Pigment red 53:1 (CI 15585:1), detected in the red areas of T5, was synthesised in 1902, confirming an early twentieth century production of this

textile, which was already suspected based on stylistic considerations. Rhodamine B (CI 45170), detected in T6, was synthesised in 1887 and is the latest dye identified in this textile, possibly pointing towards a slightly earlier production of T6 compared to T5. Overall, T5 and T6 contain multiple classes of synthetic dyes, including nitro, monoazo, disazo, anthraquinone, di- and triaryl-methane, xanthene and possibly more, thus giving an

interesting overview of the classes of dyes being produced in Europe by the end of the nineteenth century and exported to Southeast Asia. Additional examples of interesting dyeing methods also appear from the results, such as the possible use of ingrain dyes in T5, which were commercialised in 1887 and used for a relatively short period [10], and the use of chromium mordanting in T6, which was introduced in 1885 [10]. It appears reasonable to hypothesise that most of these new textile-production materials were imported from Europe as already dyed threads or yarns. However, some of the results showcase the attempts of local dyers to possibly alter the colour of these imported yarns during what can be interpreted as an experimental phase. In fact, the over-dyeing of chrome yellow with local dyes, such as *Morinda* red and lac dye, appears to have been done in Myanmar, and a mixture of diamond green B and the local dye turmeric is found in T5.

To fully understand the introduction and integration of synthetic dyes and new dyeing methods in Myanmar this research needs to be expanded and complemented by archival research into commercial trade. Nevertheless, this study represents an important starting point and constitutes a basis for further research into the penetration of European goods in Myanmar and, more in general, in Asia, in the second half of the nineteenth century, with relation to the expansion of British trade networks into these regions and increasing colonial control.

This article also highlights the utmost importance of adopting a multi-analytical protocol and a strategic approach to the analysis of textiles in museum collections. The integration of non-invasive and invasive techniques proved fundamental to limit the number of samples taken. Targeting both organic and inorganic components was also crucial to reveal the dyeing methods applied. Nevertheless, the need for additional fundamental research is evident, especially to improve the correct identification of early synthetic dyes, some of which remain unidentified due to the lack of reference samples available.

Finally, the identification of light sensitive dyes, such as turmeric, Chinese cork tree, rhodamine B and others, also informs the display of these delicate objects, which are planned to be exhibited at the end of 2023 in a major British Museum exhibition focusing on Myanmar art and history.

Abbreviations

MSI	Multispectral imaging
FORS	Fibre optic reflectance spectroscopy
OM	Optical microscopy
DM	Digital microscopy

SEM-EDX	Scanning electron microscopy energy dispersive X-ray spectrometry
HPLC-DAD-MS/MS	High-performance liquid chromatography coupled to diode array detector and tandem mass spectrometry
C.I.	Colour Index
Sp.	Species
SERS	Surface enhanced Raman spectroscopy
T1	Textile 1
T2	Textile 2
T3	Textile 3
T4	Textile 4
T5	Textile 5
T6	Textile 6
UV	Ultraviolet
VIS	Visible-reflected
UVL	Ultraviolet-induced visible luminescence
IRR	Infrared-reflected
UVR	Ultraviolet-reflected
IRRFC	Infrared-reflected false colours
UVRFC	Ultraviolet-reflected false colours
VIVL	Visible-induced visible luminescence
MBR	Multiband-reflected
TIF	Tagged image file
BM	British Museum
CID	Collision Induced Dissociation
DMSO	Dimethylsulphoxide

Supplementary Information

The online version contains supplementary material available at <https://doi.org/10.1186/s40494-023-00978-5>.

