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# A multi-scalar investigation of the colouring materials used in textile wrappings of Egyptian votive animal mummies

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## Abstract

Commonly exhibited in museum galleries, animal mummies have been the focus of interest of both visitors and researchers alike. The study of these animal remains not only provides new insights into embalming techniques, but also brings a unique perspective on religious, social and economic practices. Twenty animal mummies are discussed in this study, including cats, ibises, crocodiles, calves and birds of prey from the collections of the British Museum (London, UK) and the Museo Egizio (Turin, Italy). The external textile wrappings encasing the mummified body of the animals were investigated with the main aim of identifying the colourants used. In fact, these are mostly patterned using undyed and coloured (mostly red, brown and black) linen strips. Broadband multispectral imaging (MSI) was initially carried out to obtain preliminary information at the macro-scale on the distribution and chemical nature of the colouring agents. Fibre optic reflectance spectroscopy (FORS) was then used to survey several coloured areas of the textile wrappings. Safflower (*Carthamus tinctorius*) and red ochre were identified non-invasively. Representative samples were then taken and observed using optical microscopy (OM) and scanning electron microscopy (SEM), in order to obtain information at the micro-scale on the distribution of the colouring agents on the fibres, as well as the presence of other materials, including those from environmental contamination. Energy dispersive X-ray spectrometry (EDX) revealed the elemental composition of particles and clear areas of the fibres, whereas high performance liquid chromatography tandem mass spectrometry (HPLC–MS/MS) provided the identification of the organic dyes at a molecular level. The use of hydrolysable and condensed tannins, in combination with iron as a mordant, was found to be used in the very dark shades, which generally corresponded to the textiles with the worst state of preservation. Nevertheless, other aspects, such as fibre processing and bleaching, fungal attack and presence of coating materials appeared to play a role in the evaluation of the conservation state of these textiles. The characterisation of the dyes and the additional inorganic materials contributed to elucidating the production technology of the colours used for animal mummification, and provided insights into ancient dyeing methods.

**Keywords:** Animal mummies, Archaeological textiles, Organic dyes, Tannins, Ancient Egypt, Multi-analytical approach

## Introduction

Thousands of mummified animals have been found during archaeological excavations. Many different species were embalmed, including cats, dogs, ibises, hawks, snakes, beetles, etc., to be then buried with or without coffins, very often in dedicated necropoleis [1]. New discoveries continue to be made, with the mummies of lion

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cubs found in the Saqqara necropolis recently, causing a sensation [2].

The Egyptian pantheon is well known for the plethora of divinities taking an animal form. The animal species are manifestation of the gods and were selected to align with some of their most distinctive traits. Some animals were believed to incarnate the gods on earth. Chosen on the basis of physical characteristics, they lived within the sacred space of the temple where they would be fed and cared for until their natural death. After their death, their body was treated like that of a god. It was washed, embalmed and buried in a sarcophagus during sumptuous ceremonies [3]. These living gods are just one type of animal mummy and they are very difficult to differentiate from mummies offered to the gods by the worshippers, sometimes known as 'votive mummies'. They enclose the remains of animals that were raised, killed and embalmed to be sold as an *ex voto* to worshippers visiting the temple, who entrusted them with their supplications to the god [4]. This category, which yielded many thousands of mummies, will be the focus of the present study. In addition, pet animals, such as cats, dogs or gazelles, were sometimes mummified, and some have been found buried with their owner. The last category consists of vidual mummies: ducks, geese and pieces of meat were embalmed and placed among the grave goods to nourish the deceased for eternity [5].

Votive animal mummies are a significant portion of Egyptian collections in many archaeological and ethnographic museums. The Museo Egizio collection in Turin encompasses more than 200 animal mummies, including wrapped mummies, animals without bandages and mummies inside their containers/coffins. The oldest nucleus of the collection (some 20 items) arrived in Turin in the eighteenth century, followed by about 80 mummies from the Drovetti collection, purchased in 1823. Regrettably, the provenance of finds was still not considered of great importance in the seventeenth and eighteenth centuries and there is no record of their provenance. In the twentieth century, the collection of the Museo Egizio was expanded, mainly thanks to the excavation conducted in Egypt by the Italian Archaeological Mission (M.A.I.), directed by Ernesto Schiaparelli (museum director from 1894) and Giulio Farina (museum director from 1928, after Schiaparelli's death). This group of specimens includes about 90 animal mummies excavated in Asyut in 1910. Additional information on the archaeological context is unknown [6].

A large proportion of the mummies in the British Museum's collection also lack archaeological context. About half of the approximately 400 mummies that comprise the collection is unprovenanced. A quarter of the British Museum's collection was acquired in the early

part of the nineteenth century, by collectors and travellers, such as Henry Salt, Joseph Sams, John Gardner Wilkinson, James Burton, Giovanni Anastasi or Robert Hay [7]. The lack of archaeological record is also characteristic of the group of 20 mummies acquired in the 1870s from Selima Harris, the daughter of British collector A. C. Harris, and the additional 30, registered in 1915, as coming from Captain E. L. Gruning. Of these, none were recovered during excavations and their original context of discovery is unknown. About 30 mummies were, however, found during D.G. Hogarth excavations of Asyut and during Egypt Exploration Society digs, notably in Abydos or Saqqara.

When looked at from a scientific perspective, animal mummies are complex entities, characterised by heterogeneous materials. In addition to the organic remains, such as skeletal parts and sometimes soft tissues, embalming materials are generally present. Reeds, wooden sticks, mud and sand were sometimes used as filler materials. Finally, mummies were often wrapped in dyed and/or undyed linen bandages made from flax fibres (wool is very rarely documented) [8]. The materiality of animal mummies has attracted the interest of scientists for many years and several studies have been conducted with the aim to shed light on this fascinating practice. Radiocarbon dating has been used to try to establish a timeline for the introduction of different animal species into the mummification practice [9–11]. Computer tomography (CT) and X-ray imaging techniques are probably the most popular approach to the investigation of animal mummies, as they are non-invasive and yield extremely valuable information, including species identification, the state of preservation and, in some cases, the cause of death of the animal [12–19]. Most recently, synchrotron-based imaging techniques have been used to investigate some animal mummies [20]. Attention has also been dedicated to the embalming materials. Fourier transform infrared spectroscopy (FTIR), gas chromatography–mass spectrometry (GC–MS) and pyrolysis–gas chromatography–mass spectrometry (Py–GC–MS) have shown that the embalming mixtures include animal fats, oils, beeswax, sugar gum, petroleum bitumen, and coniferous and *Pistacia* spp. resins [21–23], and are thus of comparable complexity to those used to mummify humans [24, 25]. Isotopic analysis and DNA analysis have also recently been applied to clarify the exact species, as well as the origin (wild or domesticated) and provenance, of the animals that underwent mummification [26–29]. Multi-analytical approaches can additionally provide a tool to provide a thorough condition assessment of the animal mummies [30].

Despite the significant amount of studies on animal mummies, the textile wrappings, and in particular their

colours, have mostly been disregarded in the scientific literature. Many specimens show complex wrapping systems, with some inner layers of coarse, and probably re-used, bandages covered by an intricate system of strips arranged to create decorative patterns. The use of bands of different colours (black, brown, orange, pinkish/red) could create many variations of the same pattern. Additionally, dyed textiles and painted motifs were used to reconstruct some of the anatomical parts of the animal (eyes, ears, beak, etc.), as visible in some of the mummies under investigation (Table 1). This study is therefore intended to fill this gap in the scientific literature, by focusing on the materials used to colour the textile wrappings of animal mummies. This research builds on a previous investigation on the textile wrappings from human mummies, during which the potential of a multi-analytical approach was demonstrated, by combining non-invasive and micro-invasive techniques, such as fibre optic reflectance spectroscopy (FORS), optical microscopy with visible or UV light (Vis-OM, UV-OM), high performance liquid chromatography–tandem mass spectrometry (HPLC–MS/MS), scanning electron microscopy coupled to energy dispersive X-ray spectrometry (SEM–EDX) and micro-X ray fluorescence spectrometry (micro-XRF), to investigate both the colouring materials and the dyeing techniques [31]. Broadband multispectral imaging (MSI) has now also been included to the proposed investigative approach, in order to provide further information at the macro-scale and accentuate the importance of gathering data at multiple scales to obtain a holistic picture of the materials present.

To achieve this goal, the animal mummies selected from the Museo Egizio and the British Museum represent different animal species (cats, ibises, crocodiles, calves and birds of prey) and different decorative patterns obtained using coloured textiles, mainly brown, black and pink/red. The conservation condition also differed across the mummies selected, with brown-black fabrics being, in most cases, less well preserved. Consequently, a parallel aim of the study was to relate, at least partially, the use of certain colouring materials to the degradation state of the textiles, in order to provide conservators and curators with additional information to plan appropriate conservation and display strategies.

## Materials and methods

### Mummies and samples

Animal mummies in the Museo Egizio have undergone conservation and scientific investigation within the framework of a multi-disciplinary project aimed at preparing the forthcoming catalogue of the animal mummy collection. The project produced calibrated radiocarbon dates for 53 samples detached from the materials

employed for the bundles. Samples for radiocarbon dating were mainly obtained from undyed textiles or plant elements and only in a few cases, for which the mummified remains were exposed, from the animal itself. The results of the calibrated radiocarbon dating highlighted a distribution of the sampled materials from the seventh century BC to the third century AD, i.e. within the late Pharaonic and Roman periods.

While the Museo Egizio in Turin had made great progress in the study and conservation of their collection of animal mummies, the British Museum's collection has so far only been the focus of very targeted studies of the mummies and bronze votive boxes [32–34]. The museum is, however, currently embarking on a more comprehensive programme of investigation. Although often attributed to the Roman period, the dating of most of the British Museum's animal mummies still remains to be ascertained and will be the focus of future research.

