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A Royal Mystery: A Multianalytical Approach for Dyestuff Identification in Seventeenth Century Waistcoats

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Abstract: Early modern materials are not well represented in dye and mordant analyses despite extensive documentary evidence suggesting the enormous demand for coloured fabrics, even among those below the elite. Non-wovens likewise receive less attention than woven textiles despite their ubiquity in the early modern historical record. Knitted garments, in particular, have rarely been subjected to dye analysis. One garment is noteworthy for its colourfulness, despite not being visible in formal wear. Men throughout society wore knitted undergarments known as waistcoats from the late sixteenth century. The waistcoats under investigation here are from the collections at the London Museum and the Grimsthorpe and Drummond Castle Trust, Scotland. They are made of silk and are now a pale blue-green colour. Small samples were taken from each and subjected to a series of analytical techniques: micro-Raman spectroscopy, UV-Vis microspectrofluorimetry, and high-performance liquid chromatography (HPLC) coupled with a mass spectrometer. Using this protocol, it was possible to characterise the dyes in the waistcoats by ensuring that maximum information was gleaned from a sample before it was exhausted.

Keywords: knit; silk; dye analysis; indigo; yellow dyes; early modern

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1. Introduction

A knitted silk waistcoat (inventory number A27050) associated with Charles I was bought for the London Museum in 1924, and arrived with a note suggesting that it was worn at the king's execution on 30 January 1649. Soon after its acquisition, it was referred to as "a rather grim relic" and described as being of "faded blue silk" [1]. Its colour has not been the focus of study before. This paper reports a multi-analytical approach to identifying the dyestuff(s) used to colour it and another garment of a similar type.

The seventeenth century term for a garment worn for warmth over a shirt and under a doublet or as informal wear was "waistcoat", although the example at the London Museum has often been described as "the vest worn by Charles I on the scaffold" [1]. The king was reported to have worn a "Sky-colour satten Wastecoat" by an eyewitness to his preparations for death [2]. A satin garment would have been tailored from a woven silk fabric, not knitted, but that the king owned blue waistcoats is supported by other contemporary accounts. An account dated 1632 to 1633 kept by George Kirke, Gentleman of the Robes, confirms the purchase of "a skieculler satin waistcoat, with two silver bone laces in a seame with linings, coller, buttons and buttonholes lined with skie-culler taffetei" [3], and a portrait by Goddard Dunning from 1649 shows Charles I in a light blue woven fabric waistcoat or doublet [4]. Waistcoats also appear in the wardrobe accounts for Charles I among garments known to be knitted, such as hose and "tennis sockes", which were supplied by haberdasher Thomas Robinson [3]. Some were simply described as silk and

may therefore have been undyed, but at least one was a "fine Carnacion [pink] silke wastcoate" purchased in 1635 [5].

Previous analysis undertaken in the 1950s, the 1980s, and the 2010s focused on the stains on the front of the garment, which had been reported to be royal blood [1]. The first test took place in 1959 (on the 310th anniversary of Charles I's death) at the London Hospital Medical College. The anti-human globulin test was used to detect human protein. Some of the stains gave positive reactions, but these were weak [6]. The stains were analysed a second time in 1988 in preparation for a display in 1989 (the 340th anniversary). The waistcoat was tested for blood using the Kastle Meyer Test. This was negative, but it was noted that "blood could have degraded" and that the waistcoat could have been washed. The report stated that "blood, urine, sweat, semen, vomit, food, drink and cosmetic preparations are all possible sources of the staining, but since non-destructive tests for those substances would almost certainly give negative or misleading results after 340 years, their true nature remains a matter of conjecture" [7]. Further analytical methods were explored in 2010 (following the 360th anniversary) with the Forensic Science Service, but it was deemed unlikely that contemporary tests would lead to any clear results. Chemical tests for blood had not changed since 1989 and were, therefore, of limited value. The waistcoat was most recently shown in an exhibition in 2023, just before it reached 100 years in the collection. This made 2024 another suitable anniversary to report investigations into it again. This time around, its colour was a focus of study.

