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# BEYOND THE EYE-SIGHT: THE PUZZLE OF A JAPANESE MANCHIRA



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SCIENTIFIC PAPER

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**Abstract**

In the framework of a collaboration between the *Opificio delle Pietre Dure* and the Stibbert Museum (both Florence, Italy), a Japanese armour from the Edo period (XVII-XIX cent.), called *manchira*, was subjected to a multi-analytical diagnostic campaign. Due to the multi-layered structure of the object, constituted by silk, linen or hemp, wool, leather, and metallic parts, the diagnostic campaign entailed both non-invasive and micro-destructive techniques, such as multi-spectral photography, FORS, FT-IR, XRF and HPLC-DAD analysis, prior and in parallel to restoration practice. The main goals of the study were assessing the state of conservation in order to plan a thorough cleaning and consolidation strategy; characterizing the assembly and the materials employed; dating the object and clarifying the context of its production (e.g. the social status of the owner, the quality of the constituting materials, etc.). Reference *ex-novo* of dyed textiles were prepared accordingly to traditional Japanese recipes, by using materials purchased in Japan. The results were interpreted with the help of Japanese costume and textile historians and allowed us to clarify the nature of the constituting materials and to characterize the object under study. In this paper, the main results obtained by micro-destructive techniques are presented. In particular, the analysis by HPLC-DAD led to the identification of a black shade obtained by superimposing an indigoid dye to an iron-mordanted flavonoid dye (*Sophora japonica*) for the dark damask and of *murasaki* dye for the *kikkō*. The influence of the dyeing technique, entailing the application of an iron mordant, was fundamental in assessing the state of conservation of the object.

## 1 Introduction

The physical-chemical investigation of the materials used for manufacturing historical objects under restoration processes is an invaluable tool for assessing their state of preservation in order to plan a thorough cleaning and consolidation and preventive conservation strategies. By merging analytical data and information obtained by the artistic and historical research, it is often possible to assess the type and the quality (in terms of price and rarity) of the constituting materials, their provenance and the quality of the technique employed in their production and handicraft. The comparison with known dyeing recipes and handicraft manuals may yield information on the social status of its owner, and help to reconstruct the story of the use of the object. In the field of study of textiles and fabrics, the characterization of dyeing materials has proved to be fundamental to gain these goals[1,2,3]. The most common methods for identifying organic colorants in textiles are based on liquid chromatography interfaced with photodiode array and mass spectrometric detection. These micro-destructive techniques require the extraction of the chromophore-containing molecular markers with a treatment based on hydrolysis in acidic methanol, extraction with complexing agents, or organic solvents depending on the stability of the target analytes[3]. The unambiguous identification of the dye source can only be achieved by detecting molecular markers that are typical of selected dyes and comparing the chromatographic profiles of historical samples with the ones of ref-

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erence specimens. Especially when non-European dyes are involved, the reference libraries available in the literature or collected in specialised laboratories and networks are still incomplete, and identifying the original dye source is extremely challenging[4,5,6].

This paper deals with the study of peculiar characteristics of a Japanese *manchira*, which is a seldom described part of the outfit of a samurai warrior[7]. In particular, *manchiras* appear as elegant doublets, but included an armoured layer, which is suited for the protection of the upper part of the body, including the neck and arm pits. They were to be worn under or over the *dō* (the classic Japanese chest armour), and may include chains, armour plates or brigandine armoured parts. In particular, the object under study (see Figure 1) belongs to the Japanese section of the Stibbert Museum, but no further information is currently available on its provenance or dating. Due to its poor state of preservation, it is currently undergoing conservation at the *Opificio delle Pietre Dure*[8,9]. The *manchira* conservation project entailed a thoughtful diagnostic campaign, aimed at characterizing the materials employed and their assembly and assessing the state of conservation in order to plan a thorough cleaning and consolidation strategy. Moreover, the analytical data helped in dating the object and clarifying the context of its production, in order to confirm and better specify the information obtained by the study of its fashion and style, which refer to the Edo period (XVII-XIX cent.). Considering the multi-layered and multi-material structure of the *manchira*, different issues needed to be faced at the same time. The diagnostic campaign entailed the use of several techniques, and was supported by a thoughtful study of traditional techniques with the help of a Japanese costume and textile historian, and the preparation of *ex-novo* specimens to be used for comparison purposes. To obtain the complete characterisation of the *manchira*, several techniques and analytical procedures were exploited, in order to get data on both the organic and inorganic components of the materials. Non-invasive analytical techniques were exploited for a preliminary survey prior to sampling, followed by the application of micro-destructive techniques. In particular, visual inspection, false colour photography / multispectral imaging and X-ray radiography were first undertaken at the *Opificio delle Pietre Dure*. In order to characterise the dyestuffs employed, a set of non destructive analyses by Fiber Optics Reflectance Spectroscopy (FORS) was conducted. When sampling was possible, micro-destructive HPLC-DAD analysis was applied.