Several criteria were adopted to select the mummies among the two museums' large collections. After the direct and microscopic observation of dyed and undyed bandages of a larger number of mummies, twenty mummies were selected based on the different colours and decoration patterns exhibited, as well as available information on dating, provenance, animal species, degradation level of the fibres, and apparent stability of the colour (Table 1).

Nine mummies were selected from the British Museum's collection and eleven from the Museo Egizio's collection. The animals comprised; 5 cats, 6 ibises, 5 crocodiles, 2 calves and 2 birds of prey, probably falcons, thus representing a varied array of mummified animal species. Photographs of the animal mummies are provided in Table 1, together with a brief description and available details. A simplified labelling system with the first letter(s) of the animal typology and a sequential number was also used to avoid using complex accession numbers in the discussion. Samples (*ca.* 1 cm of a thread) were taken from the textile wrappings of all mummies in correspondence to areas representative of the colours exhibited (Table 1).

The cats are all of unknown provenance. Nevertheless, they have very intricate polychrome patterns with orange/pinkish strips dyed with fugitive colours. Only one (C4) was radiocarbon dated. The ibises are mostly of known provenance and dating. The Museo Egizio's ibises and birds of prey are from Asyut, and the British Museum's ibises are from Abydos. The classification of the decorative pattern of most ibises was not possible for some dark and brown bandages combinations, where the material was so degraded that it was almost completely lost (see for example I3 and I5). The British Museum's bird of prey (F1) is of unknown provenance. Both bull-shaped







**Table 1** Brief description and details of the animal mummies under investigation and samples taken

Image	Accession number	Label	Description	Provenance	Date <sup>a</sup>	Samples
	British Museum EA 6756	C1	Cat Length: 49.5 cm	Unknown	Unknown	Pink Brown
	British Museum EA 65502	C2	Cat Length: 39 cm	Unknown (bequeathed by Henry Swainson Cowper, 1951)	Unknown	Red
	British Museum EA 55614	C3	Cat Length: 45.5 cm	Unknown (Donated by Capt W. A. Norman, 1928)	Unknown	Red Brown
	Museo Egizio Cat. 2348/1	C4	Cat Length: 54 cm	Unknown (Drovetti Collection—1824)	400–200 BC (textile)	Pink Brown



**Table 1** (continued)

Image	Accession number	Label	Description	Provenance	Date <sup>a</sup>	Samples
	Museo Egizio Cat. 2349/3	C5	Cat Length: 39 cm	Unknown (Drovetti Collection—1824)	Unknown	Pink Brown Dark brown
	British Museum EA 52926	I1	Ibis Length: 35.5 cm	Abydos (donated by the Egypt Exploration Society, 1913)	Unknown	Reddish Brown
	British Museum EA 53938	I2	Ibis Length: 43.2 cm	Abydos (donated by the Egypt Exploration Society, 1914)	Unknown	Black
	Museo Egizio S. 11013	I3	Ibis Length: 35 cm	Asyut	390–190 BC (textile)	Dark brown
	Museo Egizio S. 11018	I4	Ibis Length: 30 cm	Asyut	40 BC–210 AD (textile)	Pink Dark brown
	Museo Egizio S. 11033	I5	Ibis Length: 36.5 cm	Asyut	Unknown	Dark brown

**Table 1** (continued)

Image	Accession number	Label	Description	Provenance	Date <sup>a</sup>	Samples
	Museo Egizio S. 11037	l6	Ibis Length: 33 cm	Asyut	370–90 BC (textile)	Dark brown
	British Museum EA 35727	Cr1	Crocodile Length: 39.5 cm	Unknown	Unknown	Light yellow Light brown Dark brown
	British Museum EA 35728	Cr2	Crocodile Length: 27 cm	El-Mabda (donated by W. Boyne, 1846)	Unknown	Red
	Museo Egizio Cat. 2351/2	Cr3	Crocodile Length: 181.5 cm	Unknown (Drovetti Collection—1824)	40 BC–210 AD (plant element)	Black
	Museo Egizio Cat. 2353/11	Cr4	Crocodile Length: 34 cm	Unknown (Drovetti Collection—1824)	370–200 BC (textile)	Light Brown Dark brown
	Museo Egizio Prov. 1443	Cr5	Crocodile Length: 53 cm	Unknown (Drovetti Collection—1824)	370–200 BC (plant element)	Light Brown Black
	British Museum EA 6773	Ca1	Calf Height: 45.7 cm Length: 73.7 cm	Said to be from Thebes (Salt collection, 1821)	Unknown	Light brown Dark brown

**Table 1** (continued)

Image	Accession number	Label	Description	Provenance	Date <sup>a</sup>	Samples
	Museo Egizio Cat. 2344	Ca2	Calf Length: 85 cm	Unknown (Drovetti Collection—1824)	400–190 BC (plant element) 410–360 BC (textile)	Light orange Brown
	British Museum EA 90471	F1	Bird of prey Length: 43 cm	Unknown	Unknown	Yellow Red Brown
	Museo Egizio S. 11040	F2	Bird of prey Length: 24.8 cm	Asyut	210–30 BC	Orange Dark brown

<sup>a</sup> Part of the mummy used for radiocarbon dating in parenthesis

mummies (calves) were acquired in the nineteenth century, one by Henry Salt and one by Bernardino Drovetti, both then consuls of Egypt, respectively for England and France. The naturalistic head is covered with a brown fabric, while the chest is decorated with an intricate polychrome pattern. Future research might allow us to confirm whether they were produced within a close time period, perhaps even in the same workshop, as suggested by their stylistic similarities and the proximity in date of their acquisition. Most crocodiles are of unknown provenance. The Museo Egizio's crocodile specimens date back from 370 BC to 210 AD. Their decorative motifs are difficult to interpret because of the brown-black heavily degraded stripes.

#### Analytical protocol

A combination of non-invasive and micro-invasive techniques was used to investigate the textiles. The analytical approach included broadband multispectral imaging (MSI) of the mummies, in order to obtain preliminary information about the colouring materials and their distribution. The experimental setup, as well as the acquisition and post-processing protocols adopted, are reported in [35]. FORS was subsequently used on selected areas

to, where possible, obtain a non-invasive identification of the colouring materials, with particular attention to red, orange and pink shades. Samples (*ca.* 1 cm of a thread) were then taken. The samples were imaged by Vis-OM and UV-OM, in order to investigate the distribution of the colouring matters on the fibres at a microscopic scale. Then, *ca.* 1–2 mm of the samples were used for high performance liquid chromatography coupled to electrospray ionisation and quadrupole time-of-flight (HPLC-ESI-Q-ToF), in order to confirm or provide identification of organic dyes as sources of colouring materials at a molecular level. The rest of each sample was used for scanning electron microscopy (SEM) investigations, to determine the extent of fibre degradation, the distribution of the colouring materials, possible contamination materials on the fibres and possibly signs of fibre processing. Energy dispersive X-ray spectrometry (EDX) was used to assess the elemental composition of particles and other areas of interest on the fibres, in order to complement the information on organic components. The methodology and instrumentations for FORS, OM, SEM-EDX and HPLC-ESI-Q-ToF are described in [31].

It was not possible to apply all the techniques described to all the mummies and samples. The techniques applied

to each mummy are summarised in Table 2, which also includes the main results obtained.

## Results

Following an initial visual examination of the mummies, several coloured areas of the wrappings were noticed. However, it was not always straightforward to attribute an exact colouration, nor to distinguish deliberately coloured areas from regions of yellowing or darkening of the linen, as to the result of ageing, possible contact with body fluids, and/or additional materials used during mummification, such as resins and lipids. For this reason, terms such as yellow, orange and light brown are mostly used as indicative colours in Table 2.

The presence and source of colouring materials on the wrappings were confirmed using the multi-scalar/multi-analytical investigative approach, and a discussion of the results obtained follows.

### Broadband MSI

As noted above, when investigating mummy wrappings, it can be challenging to discern areas of intentionally dyed textile from regions of discolouration due to natural degradation of the linen or residues from the embalming rituals. Although the use of UV lamps, and to some extent infrared photography, to aid in the inspection of mummy wrappings has a long history in conservation practice, to date no formal publication is available.

Broadband MSI techniques, a series of photographic procedures used to observe an object using wavelength ranges that include and extend beyond the capabilities of the human eye [36], can be useful in enhancing the response of certain materials, such as dyes, creating contrast and informing on the spatial location and chemical characteristics of these materials. The use of these techniques on painted surfaces and textiles is now well-established [35–39]. However, their application to mummy wrappings presents a challenge due to the specific nature of these wrappings that (i) are usually made of linen, which has its own luminescence properties in the visible region [35]; (ii) may have been coloured with light sensitive dyes, which are likely highly faded as a result of display or ageing [31]; (iii) may exhibit heavy soiling from either their burial or deposition context, or from bodily and/or embalming fluids [21]. In spite of these challenges, some key observations can be made, as exemplified by the animal mummies considered in this study.

Inorganic sources of red colourants, such as red ochre (natural earths rich in hematite) and organic dyes, such as madder or safflower, have a long tradition of use in ancient Egypt, and have been found in mummy shrouds and textile fragments from Pharaonic Egypt [31]. Both sources of red colourants were also employed as part

of the funerary practice in animal mummies. Although in some cases it may be possible to discern whether red ochre rather than organic dye was used to colour the linen, the task becomes difficult when only scant traces of red colouration are observed. This was the case with the coloured traces on the wrappings on the crocodile mummy Cr2, which were observed to absorb deeply (appear dark) under UV irradiation (images not shown). Similar behaviour was observed on the face of the cat mummy C2, when examining the wrappings with MSI. The UVL image (Fig. 1b), shows how the painted pink areas, i.e. the inserted ears and decorative “dots” on the face, appear dark. By contrast, organic reds will display at least some weak luminescence under UV irradiation, as noted from the coloured stripes of the mummy wrappings (Fig. 1b). These observations suggest the use of a red colourant that was mineral in origin in both these cases.