Approximately 60 knitted silk waistcoats are in collections worldwide, usually dated to between 1590 and 1700. Two-thirds were of more than one colour and were often knitted and/or decorated with metallic thread. These have been described as "brocade-knitted" [8]. A red and gold example is in the collection of the National Museums Scotland, Edinburgh (inventory number A.1973.28). The other third of the knitted silk waistcoats are described as "damask-knitted". These are monochrome and have motifs in patterns created by the use of different stitch types. Most of the brocade-knitted waistcoats were brightly coloured (coral, green, blue, or yellow). The damask-knitted examples are red, green or pale blue [8]. There are examples of seventeenth century silk garments that preserve their bright colours, such as a red waistcoat worn by William III (Historic Royal Palaces, inventory number 3503038), but none have been subjected to dye analysis that has been published.

The London Museum waistcoat is damask-knitted in a pale blue-green colour on the outside with a brighter blue inside and on the back of the garment (Figure 1). Some of the raised surfaces of the knitted loops are notably yellow even to the naked eye, suggesting either that the blue colour has been rubbed away, exposing the natural silk colour, or that both blue and yellow dyes were used to colour it. The hypotheses under investigation were whether the waistcoat was dyed with indigo, suggesting that it had originally been a bright blue, or that it was also dyed with a yellow dye, which would make its original colour more likely to have been green.

Previous work on seventeenth century silk garments from grave contexts has shown that a multi-technique approach is fruitful for analysing dyes [9]. High-performance liquid chromatography coupled with diode array detection (HPLC-DAD) has been used to characterise dyes in seventeenth century beige, green, and light blue silk brocade [10]. Thus far, these techniques have not been applied to any knitted silk fabrics from this period.

Information that came to light during this study provided clear evidence that the London Museum waistcoat was washed during the twentieth century. The student intern who washed it on the instructions of the then head of conservation, remembers that it changed colour slightly, becoming "a little paler" [11]. One of the limitations of this study is not knowing how likely it is that dyestuffs were washed away entirely either during this event or previously in the garment's history.

Figure 1. A knitted silk waistcoat (inventory number A27050) associated with Charles I. Image: © London Museum.

The initial research on the London Museum's waistcoat used microfadeometry to aid decisions about lighting for its future display. Five small areas (0.3 mm each) were subjected to a probe of bright light to induce accelerated colour change by specialists at the National Archives at Kew (UK). This demonstrates the rate at which the colour is likely to fade according to the international standard for assessing lightfastness (ISO 105 B02). It showed that the waistcoat had already faded considerably and indicated the presence of indigo and the possibility of a yellow dye.

A similar example to the waistcoat in the London Museum is in the collection of the Grimsthorpe and Drummond Castle Trust, Scotland (inventory number DCT0032) in the same pale blue-green colour with yellow raised areas (Figure 2). It is said to have been worn by the Earl of Perth in the seventeenth century, but precisely which of the earls is not clear. Two other similar waistcoats are at the National Museum, Oslo (inventory numbers OK-dep-01162 and OK-08800), although they are embellished with silver thread over the pale blue-green knitted fabric.

The opportunity for the collection of samples from these two waistcoats allowed for the characterization of the dyes present, shedding light on their manufacture. A multi-analytical approach was used: micro-Raman spectroscopy, molecular fluorescence in the visible, and high-performance liquid chromatography (HPLC) coupled with mass spectrometry (MS).

The original colours are poorly preserved in the samples (the pale blue-green silk waistcoats) (Table 1). HPLC-MS analysis was able to identify the dye sources present. However, to provide a database of techniques that do not require sample pre-treatment, Raman and molecular fluorescence data were presented. This will allow these dyes to be identified in textiles which cannot be sampled in the future.

Table 1. The two early modern waistcoats studied.

Location	Fibre	Inventory Number	Colour (by Visual Observation)
London Museum	Silk	A27050	Pale blue-green
Drummond Castle	Silk	DCT0032	Pale blue-green

Figure 2. A knitted silk waistcoat (inventory number DCT0032) in the collection of the Grimsthorpe and Drummond Castle Trust, Scotland. Image: © Jane Malcolm-Davies.

The use of these techniques permitted a thorough characterisation of the two waistcoats, leading to a better understanding of the production of such textiles in the seventeenth century. It also initiated the development of a database for further studies of waistcoats from the early modern period.