Figure 1: The *manchira* on its original stage[8].

This paper summarises the results obtained by non-destructive techniques[8,9] and focuses on the analytical data obtained by micro-destructive analysis of dyed yarns, with particular attention to HPLC-DAD analyses of the organic dyes.

## 2 Materials and Methods

### 2.1 FORS

The analyses were undertaken at the IFAC-CNR (Florence) using a portable Zeiss mod. MCS601 UV-VIS-NIR spectro-analyser with quartz optical fibres with a CCD detector (1024 diodes) with 0.8 nm/pixel resolution and operating in the 200-1050 nm range, equipped with a stabilized halogen lamp (20 W, mod. CLH600) with heat temperature around 3000 K. A Zeiss mod. MCS611 NIR spectrophotometer with CCD detector (512 diodes) with 6 nm/pixel resolution and operating in the 900-2250 nm range was also used. The measuring heads are two hemispheric probes designed by IFAC characterized by 2 mm acquisition spot, with probe configuration 2x45°/0° and 1x8°/0° geometries, respectively[10].

### 2.2 Optical Microskopy (OM)

All the samples (single threads or single fibrils) were placed on glass slides and observed under the optical microscope in visible and UV light (both reflected and transmitted) without any further treatment. Selected samples were also embedded in a polyester resin and observed under the microscope both in diffused and in UV light. The optical microscope used was a Zeiss Axioplan allowing magnification from 5x to 20x and equipped with different light sources such as halogen lamp for the visible and HBO mercury vapours lamp for the UV range.

### 2.3 Liquid Chromatography

HPLC-DAD analyses were performed on a PU-2089 Quaternary Gradient Pump with degasser (Jasco International Co., Japan), equipped with a Rheodyne Model 7125 injection valve and coupled to a spectrophotometric diode array detector MD-2010 (Jasco International Co., Japan) was used. The data were

processed by ChromNav® software. The chromatographic separation was performed on an analytical reverse phase C-18 column Synergi 4u Fusion-RP 80A 100 x 2 mm. The eluents were: A, water with 0.1% trifluoroacetic acid; B, acetonitrile with 0.1% trifluoroacetic acid. The programme was: 85% A, 15% B for 1 minute; then to 50% A, 50% B in 7 minutes; then to 10% A, 90% B in 5 minutes and hold for 10 minutes. The flow rate was 0.3 mL/min. The detection wavelength was set at 275 nm and the acquisition of DAD spectra was achieved in the range 200-650 nm, 4 nm steps. The injection volume was 2 µL.

### 2.4 Reference Materials

*Ex-novo* reference specimens were prepared in order to obtain suitable comparison for indigo and *murasaki* dyed textiles. In detail, two recipes, kindly provided by "Yoshioka Textiles" (Kyoto) and dated back to Edo period (XVII cent.), were followed for dyeing in indigo and in *murasaki* (purple) colours.

**Indigo (藍) dyeing from indigo plant (*Polygonum tinctorum*)**

Ingredients: 23 L of water (soft water); 1 Kg of fermented indigo leaves (*sukumo*), obtained after drying from the *Tade-ai* plant (*Polygonum tinctorum* L.); 700 g of oak ashes (*kungi-moku-ai*, *Quercus acutissima*); 100 g of bran (*husuma*); 20 g of calcium hydroxide ($Ca(OH)_2$); 25 x 50 cm piece of linen fabric.