Broadband MSI methods were also useful in differentiating between various sources of organic reds. As noted above, organic sources of red colourants will display at least some weak luminescence under UV irradiation and the intensity and wavelength range of this luminescence can be indicative of the dye source. For madder, various factors can impact the results, with deeper reds absorbing UV and appearing darker or very weakly emitting in the UVL images, whereas lighter-coloured reds or dark pinks are observed to emit pink luminescence [35]. Weakly emitting areas, such as those observed in the stripy wrappings of C2 (Fig. 1b), and also in alternating rows of linen strips in the diamond-patterned wrappings of cat mummy C3 (Fig. 2b), may thus indicate the use of madder. Typically, further evidence of this would be sought from the IRRFC and UVRFC images, where wool and silk fibres coloured with madder appear yellow and dark green/brown, respectively [35, 37]. The areas of the wrappings under consideration in C2 and C3 appear yellow in IRRFC and brown in UVRFC (Figs. 1c, d and 2c, d), which is in keeping with the possible use of madder in these areas.

Textiles dyed red or pink with safflower have a very different response under UV irradiation from that described for both madder and red ochre. Distinct orange luminescence has been observed from safflower-dyed yarns [37, 40], however, the intensity of the emission will be impacted by how much light exposure the textile has previously received. In protected areas, such as those covered by the brown criss-cross strips on the wrappings of cat mummy C1 (Fig. 3), strong orange luminescence is still observed in the UVL image (Fig. 3b). This contrasts with the exposed areas, which were probably also, once, brightly coloured with safflower dye, but are now too faded to exhibit perceptible luminescence.



**Table 2** Summary of the results obtained from all the techniques applied to each mummy textile wrapping/sample

Accession number	Label	Areas of interest/ samples <sup>a</sup>	MSI	FORS	Vis-OM	UV-OM	SEM	EDX	HPLC-ESI-Q-ToF
EA 6756	C1	Pink from protected area, (covered by the brown criss-cross strips)	UVL—orange luminescence IRRF—yellow (faint) UVRFC—brown (faint) Indicative of safflower	Safflower	Pink/orange non-homogeneous colouration	Intense orange luminescence of a few areas	Almost intact fibres, reduced processing, presence of particles	Ca and Cl as most abundant elements	Safflower
		Brown from criss-cross strips	UVL—strongly absorbing IRRF—dull orange UVRFC—dark greenish brown Indicative of tannins	—	Non-homogeneous brown colouration	Prevalent absorbance of the UV radiation	Brittle fibres, transversal cracks, advanced processing, presence of particles	Si, Al, and Fe present in particles	Hydrolysable tannins
EA 65502	C2	Red from stripy wrappings	UVL—weak pinkish luminescence IRRF—yellow UVRFC—brown May suggest madder	Not conclusive	Relatively homogeneous light red colouration and presence of agglomerates	Weak pinkish luminescence from the fibres (especially “knee-joints”) and reddish luminescence from agglomerates	Well-preserved fibres, intermediate processing, scarce particles	General low concentration of elements	No dye detected
		Painted pink areas of the face and ears	UVL—Strongly absorbing Indicative of red ochre	Red ochre	—	—	—	—	—
EA 55614	C3	Red from diamond pattern	UVL—weak pinkish luminescence IRRF—yellow UVRFC—brown May suggest madder	Not conclusive	Relatively homogeneous light red colouration	Weak pinkish luminescence from the fibres (especially “knee-joints”)	Well-preserved fibres, intermediate processing	Si, Al, and Fe present in particles	No dye detected
		Brown from diamond pattern	UVL—strongly absorbing IRRF—deep orange/red UVRFC—dark brown Indicative of tannins	—	Relatively homogeneous brown colouration	Prevalent absorbance of the UV radiation	Advanced brittleness, abundant transversal cracks	Si, Al, and Fe present in particles, Ca generally present	Condensed tannins (?)

**Table 2** (continued)

Accession number	Label	Areas of interest/ samples <sup>a</sup>	MSI	FORS	Vis-OM	UV-OM	SEM	EDX	HPLC-ESI-Q-ToF
Cat. 2348/1	C4	Pink from banded net pattern	–	–	Homogeneous yellowish colouration	Non-homogeneous yellowish luminescence of a few areas	Well-preserved fibres; a few tangential cracks	Si, Al, and Fe present in a few particles	No results
		Brown from the neck area	–	–	Relatively homogeneous brown colouration	General absorbance of the UV radiation and presence of UV-absorbing particles/areas	Abundant presence of particles with different shapes	Na and Cl abundant in crystals; Si, Al and Fe abundant in other particles	Hydrolysable tannins
Cat. 2349/3	C5	Pink from internal meander pattern	–	Safflower	Pink/orange non-homogeneous colouration and presence of coloured particles/agglomerates	Intense orange luminescence of a few areas and of the particles/agglomerates	–	–	–
		Brown from meander pattern	–	Possible tannins	Relatively homogeneous brown colouration	Prevalent absorbance of the UV radiation	–	–	–
		Dark brown from meander pattern	–	Possible tannins	Relatively homogeneous dark brown colouration	Prevalent absorbance of the UV radiation	–	–	–
EA 52926	I1	Reddish from herringbone pattern	–	Featureless	Relatively homogeneous light red colouration	Non-homogeneous yellowish luminescence of a few areas	Well-preserved fibres; intermediate processing	Si, Al, and Fe present in particles	No results
		Brown from herringbone pattern	–	–	Relatively homogeneous dark brown colouration	Prevalent absorbance of the UV radiation	Advanced brittleness; few particles	Si, Al, and Fe present in particles	Hydrolysable tannins
EA 53938	I2	Black from coffer diagonal pattern	–	–	Relatively homogeneous very dark brown colouration	Prevalent absorbance of the UV radiation	Advanced brittleness; abundant transversal cracks	Si, Al, and Fe present in particles; Fe abundantly present in clear areas	Hydrolysable and condensed tannins
S. 11013	I3	Dark brown from herringbone pattern	–	Possible tannins	Relatively homogeneous dark brown colouration	Prevalent absorbance of the UV radiation and yellowish luminescence of a few areas	Advanced degradation; transversal and tangential cracks; abundant elongated crystals	Na, Ca, S abundant in crystals (but no Cl); Si, Al and Fe present in other particles	Hydrolysable tannins and degradation products



**Table 2** (continued)

Accession number	Label	Areas of interest/ samples <sup>a</sup>	MSI	FORS	Vis-OM	UV-OM	SEM	EDX	HPLC-ESI-Q-ToF
Cat. 2351/2	Cr3	Black from protected area (covered by other strips)	–	Possible tannins	Homogeneous black colouration and presence of particles	Prevalent absorbance of the UV radiation accompanied by some luminescence of non-fibrous plant material	Fibres covered by non-fibrous plant materials and abundant particles	All common elements present; Na, Fe and Si abundant; Fe present in clear areas	Hydrolysable and condensed tannins
Cat. 2353/11	Cr4	Light brown	–	Featureless	Relatively homogeneous light brown colouration	Weak orange/pinkish luminescence from the fibres (especially “knee-joints”)	Brittleness, presence of cracks, abundant particles, reduced processing	Particles rich in Fe, Al and Si; clear areas with few elements	No results
Prov. 1443	Cr5	Dark brown	–	Possible tannins	Relatively homogeneous dark brown colouration and presence of particles	Prevalent absorbance of the UV radiation accompanied by some luminescence of non-fibrous plant material	Advanced brittleness, presence of cracks, abundant particles, very reduced processing	Particles rich in Fe, Al and Si; clear areas also rich in Fe	Hydrolysable tannins and degradation products
EA 6773	Ca1	Light brown from face	–	–	Homogeneous yellowish colouration	Very weak yellow/orange luminescence from the fibres (especially “knee-joints”)	Relatively good preservation, few cracks and particles	General elements present with low concentration	No results
		Black	–	–	Relatively homogeneous black colouration and presence of particles	Prevalent absorbance of the UV radiation	Advanced brittleness, transversal and tangential cracks, reduced processing, abundant particles	General elements present in both particles and clear areas	Hydrolysable and condensed tannins
		Dark brown from face	–	–	Relatively homogeneous light brown colouration	General absorbance of the UV radiation and presence of UV-absorbing particles/areas (including “knee-joints”)	Brittleness, transversal cracks, reduced processing, some particles	Particles rich in Si and Al; general elements present in clear areas, but no Fe	Hydrolysable tannins
		Dark brown from face	–	–	Relatively homogeneous dark brown colouration	Prevalent absorbance of the UV radiation	Advanced brittleness, numerous cracks, some particles	Ca, Si, Al and Fe abundantly present in particles; Fe also present in clear areas	Hydrolysable tannins

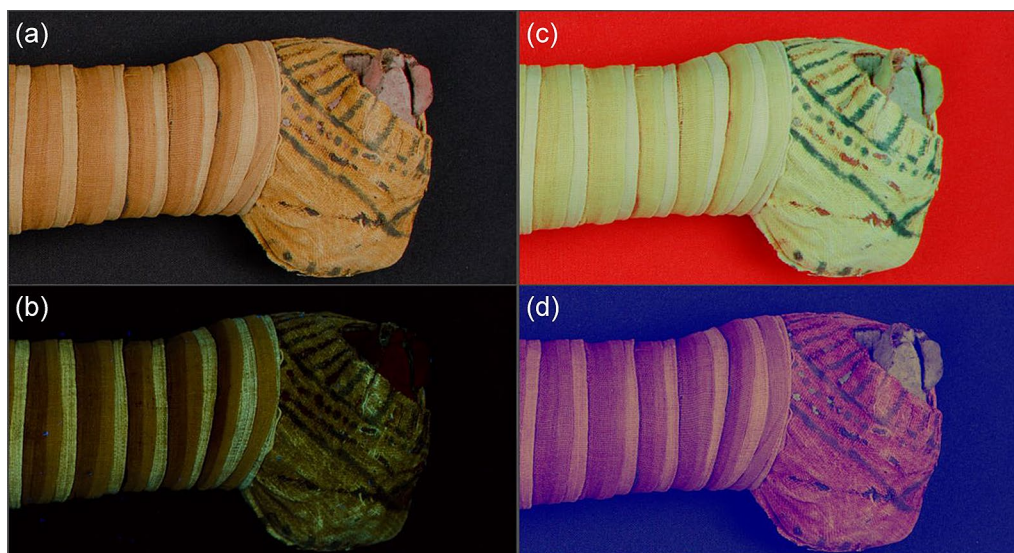
**Table 2** (continued)