2. Materials and Methods

2.1. Materials

Spectroscopic or equivalent grade solvents and Millipore-filtered water were used during all the experimental work. Qualitative filter paper from Filter Lab was used. Each chromophore (luteolin, luteolin-7-*O*-glucoside, and quercetin) was complexed with Al^{3+}. Solutions were prepared in methanol/water (70:30, v/v), at 5×10^{-4} M. Complexation of molecules with Al^{3+} (where Al^{3+} is present $\times 100$ in respect to the molecule) was performed with the addition of $AlCl_3$ (0.1 M). Six drops of each solution were then applied on filter paper with a micro-pipette (10 µL). Three replicates for each reference were prepared, and analyses were carried out on the same day as (or the day after) application on filter paper. The filter paper is composed of almost pure cellulose without any additives, providing a support for the analysis of these chromophores. Data acquired in these reference materials composed a database, which was compared with the case studies.

2.2. Sampling

Permission for sampling the waistcoat was granted for dye analysis according to the London Museum's strict criteria. The process of taking samples followed advice from relevant literature, although it proved contradictory. It is often preferable to remove a sample from a part of the object that is already damaged to avoid further injury to its integrity. In such cases, taking a fragment that is still attached by at least one thread ensures it is part of the original [12]. However, other advice suggests avoiding deteriorated parts of the textiles altogether, as these are more likely to be compromised and yield misleading results [13]. A previous protocol for sampling knitted garments at the museum devised during the *Knitting in Early Modern Europe* project, which looked at sixteenth century knitted caps, was also consulted (Table 2) [14,15].

Table 2. Sampling strategy for knitted caps at the London Museum.

No sampling will be considered where previous conservation treatments have distorted the item's original shape.
Items must have existing damage. Caps in pristine or good condition, without any existing areas of loss, will not be sampled.
Areas for sampling are to be hidden from regular view. Sampling from the interior will be preferable to the exterior. External decorative features will not be sampled.
Samples will be taken from areas away from key features.
Multiple sampling of one item will be considered if the item has multiple parts, such as a separate lining, decoration or a strap.
No sampling will be considered in or around a hole where most or all of the material exists around the area of loss (if the hole could be completely closed through conservation).
Sampling items which have not undergone obvious conservation interventions is preferred.
No sampling will be undertaken where a proper assessment of the item is currently impossible (for example, if it has been stitched to a mount).
The maximum sample shape and size is square (0.5 cm × 0.5 cm) or rectangular (1 cm × 2 cm).
Loose dust or hairs in the item's storage box (which would otherwise be discarded) may be collected as samples.

A comprehensive survey of the garment identified two potential locations from which to take samples: fragments from the damaged front and silk thread ends from inside the garment. The damaged section was badly stained, whereas the thread ends were in very good condition. Samples were taken from each to comply with the conflicting sampling advice mentioned above. Similar samples were collected from the Drummond Castle waistcoat.

2.3. Confocal Micro-Raman Spectroscopy

Confocal micro-Raman analysis (micro-Raman or benchtop Raman) was carried out using a Horiba Jobin-Yvon LabRAM 300 benchtop spectrometer, equipped with a diode laser providing excitation at 785 nm and a maximum laser power of 37 mW at the sample. The laser beam was focused through a 50× Olympus objective lens, resulting in a spot size of 4 μm. The laser power at the sample surface was kept between 9.5 and 0.37 mW. No evidence of fibre degradation was observed during or after spectra acquisition. This system enables data acquisition in the 100–4000 cm^{-1} spectral range, with a 3 cm^{-1} spectral resolution, and a grating 1800 gr/mm. Spectra were acquired as a sum of 10–15 scans, with a 15–25 s integration time. A minimum of three measurements were collected from the same sample to ensure data reproducibility, and a silicon reference was used for calibration.

2.4. Molecular Fluorescence in the Visible

Fluorescence excitation and emission spectra were recorded with a Jobin–Yvon/Horiba SPEX Fluorog 3-2.2 spectrofluorometer coupled to an Olympus BX51M confocal microscope, with spatial resolution controlled by a multiple-pinhole turret, corresponding to a minimum 2 μm and maximum 60 μm spot, equipped with a 50× objective. Beam-splitting is obtained with standard dichroic filters mounted at a 45° angle in a two-place filter holder. For a dichroic filter of 500 nm, excitation may be undertaken up to about 490 nm, and emission collected after 510 nm. The signal was optimised daily for all pinhole apertures through mirror alignment, following the manufacturer's instructions and using a rhodamine standard (or other adequate references). For this study, one set of dichroic filters was employed: 430 nm and 500 nm, exciting at 420 nm and reading the emission signal at 510 nm, respectively.