Additional file 1: Figure S1. Details of Textile 1 (accession number As1979,Q.101), including some sampling areas. © The Trustees of the British Museum. **Figure S2.** Details of Textile 2 (accession number As.7765), including some sampling areas. © The Trustees of the British Museum. **Figure S3.** Details of Textile 3 (accession number As,+6880), including some sampling areas. © The Trustees of the British Museum. **Figure S4.** Details of Textile 4 (accession number As1919,0717.200), including some sampling areas. © The Trustees of the British Museum. **Figure S5.** Details of Textile 5 (accession number As1966,01.481), including some sampling areas. © The Trustees of the British Museum. **Figure S6.** Details of Textile 6 (accession number As1901,0318.217), including some sampling areas. © The Trustees of the British Museum. **Figure S7.** a) VIS, b) UVL, c) IRR, d) UVR, e) IRRFC, f) UVRFC, g) MBR, h) VIVL images of Textile 1 (accession number As1979,Q.101). © The Trustees of the British Museum. **Figure S8.** a) VIS, b) UVL, c) IRR, d) UVR, e) IRRFC, f) UVRFC, g) MBR, h) VIVL images of Textile 2 (accession number As.7765). © The Trustees of the British Museum. **Figure S9.** a) VIS, b) UVL, c) IRR, d) UVR, e) IRRFC, f) UVRFC, g) MBR, h) VIVL images of Textile 3 (accession number As,+6880). © The Trustees of the British Museum. **Figure S10.** a) VIS, b) UVL, c) IRR, d) UVR, e) IRRFC, f) UVRFC, g) MBR, h) VIVL images of Textile 4 (accession number As1919,0717.200). © The Trustees of the British Museum. **Figure S11.** a) VIS, b) UVL, c) IRR, d) UVR, e) IRRFC, f) UVRFC, g) MBR, h) VIVL images of Textile 5 (accession number As1966,01.481). © The Trustees of the British Museum. **Figure S12.** a) VIS, b) UVL, c) IRR, d) UVR, e) IRRFC, f) UVRFC, g) MBR, h) VIVL images of Textile 6 (accession number As1901,0318.217). © The Trustees of the British Museum. **Figure S13.** Detail of UVL image of Textile 1 showing different UV-induced visible luminescence from the *Coix* seeds (top left) and digital microscopy images of the different types of seeds showing different preservation of the surface. Triangle: skittle-shape seeds; rectangle: long-elongated seeds; oval: short-elongated seeds. © The Trustees of the British Museum. **Figure S14.** Reflectance spectra obtained from the coloured areas of T1. **Figure S15.** Reflectance spectra obtained from the coloured areas of T2. **Figure S16.** Reflectance spectra obtained from the coloured areas of T3. **Figure S17.** Reflectance spectra obtained from the coloured areas of T4. **Figure S18.** Reflectance spectra obtained from the coloured areas of T5. **Figure S19.** Reflectance spectra obtained from the coloured areas of T6. **Figure S20.** Tandem mass spectra obtained in

negative ionisation mode at CID = 40 eV for precursor ions **a**) 563.1406 *m/z* and **b**) 593.1512 *m/z* tentatively identified as morindone-6,8-C-hexosyl-C-pentoside and morindone-6,8-di-C-glucoside, respectively (molecular structures shown). **Figure S21.** Extracted ion chromatograms obtained by HPLC-DAD-MS/MS analysis of T3_2 (negative ionisation). **Figure S22.** Tandem mass spectra obtained **a**) in negative ionisation mode at CID = 20 eV for precursor ion 481.1154 *m/z* and **b**) in positive ionisation mode at CID = 40 eV for precursor ion 483.1299 *m/z* identified as chrysamine G (C.I. 22250). **Figure S23.** Tandem mass spectra obtained **a**) in negative ionisation mode at CID = 40 eV for precursor ion 725.1564 *m/z* and **b**) in positive ionisation mode at CID = 60 eV for precursor ion 727.1742 *m/z* found in sample T5_1. **Figure S24.** Tandem mass spectra obtained **a**) in negative ionisation mode at CID = 20 eV for precursor ion 375.0212 *m/z* and **b**) in positive ionisation mode at CID = 20 eV for precursor ion 377.0357 *m/z* identified as PR 53:1 (C.I. 15585:1). **Figure S25.** Tandem mass spectra obtained in negative ionisation mode for precursor ions **a**) 675.1126 *m/z*, **b**) 803.1970 *m/z*, **c**) 817.2129 *m/z*, **d**) 945.2935 *m/z* and **e**) 959.3121 *m/z* found in sample T5_4. CID = 50 eV for a, b and c; CID = 60 eV for d and e. **Figure S26.** Extracted ion chromatograms obtained by HPLC-DAD-MS/MS analysis in negative ionisation mode of **a**) T6_4 and **b**) T6_7.

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Author contributions

Conceptualization: DT and AG; Data curation: DT, CRC, JD; Formal analysis: DT, CRC, JD; Investigation: DT, CRC, JD; Supervision: DT; Visualization: DT; Resources: DT, CRC, JD, AG; Writing – original draft: DT; Writing – review & editing: DT, CRC, JD, AG. All authors read and approved the final manuscript.

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Availability of data and materials

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Declarations

Competing interests

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