Accession number	Label	Areas of interest/ samples <sup>a</sup>	MSI	FORS	Vis-OM	UV-OM	SEM	EDX	HPLC-ESI-Q-ToF
Cat. 2344	Ca2	Light orange from polychrome double sided coffer pattern	–	Featureless	Relatively homogeneous light orange colouration	Weak yellow/orange luminescence from the fibres (especially “knee-joints”)	Advanced brittleness, transversal cracks, some particles	Some particles rich in S, K and Ca; other particles rich in Si and Al	No results
		Brown from polychrome double sided coffer pattern		Possible tannins	Relatively homogeneous brown colouration	Prevalent absorbance of the UV radiation	Advanced brittleness, transversal cracks, reduced processing, abundant particles	General elements present in particles; Fe also present in clear areas	Hydrolysable tannins
EA 90471	F1	Yellow from external pattern	UVL—faint yellow luminescence May indicate yellow dye source	–	Relatively homogeneous yellowish colouration	Weak yellow/orange luminescence from the fibres (especially “knee-joints”)	Relatively good preservation, reduced processing, few particles	Some particles rich in S, K and Ca	No results
		Red from internal square pattern	UVL—orange luminescence IRRF—yellow (faint) UVRF—brown (faint) Indicative of safflower	Possible safflower	Relatively homogeneous reddish colouration	Orange luminescence (especially from “knee-joints”)	Relatively good preservation, reduced processing, fungal spores and hyphae	Some particles rich in S, K and Ca; other particles rich in Si and Al	Safflower
		Brown from internal square pattern	UVL—strongly absorbing IRRF—deep orange/red UVRF—dark brown Indicative of tannins	–	Non-homogeneous brown colouration and presence of light brown non-fibrous material	Absorbance of the UV radiation on fibres and dark orange luminescence from non-fibrous material	Advanced degradation, abundant fungal hyphae and spores, very reduced processing	Some particles rich in S, K and Ca; general elements present at low concentration	Hydrolysable tannins

**Table 2** (continued)

Accession number	Label	Areas of interest/ samples <sup>a</sup>	MSI	FORS	Vis-OM	UV-OM	SEM	EDX	HPLC-ESI-Q-ToF
S. 11040	F2	Orange from inter- nal strips	–	Featureless	Relatively homoge- neous light orange colouration	Very weak yellow/ orange lumines- cence from the fibres (especially “knee-joints”)	Relatively good preservation, some transversal cracks, few particles	General elements present in particles, but not in clear areas	No results
		Dark brown from protected area		Possible tannins	Relatively homoge- neous dark brown colouration	Prevalent absorp- tion of the UV radiation and presence of UV- absorbing particles/ areas (including “knee-joints”)	Transversal cracks, reduced process- ing, presence of particles	Some particles rich in S, K and Ca; other particles rich in Si and Al; Fe generally low	Hydrolysable tannins and degradation products

<sup>a</sup> Colours as observed on the mummy wrappings. These were often difficult to define and are likely to differ from their original shades



**Fig. 1** **a** Visible-reflected (VIS); **b** UV-induced visible luminescence (UVL); **c** infrared-reflected false colour (IRRFC); **d** UV-reflected false colour (UVRFC) images of the proper right top section of C2

Although weaker than in C1, similar orange luminescence, indicative of safflower, is also observed in the wrappings of the falcon mummy F1, particularly in the three strips that traverse the body horizontally (Fig. 4b). In both these cases, the high degree of fading makes the response in the IRRFC and UVRFC images (usually yellow and brown, respectively [37]) almost indiscernible from that of the surrounding areas.

A class of colourants that shows a distinct response across images when investigated with broadband MSI, are tannin-based dyes. Several of the animal mummies investigated show the use of linen strips dyed to varying degrees of dark brown in their wrappings. These areas are very highly absorbing under UV illumination, appearing very dark in the UVL images. They also exhibit characteristic reflectance behaviour in the IRRFC images, appearing dull orange to red, depending on the depth of applied colour. This combined absorption/reflectance behaviour is characteristic of the presence of tannins [35] and is most clearly observed in the brown criss-cross strips on the wrappings of cat mummy C1 (Fig. 3b, c). Other instances where the response in the IRRFC images is a deeper red/orange, include areas of the diamond-patterned wrappings of C3 (Fig. 2c) and F1 (Fig. 4c).

Finally, broadband MSI techniques are useful in discerning areas of the wrappings that are soiled or only discoloured through ageing.

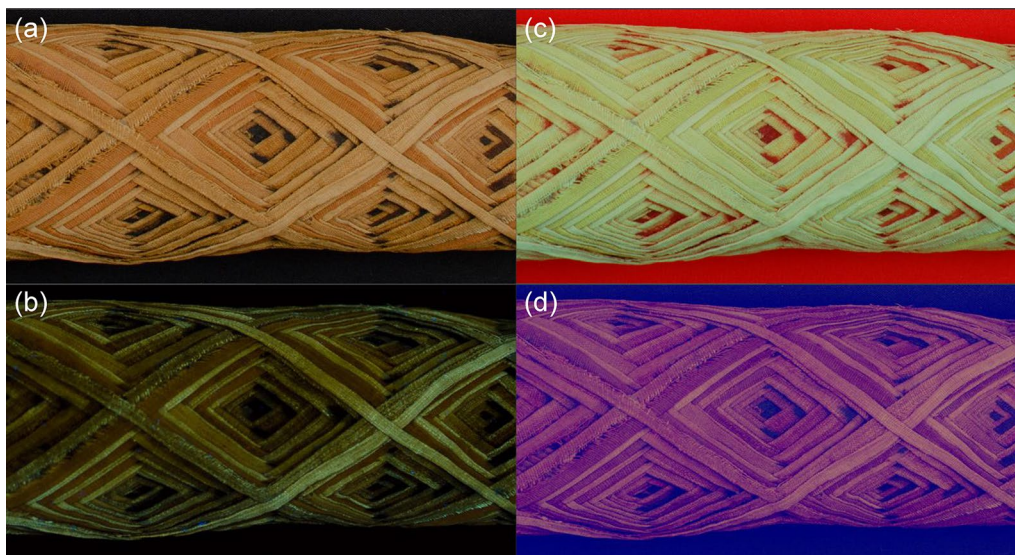
It has previously been noted that, under UV illumination, lignocellulosic materials, such as cotton, linen or hemp, exhibit beige or slightly pink luminescence even when aged [35]. In contrast, materials with proteinaceous

origins, such as wool and silk, usually appear bluish under UV [35]. In keeping with these observations, areas of the wrappings of C2 and C3 appear beige in the UVL images (Figs. 1b and 2b), which may indicate that they are undyed or that any remaining dye has faded. However, the wrappings of C1 appear slightly bluish in the UVL image (Fig. 3b), and may suggest the presence of a proteinaceous or other blue-luminescing material. Although no reference is made to this in the conservation record for the object, the use of proteins in consolidation treatments was common practice in the past, particularly where brittle wrappings were in danger of unravelling.

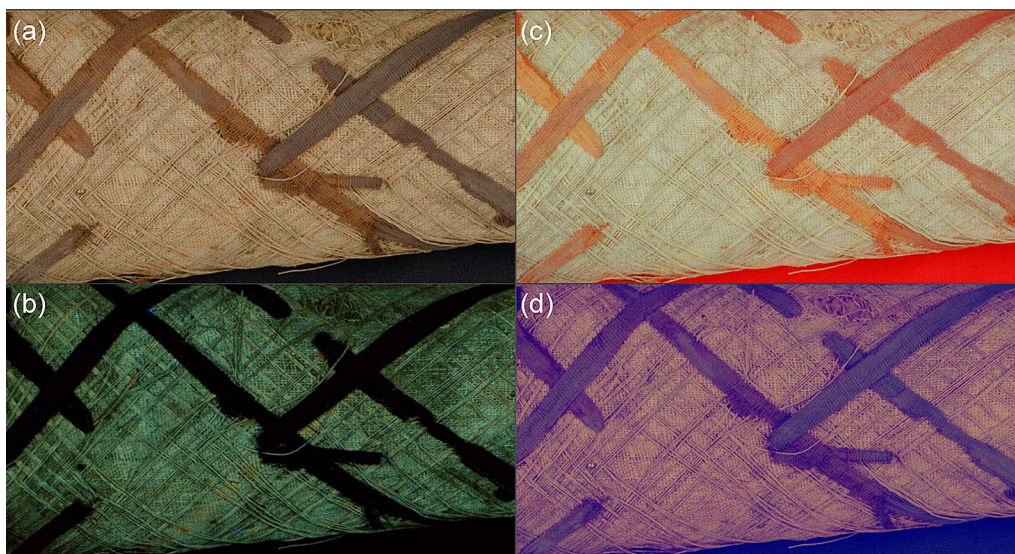
By comparison, areas of soiling are usually characterised by strong absorptions in the UVL images. This is most clearly observed in areas of the wrappings of F1, particularly on the left side of the falcon, which appears dark in the UVL image (Fig. 4b). In addition, the VIVL image (Fig. 4e) enhances the contrast between the areas of soiling and the surrounding wrappings.