Emission from a continuous 450-W xenon lamp, providing an intense broad spectrum from the UV to near-IR, was directed into a double-grating monochromator, and spectra were acquired after focusing on the sample (eye view) followed by signal intensity optimisation (detector reading). The pinhole aperture that controls the measurement area was selected based on the signal-to-noise ratio. In this work, due to very weak signals, a 30 μm

spot was used (pinhole 8) with the following slit set: emission slits = 3/3/3 mm (6 nm bandpass) and excitation slits = 5/3/0.8 mm (final bandpass of 2 nm). Emission and excitation spectra were acquired on the same spot. When needed, the spectra were normalised by area. When comparing spectra acquired from different samples, four different signals must be considered: the maxima of excitation and emission, the signal-to-noise ratio, the intensity of the signals, and the shape (see [15–18]).

2.5. High-Performance Liquid Chromatography (HPLC) Coupled with Mass Spectrometry (MS)

The dyes from the samples were extracted using a soft-extraction method in order to prevent any molecule degradation [19]. The textile microsamples were placed in a microtube with 400 µL of oxalic acid (0.2 M)/methanol/acetone/water (0.1:3:3:4, *v:v*) at 60 °C for 30 min. The solution was left to evaporate under vacuum, and the residues were then dissolved in 400 µL of methanol/water, 7:3 (*v*/*v*); the tubes were centrifuged, and the upper 25 µL of the solution was removed for analysis.

HPLC-ESI-Q-Orbitrap-MS analyses were performed in a HPLC Vanquish (Thermo Fischer Scientific, Bremen, Germany) coupled to an Orbitrap Exploris 120 mass spectrometer (Thermo Fischer Scientific, Bremen, Germany) controlled by Orbitrap Exploris Tune Application 2.0.185.35 and Xcalibur 4.4.16.14. The MS was operated in the ESI positive and negative ion modes, with the following optimised parameters: ion spray voltage, ±4.5 kV; capillary voltage, 16/−18 V; tube lens offset, −70/58 V; sheath gas (N_2), 40 arbitrary units; auxiliary gas (N_2), 20 arbitrary units; capillary temperature, 270 °C. The spectra typically corresponded to an average of 20–35 scans, and were recorded in a range between 100 and 1000 Da. The stationary phase was an Agilent Poroshell 120 CS-C18 column (150 × 4.6 mm i.d., 2.7 µm) at 35 °C. The mobile phases were composed by solvent A, 0.1% (*v*/*v*) formic acid, and solvent B, 100% (*v*/*v*) acetonitrile. The flow rate was 0.30 mL/min, the injection volume was 15 µL, and the gradient method started with a 2 min isocratic 7% B gradient, followed by a linear gradient ranging from 7% B to 80% B in 20 min, and then reaching 100% B in 1 min followed by a linear isocratic four minute 100% gradient; then, the column was re-equilibrated with 7% B for seven minutes [20].

3. Results and Discussion

The complementarity of the analytical techniques permitted a general overview of the materials used and their conservation conditions. While HPLC-MS identified the main molecules present and the degradation products, other techniques that do not require sample pre-treatment, such as Raman spectroscopy and molecular fluorescence in the visible, were also used. These techniques allow for the characterisation of artworks in situ or using microsamples, which are afterwards available for further analysis. By combining these techniques, it has been possible to start building a database of information that will facilitate future studies of comparable material using only Raman and/or molecular fluorescence.

3.1. HPLC-MS Analysis

The HPLC-Orbitrap-MS analysis (Table S1) allowed the identification of indigotin (Figure 3) in both of the blue-green London Museum and the Drummond Castle silk waistcoat samples ($[M + H]^+$ *m*/*z* 263.08).

No yellow dyes were identified in the London Museum's waistcoat samples, but one was found in both Drummond Castle samples. A correspondence with daphnetin ($[M + H]^+$ *m*/*z* 179.01) with MS^2 133.05 suggested that it may have been dyed with flax-leaved daphne (*Daphne gnidium*), a plant native to the Mediterranean used for dyeing yellows and greens (Figure 4). It appears in Arabic texts in the twelfth century and in a regulation published in France in 1671 warning of its health hazards. Nevertheless, it was recorded as achieving green colours from Seville in the eleventh century to Morocco in the twentieth century [21].

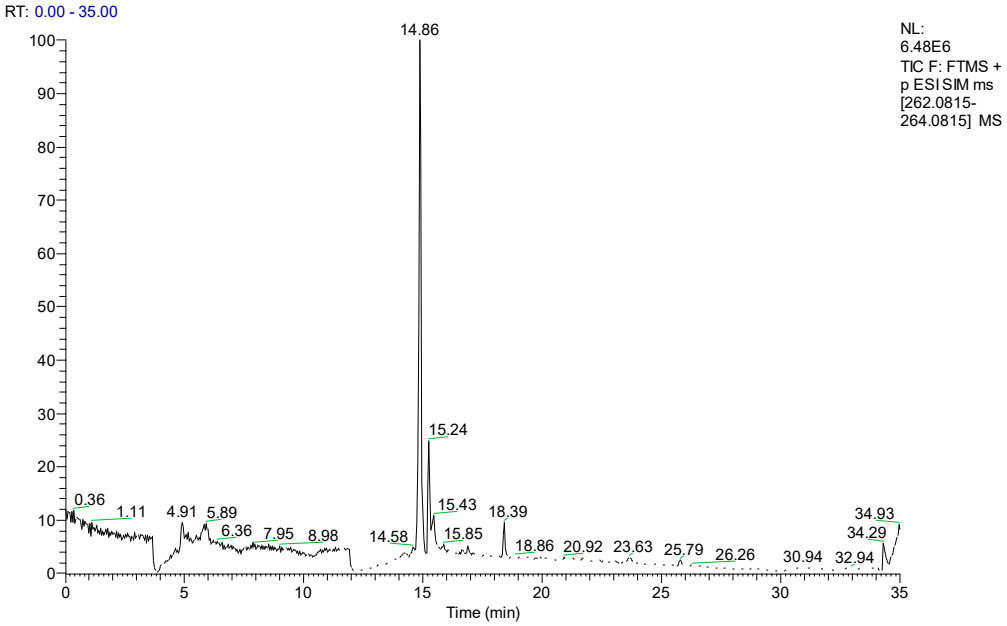

Figure 3. Single ion monitorization LC-MS chromatogram for $[M + H]^+$ m/z 263.08 signal, identified as indigotin.

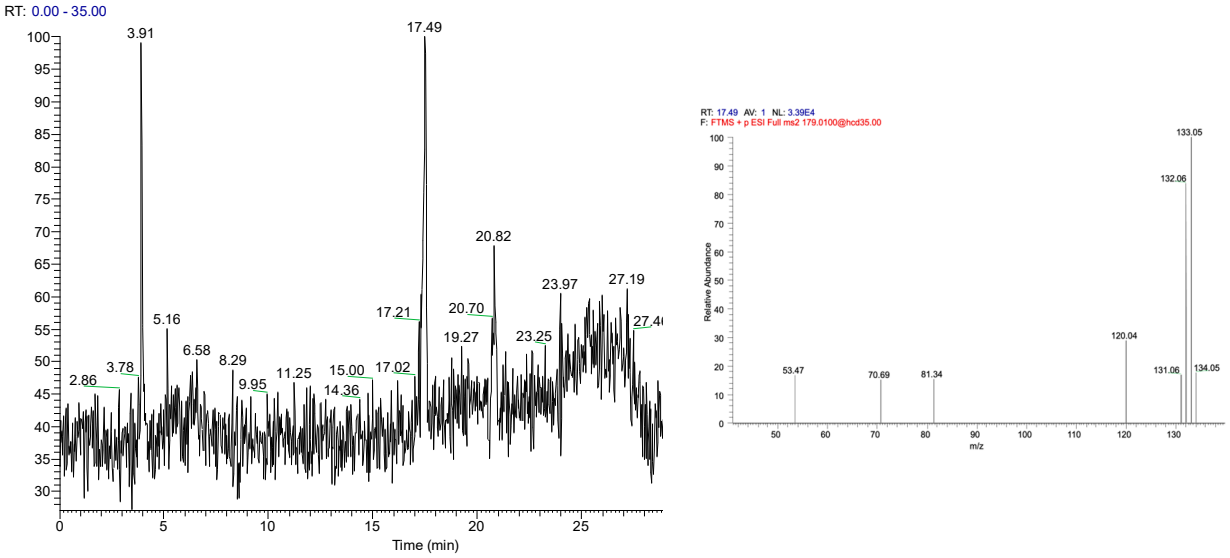

Figure 4. Single-ion monitorization LC-MS chromatogram for $[M + H]^+$ m/z 179.01 signal, identified as daphnetin, with MS2 spectrum fragmentation signal m/z 133.05.