It should be noted that, although recorded for the other mummies, the VIVL images have not hitherto been discussed. This is because the use of this technique for the visualisation of the luminescence from faded wrappings is limited, as the generally weak response from faded dyes is overwhelmed by luminescence from the linen in the same wavelength range.

Another notable feature of the wrappings of F1 is that they appear to exhibit yellow luminescence under UV illumination (Fig. 4b), particularly the head and vertical strips along the right side of the body. Although no further information can be gleaned from the other available



**Fig. 2** a Visible-reflected (VIS); b UV-induced visible luminescence (UVL); c Infrared-reflected false colour (IRRFC); d UV-reflected false colour (UVRFC) images of the front central section of C3



**Fig. 3** a Visible-reflected (VIS); b UV-induced visible luminescence (UVL); c Infrared-reflected false colour (IRRFC); d UV-reflected false colour (UVRFC) images of the front central section of C1

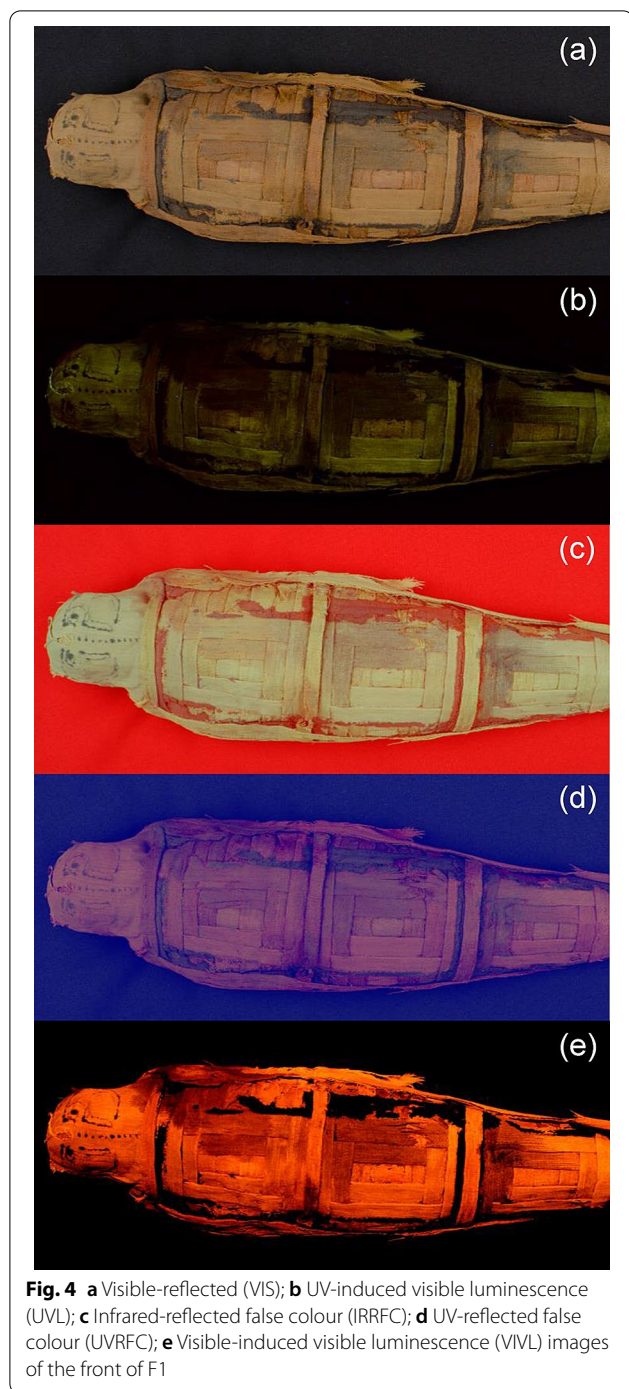
images, the clear difference when compared with the other mummies investigated may indicate that these linen wrappings were once dyed yellow.

**FORS**

In a similar way to that described for the broadband MSI observations, the results obtained non-invasively by FORS also fall into five categories, which are represented in Fig. 5.

The reflectance spectra taken from the pink/reddish areas of the wrappings of C1, C5, I4 and F1 showed a weak absorption band centred at *ca.* 530–540 nm (Fig. 5a). The red dye extracted from safflower produces a distinctive reflectance spectrum with a single relatively sharp absorption feature around these wavelengths [31, 41]. Although the positioning of the absorption band in the pink/reddish areas of mummies C1, C5, I4 and F1 corresponds to safflower red, the weakness of the signal





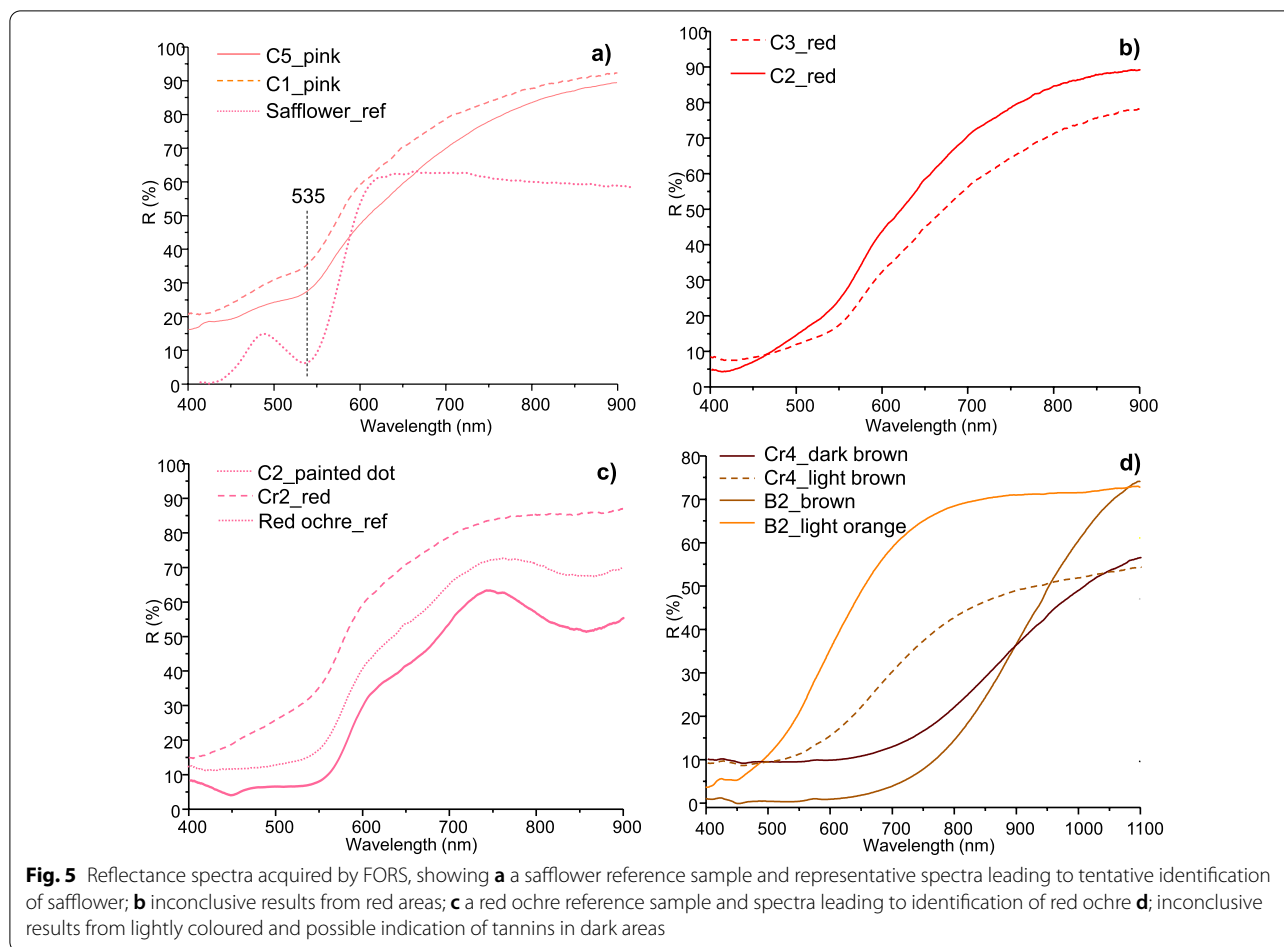
enabled safflower to only be tentatively identified as the source of colour. This is due to the very light and faded colouration left on these textiles. The result is in agreement with MSI observations of C1 and F1. In the case of mummies C2 and C3, the reflectance spectra of the reddish areas of the wrappings showed a weak absorption at *ca.* 545–555 nm (Fig. 5b). MSI observations had pointed

to the presence of madder in these areas, but only weak and inconclusive structural features were observed in the reflectance spectra. Red ochre was identified in the painted areas present on the face of the cat mummy C2 and on the wrappings of Cr2 (Fig. 5c). In the case of red ochre, all the characteristic absorption features were evident, such as the two inflection points around 575–590 nm and 700 nm, the characteristic positive slope in the region above 600 nm, and the absorption band due to ligand field transition centred around 850–900 nm [31, 41]. Regarding the black and brown textiles, FORS spectra suggest that the colour may have been obtained by using tannins in combination with iron, as already found for basketry [42] and leather tanning [43]. Although spectral features are poor, the general trend of the spectra—with an inflection point occurring above *ca.* 730 nm (Fig. 5d)—has been highlighted as characteristic for a variety of hues in the range from light brown to black obtained with iron-tannin complexes [44]. In the case of light colours, such as yellow, beige, light orange, etc., the absence of distinguishing features in the FORS spectra (two examples are reported in Fig. 5d) was interpreted as the possible absence of colouring material, although the presence of yellow dyes, as suggested for F1, could not be excluded based solely on these data.

#### Visible and UV optical microscopy

Samples were taken from specific areas of interest. It was demonstrated in a previous article that the observation of the samples with an optical microscope under visible and UV light can produce valuable information about the nature and distribution of the colouring materials on the fibres, and consequently about the dyeing methods used to apply them [31]. The images obtained for textile threads detached from the animal mummy wrappings can also be grouped into categories, as represented in Fig. 6.

The samples from the pink/reddish areas of C1, C5 and I4 exhibited an uneven distribution of the colour on the fibres. The intensely coloured areas showed a bright orange UV-induced luminescence (Fig. 6a–a'), in agreement with the use of safflower [31, 37], as also suggested by FORS and MSI. However, FORS and MSI suggested the presence of safflower in the red area of F1 as well. When observed under the microscope, the red sample from F1 (not shown) showed similar features to the red samples from C2 and C3 (Fig. 6b–b'). In particular, weak pinkish UV-induced luminescence was observed, which was not useful to indicate a specific red colourant. All the other “lightly coloured” samples, which produced featureless FORS spectra, looked similar with OM: yellowish under visible light with a weak yellow/beige appearance under UV illumination (Fig. 6c–c'), which is



typical of lignocellulosic fibres, such as flax [35]. Interestingly, a brighter luminescence was generally observed from the “knee-joint” (also referred to as kink bands [45]) areas of the flax fibres, whether the fibres were dyed or not. It appears that these characteristic anatomical areas have a slightly different chemical composition in terms of relative abundance of lignocellulosic material [46], which may justify the brighter emission observed. This difference in chemical composition also appears to result in stronger bonds with the dyeing materials compared to other areas of the flax fibres, and ultimately in the fact that all the luminescence/absorbance phenomena are emphasised in these “knee-joint” areas.

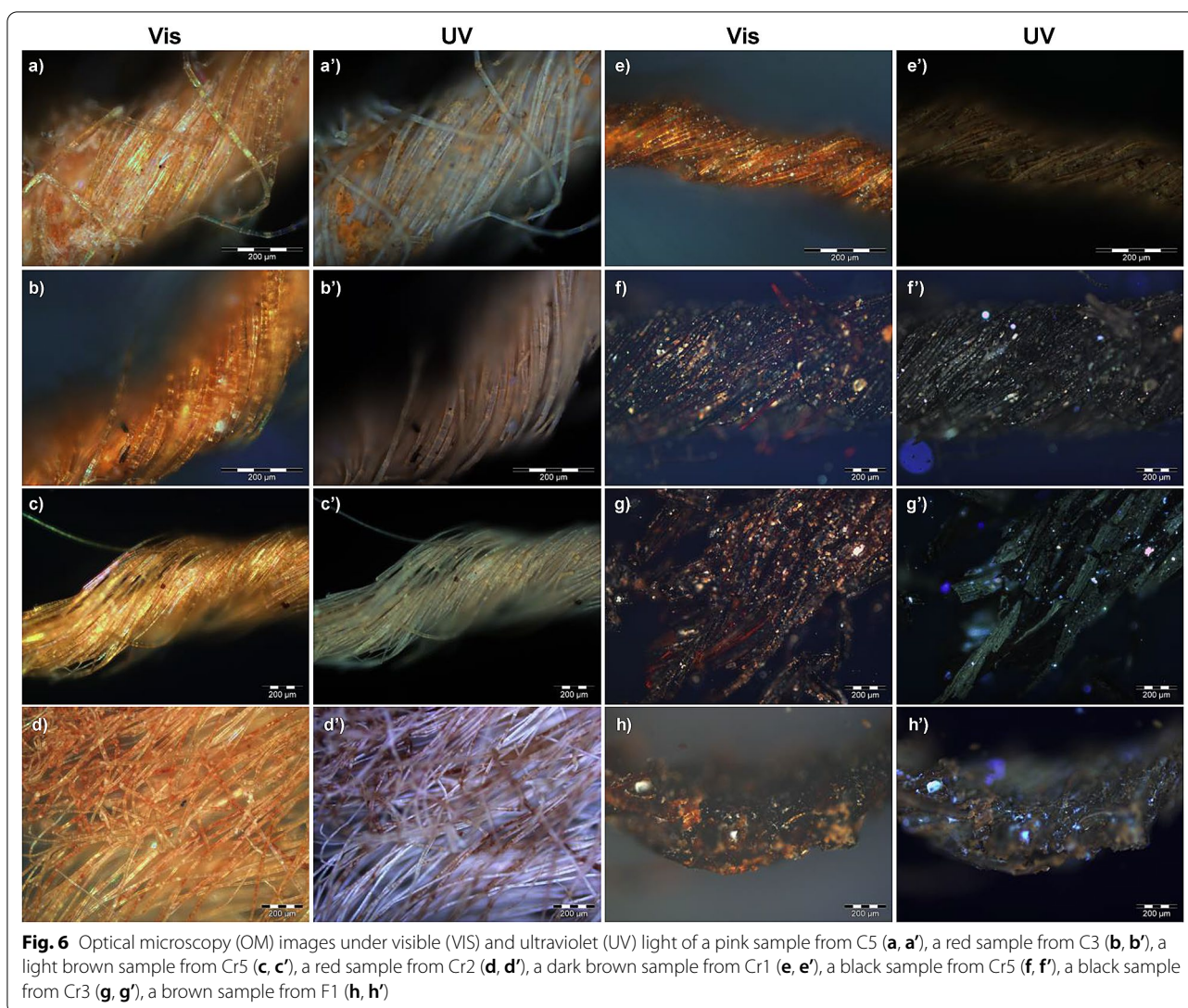
As for the red sample from Cr2, the fibres appeared non-homogeneously coloured in vis-OM, and a fine dispersion of absorbing particles was clearly observed under UV illumination in correspondence to the red areas (Fig. 6d–d’). This is consistent with the UV-absorption properties of red ochre [47] and previous observations on textile samples from human mummies [31].

Most brown samples showed an uneven distribution of the colour in Vis-OM and a prevalent absorbance of

the radiation in UV-OM (Fig. 6e–e’). The extent of the absorbance generally varied with the brown shade of the samples. In lighter brown samples (C1, C3, C4, C5, Cr1, Ca1, Ca2, F2) the fibres were still visible under UV light, and the absorbance of these was enhanced in the “knee-joint” areas. In darker brown samples (I1, I4, I6, Cr4, Ca1) the colour was more homogeneous and UV radiation was almost completely absorbed. A similar scenario was observed for the black samples, such as Cr5 (Fig. 6f–f’). The strong absorbance of UV radiation is in agreement with the use of tannins [35].

The black sample from Cr3 showed unusual characteristics under the microscope, with some of the fibres looking flattened and emitting a greenish UV-induced luminescence (Fig. 6g–g’). These observations were interpreted as the presence of abundant non-fibrous plant material from a reduced processing of the flax fibres. This topic is examined in more detail in the Discussion section.

Some dark samples from I2, I5 and F1 showed an abundance of organic material “coating” present on the fibres. Some areas emitted a dark yellow luminescence



(Fig. 6h–h') under UV light, which might be indicative of this material being mainly lipidic in composition [48]. Although the identification of all embalming/mummifying materials was beyond the scope of this study, some additional information is given in the next section.

#### HPLC-ESI-Q-ToF

The use of safflower was confirmed in the pink/red samples from C1, C5, I4 and F1, thanks to the detection of carthamin ( $[M-H]^-$  at 909.210  $m/z$ ) and two carthamin degradation products ( $[M-H]^-$  at 449.109 and 477.104  $m/z$ ), in agreement with published data [49–51].

The red samples from C2 and C3 did not produce any significant results in terms of coloured organic molecules, leaving the identification of the colourant used in these mummies uncertain. Although the weak UV-induced luminescence from these areas and the observation of

some structure in the FORS spectra may suggest the use of an organic red dye, such as madder, the sensitivity of HPLC-ESI-Q-ToF should have allowed for the detection of the anthraquinones, despite the very small amount of sample used (*ca.* 1 mm of thread). Nevertheless, the fading and degradation of the sampled area might have hindered the ability of the technique to identify dye molecules. Further investigation, probably with slightly larger samples, would be needed to clarify this point.

The other lightly coloured samples, described as light yellow, light orange, pink, light brown, etc., also produced no results from HPLC analysis, confirming the hypothesis that these shades of colour are likely to correspond to the natural yellowing of the linen and/or the presence of additional organic materials. In this regard, although the HPLC method was not optimised for the detection of materials other than dyes, several non-coloured

molecules were detected in all samples. The molecular ions of three of these molecules were identified at  $m/z$  171, 187 and 203, which correspond to octanedioic, nonanedioic and decanedioic acids, respectively. These are products of the autoxidation of siccative oils and other lipids [52, 53], confirming the presence of such materials as part of the embalming and mummification processes.