The MS signals suggested potential animal and/or microbial contamination including D-glucosamine (D-GlcN), which is an amino sugar and a prominent precursor in the biochemical synthesis of glycosylated proteins and lipids [21]; or acetylglucosamine (GlcNAc), which is an amide derivative of the glucose and part of a biopolymer in the bacterial cell wall. GlcNAc is the monomeric unit of the polymer chitin, which forms the exoskeletons of arthropods such as insects and crustaceans [22].

HPLC-Orbitrap-MS analysis (Table S1) determined whether the materials had suffered degradation [23]. The London Museum silk sample (A27050) did not show any correspondence to 4-hydroxybenzoic acid ($[M\text{-}H]^-$ m/z 137.03) and had not, therefore, degraded to any significant degree. The Drummond Castle silk samples (DC001 and DC004) corresponded with 4-hydroxybenzoic acid ($[M\text{-}H]^-$ m/z 137.03) with MS^2 93.05, suggesting that the silk had degraded.

3.2. In-Situ Analysis: Raman and Molecular Fluorescence

Raman spectroscopy allowed the identification of indigo in the London Museum and the Drummond Castle samples. The main bands from indigo that were identified were 254 cm^{-1} (γ(C=C)$_2$), 545 cm^{-1} (δ(C=C–CO–C)), 599 cm^{-1} (δ(C=O), δ(CNHC)), and 573 cm^{-1} (ν(C=C)) (Figure 5) [22].

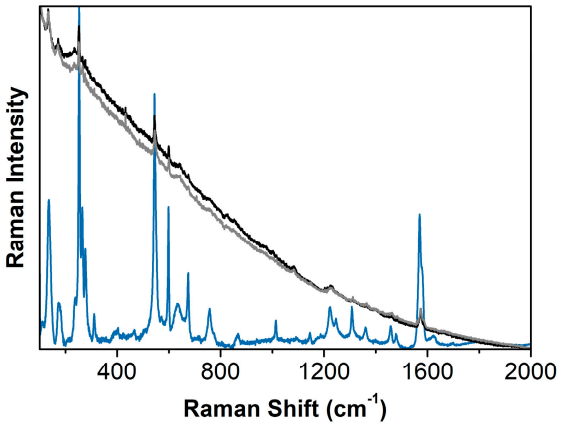

Figure 5. Raman spectra of the London Museum (black) and the Drummond Castle (grey) waistcoats, with an indigo reference in blue.

On the other hand, molecular fluorescence was used to identify the presence of a yellow dye in the Drummond Castle samples. Although microfadeometry indicated the possible use of a yellow dye in the London Museum waistcoat, the analysis using molecular fluorescence in the visible indicated only the presence of yellow silk (Figure 6). This undertone of yellow is possibly due to the degradation of the silk fabric due to its various uses and conservation conditions.

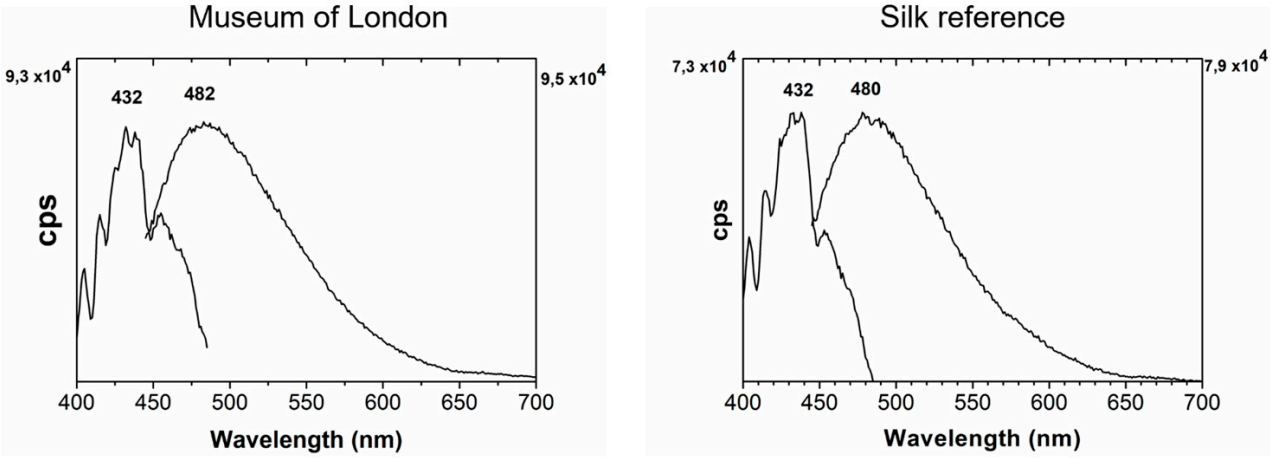

Figure 6. Molecular fluorescence spectra of London Museum waistcoat (**left**) and natural silk (**right**).