All the brown and black samples showed the presence of tannins. Tannins are a broad category of chemical compounds divided into two main categories, namely, hydrolysable and condensed tannins. Ellagitannins and gallotannins are classes of hydrolysable tannins, in which glucose units form polyester compounds with ellagic and gallic acid units, respectively. Condensed tannins are polymeric compounds of catechin, anthocyanidin or their derivatives, also referred to as proanthocyanidins [54]. The identification of the plant source of tannins is still very challenging by HPLC in most cases, as tannins are large molecules generally containing many isomers, which make the chromatographic separation very difficult [55]. Even with the adoption of mass spectrometry detection (HPLC–MS) [56–58], the common scenario with archaeological samples is to detect some of the degradation products of hydrolysable tannins (gallic acid, ellagic acid, hydroxybenzoic acids, etc.) [59, 60], or to observe clusters of  $m/z$  values that are indicative of the general presence of condensed tannins [59, 61]. This was the case for our samples. All dark samples, except for C3, showed the presence of ellagic acid ( $[M-H]^-$  at 300.999  $m/z$ ), indicative of the use of ellagitannins. Additionally, the brown samples of I3, I6, Cr4 and F2 showed significant amounts of other degradation products of hydrolysable tannins, such as 4-hydroxybenzoic acid ( $[M-H]^-$  at 137.024  $m/z$ ) and 3,4-dihydroxybenzoic acid ( $[M-H]^-$  at 153.019  $m/z$ ). The brown and black samples from C3, I2, I5, Cr3 and Cr5 also showed the possible presence of condensed tannins, suggesting that more

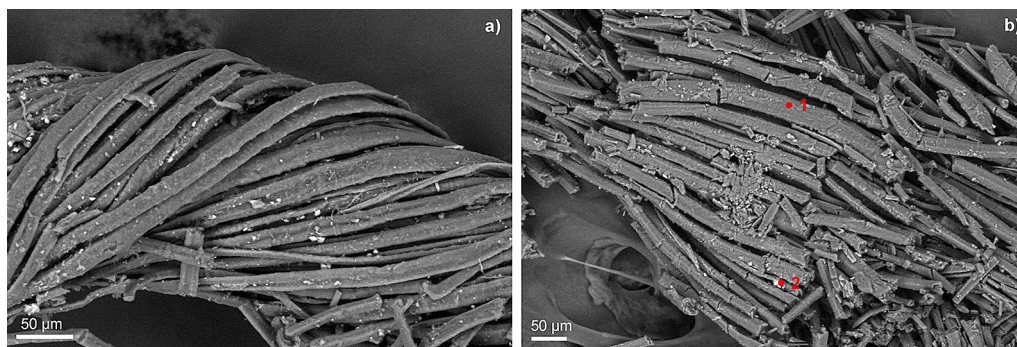
than one source of tannins were likely used to obtain very dark colours. It is worth highlighting that bitumen has also been hypothesised as a material used to decorate archaeological textiles, for example the late 5th millennium BCE textiles from the Cave of the Warrior (Jericho, Israel), by smearing and pressing of the material onto the textile [62]. As bitumen has to be targeted with specific analytical protocols [24, 63–65], this fell beyond the scope of this article. However, research is in progress to clarify the possible use of bitumen in this context.

### SEM–EDX

The SEM images enabled the preservation state of the fibres to be evaluated at the micro-scale. As a general trend, the dark samples showed a more advanced state of degradation than the red or lightly coloured samples, as detailed in Table 2 and represented in Fig. 7. For relatively well preserved fibres (Fig. 7a), a smooth surface with few defects and cracks was observed. In poorly preserved samples (Fig. 7b), the fibres appeared brittle with numerous cracks both tangential and transversal.

Particles were also generally present, in some cases in low quantity and, in other cases, highly abundant. The elemental composition of the particles was studied by EDX in an attempt to distinguish between environmental contamination and other more significant particles. In addition, EDX spectra were taken on areas of the fibres that appeared free from contamination and/or particles, in order to investigate the possible presence of mordants, especially iron, as this was expected to be used in combination with tannins to obtain dark colours.

In general terms, in addition to C and O, common elements, including Al, Si, S, K and Ca, were detected in very low amounts in cleaner areas of the fibres (Table 3, spectrum 1). This result was therefore considered as the baseline when interpreting the results on other, more complex areas of the fibres. The most common particles,



**Fig. 7** Back-scattered electron SEM images of **a** the red sample and **b** the brown sample from C3. The red dots indicate the locations of EDX point analysis, the results of which are reported in Table 3

**Table 3** Results of EDX point analysis at the locations shown in Figs. 7 and 8

	C	O	Na	Mg	Al	Si	P	S	Cl	K	Ca	Fe	Ag
1	98.55	0.80			0.11	0.14		0.07	0.14	0.07	0.12		
2	45.50	43.40		0.74	2.75	5.69				0.27	0.49	1.16	
3		20.37	40.73						38.9				
4	25.01	64.51	0.41	0.65	1.3	3.84	0.2		0.31	0.53	0.26	2.98	
5	32.76	66.09	0.67					0.05	0.33	0.05	0.04		
6	29.96	64.2	1.4						0.86		3.58		
7	41.81	46.03			6.90	5.25							
8	50.17	35.04	0.37	1.25	4.02	5.08	1.18				1.25	1.42	
9	66.76	31.73	0.36		0.23	0.73		0.11	0.26	0.28	0.14	0.47	
10	42.19	40.39	0.87	0.75	1.07	4.52	0.21	0.87		0.49	4.28	4.28	0.09
11	92.54	4.63				0.4		0.87	0.36		0.07	1.13	
12	60.38	21.52		0.25	2.03	12.66		0.21		0.49	0.98	1.29	
13	91.43	4.27						0.30	0.45	0.36	1.56	1.62	

Figures should be interpreted as semi-quantitative estimations of atomic concentrations (%)

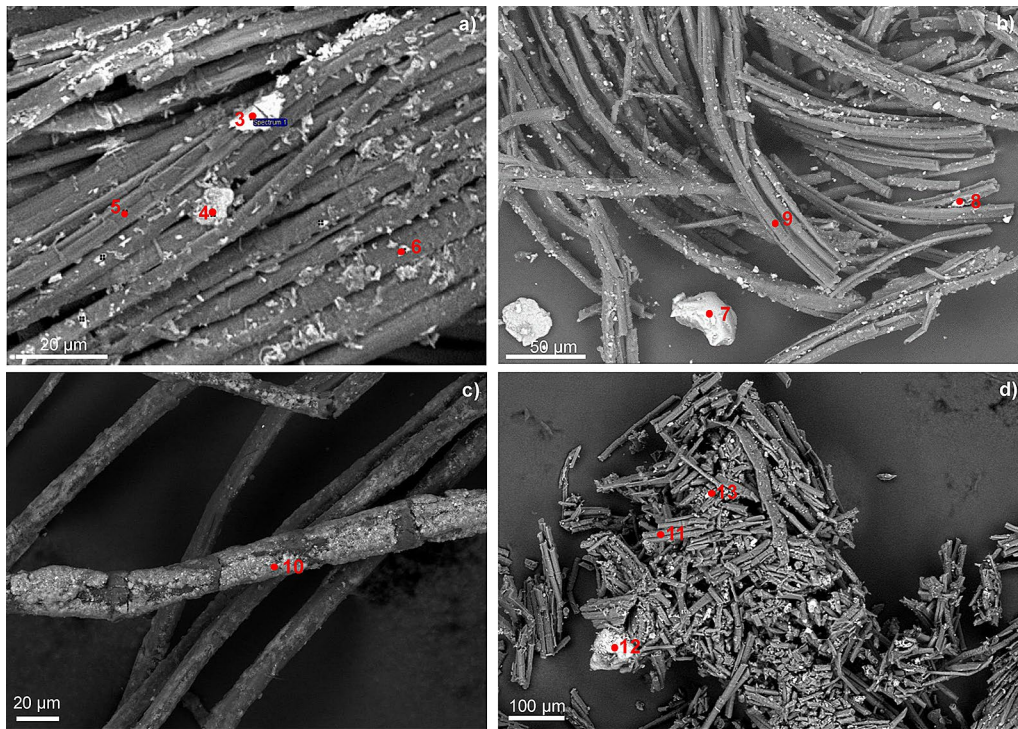
such as those observed in both samples shown in Fig. 7, were rich in Al and Si with smaller amounts of Fe (Table 3, spectrum 2). These were considered to be environmental contamination, as this composition is typical of dust, soil or dirt [31]. Consequently, we determined the use of Fe in combination with tannins, only when Fe was significantly present in non-contaminated areas of the fibres. This was clearly the case for the dark samples from I2, I3, I4, I5, I6, Cr1, Cr3, Cr4, Cr5, Ca1 and Ca2. An example of the elemental composition obtained in the black sample from I2 is provided in Table 3 (spectrum 11). Interestingly, in the case of Cr4 and Ca1, for which both light brown and dark brown colours were present, only the dark brown areas appeared to contain a significant amount of Fe.

In addition to the possible use of Fe in combination with tannins, a series of additional observations were obtained from SEM–EDX data. Some samples showed the presence of particles with an elongated crystal shape, which were found to be mainly composed of Na and Cl (Fig. 8a). This was particularly evident in C4, I3 and I5, but the presence of Na and Cl in small amounts was relatively common in most of the textile wrappings. This observation may be interpreted as the presence of natron, a naturally occurring mixture of sodium carbonate decahydrate ( $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ ), sodium bicarbonate ( $\text{NaHCO}_3$ ), sodium chloride ( $\text{NaCl}$ ) and sodium sulfate ( $\text{Na}_2\text{SO}_4$ ), which was used as a drying agent during the mummification process [13]. However, the presence of Na, Cl, and even Ca and K, opens up an additional aspect to be considered: the possible bleaching of linen. Flax whitening was a common practice in ancient Egypt and different methods are known. One entailed the simple exposure of the linen cloths to sunlight, whereas another

implied the use of a water solution containing lye from wood ashes (mostly NaOH and/or KOH) and lime (a mixture of Ca oxides and hydroxides) [66, 67]. The use of such methods would possibly result in the presence of Na, Ca, K and Cl on the fibres, and the presence of alkaline compounds has already been tentatively linked to bleaching procedures [68]. These elements were found on both dyed and undyed samples, which, in the light of the bleaching hypothesis, would mean that whitening was performed before dyeing, and not only to obtain whiter fibres, but possibly in an attempt to obtain a better final colour. Further scientific investigation could provide additional evidence for the practice of bleaching linen in ancient Egypt.

A separate discussion is needed for Cr2. The presence of red ochre was determined in the reddish areas of the wrappings of this crocodile mummy. The SEM images (Fig. 8c) showed the fine dispersion of Fe-rich particles on the fibres, confirming similar observations obtained on other mummy wrappings coloured with red ochre [31]. Impurities of Ag were also detected (Table 3, spectrum 10), testifying to the variety of elements that are occasionally detected when scrutinising these ancient fibres.