In contrast, the Drummond Castle sample indicated the presence of a yellow dye (Figure 7) due to its two excitation bands at 436 and 460 nm and emission maxima at 490 nm. Because these signals do not match those obtained from luteolin-based dyes, namely weld, it is clear that the yellow source used is different. Nevertheless, the flavonoid–aluminium references (Figure 7) have shown that these intervals are standard for benzopyran-based molecules, such as flax-leaved daphne (daphnetin). The lack of thorough references for all yellow dyes used historically throughout Europe has prevented the identification of the exact source. This emphasises the importance of building a reference database.

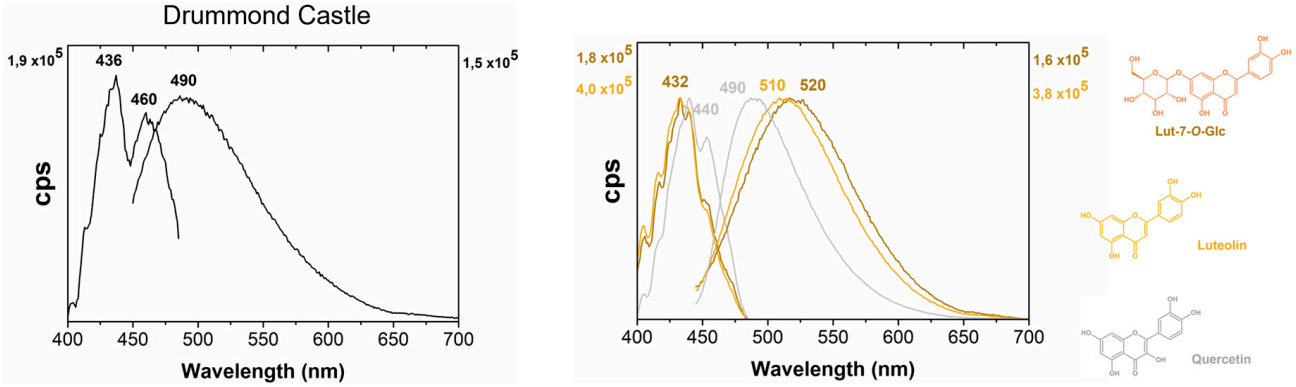

Figure 7. Molecular fluorescence spectra of the Drummond Castle waistcoat (**left**) and luteolin, luteolin-7-*O*-glucoside and quercetin, complexed with Al^{3+} (×100), in filter paper (**right**).

4. Conclusions

The dye analysis undertaken in this study indicates that the hypothesis that a blue dye alone was used to colour the London Museum silk waistcoat is proven. However, the identification of both blue and yellow dyestuffs in the Drummond Castle silk waistcoat makes green a more likely colour for it, proving hypothesis two.

Further dye analysis of knitted waistcoats of similar blue-green colours (all of which are silk with silver thread) is recommended. Examples are held at the Stiftelsen Kunstindustrimuseet, Nasjonalmuseet, Oslo, Norway (inventory numbers OK-dep-01162 and OK-08800); Bergen, Norway (inventory numbers VK4074 and BY03948); and Stålheim, Tønneberg, Norway (no inventory number). Work on green examples is also desirable. These are at Stiftelsen Kunstindustrimuseet, Nasjonalmuseet, Oslo, Norway (inventory number OK 11661) and in Trondheim, Norway (inventory number NK 747).

The development of a comparative set of data from similar garments will permit speculation on the original colour of the London Museum waistcoat and broader conclusions to be drawn about the conventional dyestuffs and colours for early modern waistcoats.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/heritage7080189/s1, Table S1: HPLC-ESI-Q-Orbitrap-MS sample dye analysis.

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Data Availability Statement: All relevant data is available on request.

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

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