Additional observations can be made on the processing of the flax fibres. Flax fibres are bast fibres, present in the phloem (inner bark) of the plant, and, in order to be separated from the xylem, a retting process is generally required [69]. During the prolonged soaking of the stalk in water, pectin, the substance keeping the phloem and xylem together, breaks down, and the fibres are separated from the other phloem cells, so that they can be further processed and spun into yarn. The accuracy of the processing generally determines the final quality of the fibres

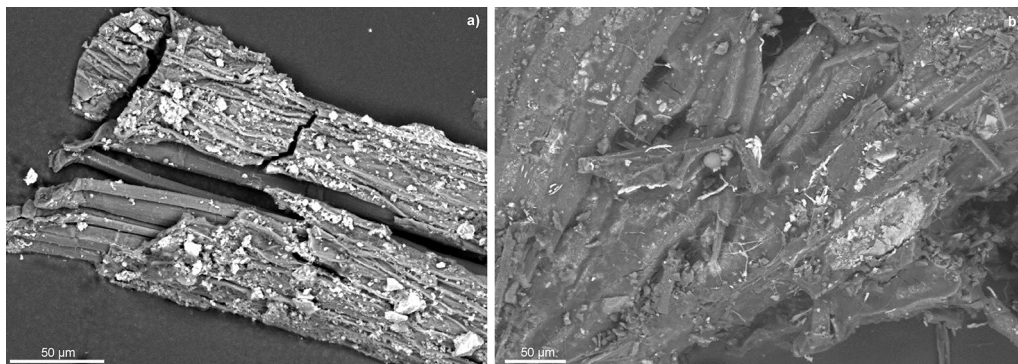


**Fig. 8** Back-scattered electron SEM images of **a** the brown sample from C4, **b** the dark brown sample from Cr1, **c** the red sample from Cr2, and **d** the black sample from I2. The red dots indicate the locations of EDX point analysis, the results of which are reported in Table 3

and threads [45]. It was evident in some samples (see summary in Table 2) that the additional plant material was still covering the bast fibres, as can also be observed in Fig. 9a for the Cr3 mummy. This was also partially observed using OM for this sample (Fig. 6g–g'). The method in use from the Pre-Dynastic Period of ancient Egypt to join by hand a bundle of flax fibres into a continuous yarn is known as splicing [70]. Recently, it has been reported that splicing, unlike drafting, involved partial or no retting of the plant, as the fibres were mechanically

removed from the dry, unretted stalks by a process called decortification. As a result, pectin and outer layers of plant tissue remain on the surface of the fibres, which are collected as strips and joined individually into a yarn [69].

In the case of F1, very different SEM images were obtained, especially for the brown sample (Fig. 9b). In addition to showing the presence of a coating material, which was already suspected based on OM, the presence of fungal hyphae and spores was evident, thus indicating a possible on-going biodegradation of the sample and,



**Fig. 9** Back-scattered electron SEM images of **a** the black sample from Cr3 and **b** the brown sample from F1

more generally, of the mummy. In fact, the phenomenon was also visible in the yellow and red samples from F1, although to a lesser extent. The presence of a coating material might also be related to the splicing process, in terms of both partial retting, which implies pectin to remain on the fibres, and possible addition of a sort of adhesive, which is sometimes necessary to obtain yarns from spliced fibres [69].

## Discussion

The high number of analyses created a large amount of data, and their interpretation was not always straightforward. The main aim of the study was the identification of the colouring materials used in the wrappings of the animal mummies under investigation. In this regard, the investigation was successful, as both safflower and red ochre were identified as the main materials used to obtain red/pink colours. In a previous investigation on human mummies, madder was also identified as a source of red [31]. Although madder was not identified in the present study, we do not exclude its use in animal mummies wrappings, especially considering the availability of madder as a common botanical source of the dye in Egypt.

The identification of tannins, both hydrolysable ellagitannins and condensed tannins, was obtained in all the dark coloured areas. Very dark colours were found to correspond with the use of condensed and hydrolysable tannins in combination with an iron mordant, as opposed to lighter brown colours, which were possibly obtained by using only hydrolysable tannins without iron. It is widely accepted within the field of conservation that iron and tannins are a dangerous combination when it comes to fibre damage and that, generally, very dark colours correspond to poorly preserved textiles [71]. The main reason for this degradation is attributed to the presence of iron ions, which trigger autoxidation of the long chain polymeric carbohydrates [72, 73]. Degradation due to iron compounds in wrappings of animal mummies has been suggested by Bruno [74]. This effect appeared to also be generally true for the textiles under investigation in this study, in which the black textiles of the ibises, for example, showed higher levels of iron and the worst states of preservation. Nevertheless, the SEM–EDX observations enabled attention to be drawn to additional important causes of fibre degradation, which should be taken into consideration, such as the possible use of natron and bleaching agents, the effectiveness of the flax fibre processing (especially retting), the yarn-making technology adopted, and the possible presence of fungal attack. With particular attention to processing and technology of the flax fibres, a recent publication provides guidelines to identify the use of splicing [69]. It was not straightforward

to identify splicing in the samples discussed in this study, but different levels of fibre processing were observed, as summarised in Table 2, where “reduced” processing is an indication of abundant non-fibrous material visible in the SEM images, as opposed to “advanced” processing, which indicates relatively clean fibres.

All these factors can play a role in the degradation of the cellulosic fibres [75], therefore the correlation between the preservation state of the fibres and the colouring materials used to dye them appears less straightforward than a simple correspondence with high levels of iron and/or use of tannins. Non-fibrous material left after reduced retting, for example, may in principle protect the flax fibres. However, pectin and other fibre coating materials (either naturally present or added during conservation treatments) may attract micro-organisms and promote biodegradation.

The topic of technologies that were used to purposefully alter the integrity of the fibres to enhance the effect of dyes is also part of a broader conversation, as recently reported by Späth et al. [76], who suggest that the bright colours of the Pazyryk carpet are at least partially related to a process of wool fermentation carried out before dyeing the fibres. It is likely that similar practices were more widespread than what currently reported.

## Conclusions

The textile wrappings of twenty animal mummies from the collections of the British Museum (London, UK) and the Museo Egizio (Turin, Italy) were investigated using a multi-scalar/multi-analytical approach with the main aim to identify the colouring materials used to dye the textiles. Non-invasive techniques, such as broadband MSI and FORS, provided fundamental preliminary information about the distribution of the colourants, as well as the distinction between intentionally dyed areas and colours perceived as a result of the natural ageing of linen, staining or the presence of additional organic materials. A tentative identification of safflower (*C. tinctorius*) was also possible non-invasively in some of the red/pink areas, whereas red ochre was undoubtedly identified by FORS. However, confirmation of the organic dyes used was obtained by HPLC-ESI-Q-ToF, which provided ultimate identification of safflower and tannins, both hydrolysable and condensed, at a molecular level.

The observation of the samples with OM under visible and UV light produced valuable additional information about the distribution of the colouring agents on the fibres, and about the presence of additional organic materials. SEM images were useful in studying fibre processing, degradation and environmental contamination. EDX point analysis enabled the use of iron in combination with tannins to be highlighted in the darkest

and most degraded samples. Intriguing observations were also made concerning the processing of the flax fibres—particularly reduced retting and possible use of a splicing technique -, as well as the possible use of bleaching agents and the presence of fungal degradation. This shows that the evaluation of the degradation state of these textiles is a more complex matter than the simple presence of iron/tannins. Synergistic effects are most likely to occur and trends in degradation are not straightforward, thus making it necessary to carry out evaluations on a case-by-case basis.

Finally, these results underline the fundamental importance of a combination of different analytical techniques in this type of investigation, thus connecting information discerned from the macro-scale down to the molecular level. This dataset also represents a solid background for further scientific research aimed at clarifying significant aspects of fibre and textile processing in ancient times, in order to provide conservators and curators with additional tools to plan the preservation and display of animal mummies.

#### Abbreviations

MSI: Multispectral imaging; FORS: Fibre optic reflectance spectroscopy; OM: Optical microscopy; SEM: Scanning electron microscopy; EDX: Energy dispersive X-ray spectrometry; HPLC–MS/MS: High performance liquid chromatography tandem mass spectrometry; CT: Computer tomography; FTIR: Fourier transform infrared spectroscopy; GC–MS: Gas chromatography–mass spectrometry; Py–GC–MS: Pyrolysis–gas chromatography–mass spectrometry; UV: Ultraviolet; micro-XRF: Micro-X ray fluorescence spectrometry; HPLC–ESI–Q–ToF: High performance liquid chromatography electrospray ionisation quadrupole time-of-flight; VIS: Visible; UVL: UV-induced visible luminescence; IRRFC: Infrared-reflected false colour; UVRFC: UV-reflected false colour; VIL: Visible-induced visible luminescence.

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#### Authors' contributions

DT: Investigation (HPLC–ESI–Q–ToF); Formal analysis; Data Curation; Writing—Original draft. JD: Investigation (MSI and FORS); Visualisation; Data Curation; Writing—Review and editing. MV: Conceptualisation, Resources, Writing—Review and editing. MB: Conceptualisation, Resources, Writing—Review and editing. DA: Conceptualisation, Investigation (optical microscopy), Writing—Review and editing. MA: Investigation (FORS), Writing—Review and editing. CO: Conceptualisation, Resources (sampling); Writing—Review and editing. FF: Conceptualisation, Resources, Writing—Review and editing. SA: Conceptualisation, Resources, Writing—Review and editing. PD: Investigation (SEM–EDX), Writing—Review and editing. MG: Supervision, Data curation. Writing—Review and editing. All authors read and approved the final manuscript.

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#### Availability of data and materials

The datasets used and/or analysed during the current study are available from Dr Monica Gulmini, Dr Diego Tamburini and Dr Joanne Dyer upon reasonable request.

#### Declarations

##### Competing interests

The authors declare that they have no competing interests.

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