Recipe: Preparation of alkaline solutions of oak ashes

Mix the ashes with 7 L of boiling water. Let the solution rest for 2 days in order to allow the leftover to precipitate, then filter to obtain alkaline solution #1. Mix the ashes with 14 L of boiling water and let the solution rest for 3 days. Filter to obtain alkaline solution #2, then repeat the procedure with 21 L of water to obtain alkaline solution #3.

Preparation of indigo dye bath

First day: mix 1 Kg of plant material with 5 L of solution #2. Mix with a bamboo stick, keeping the temperature of the solution around 20 °C.

Second day: add 5 L of solution #3 to the dye bath and left it rest 7-10 days, stirring twice a day until a film layer will form upon the solution.

$7^{th}$-$10^{th}$ day: add to the dye bath a solution of 100 g of bran in 2 L of water previously prepared by heating the mixture for 20-30 minutes and then left cooling down. The bran will catalyse the indigo fermentation.

$9^{th}$-$12^{th}$ day: add 5 L of solution #1 to the dye bath and let it rest for one day. Afterwards, the dyeing procedure may start (the pH of the dye bath should be in the range of 11-12, and can be adjusted by adding proper quantities of $Ca(OH)_2$).

Dyeing:

Rinse the fabric in hot water (40-50 °C), then dip it in the dye bath for 5 minutes at room temperature, stirring. The fabric is then pulled out of the dye bath: the dye readily oxidises in the air. The fabric is then rinsed in cool, clean water to remove the excess of the dye. The dyed fabric is then left drying in a cool, dark place. In order to obtain a darker shade of blue, the whole procedure may be repeated.

**Murasaki (紫) dyeing**

Ingredients: 10 L of water (soft water); 400 g of *murasaki* (*shikon*, roots of *Lithospermum purpurocaeruleum*); 40 g of camellia ashes (*tsubaki*, *Camelia japonica*); 100 g of textile.

Recipe: Preparation of the ash solution and of the fabric

Admix 40 g of ashes with 2 L of boiling water, and let the ashes precipitate for 2 days. Then filter. Rinse the fabric in hot water (40-50 °C).

Extraction of the dyestuff

Wash 200 g of *shikon* and then soak the root chips in hot water (50 °C). Then, smash the roots in a wooden mortar and put them in a linen bag. The dye is extracted in 3 L of hot water (40-50 °C). The final solution is filtered and stored. The roots are again smashed and subjected to hot water extraction two more times, and the obtained solutions are admixed with the first one and diluted to obtain 9 L of the final dye bath.

Dyeing

Dip the textile into the obtained dye bath and let it rest for 1 hour. Then slowly heat the dye bath up to 40 °C and stir. Rinse the dyed textile in cool water to remove the excess of the dye. Add 200 mL of the ashes solution in the dye bath, and slowly heat it up to 40 °C. Then, dip the fabric in the dye bath again and stir for 20 minutes. Rinse again the fabric in fresh water. Repeat the whole procedure twice (including the addition of ashes to the dye bath). Rinse the fabric and let it dry in a dark cool place. Repeat the procedure to obtain darker shades, if needed.

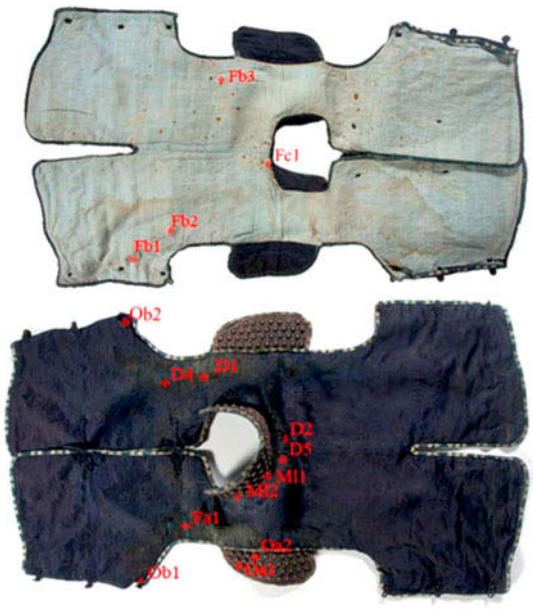


Figure 2: Sampling points for micro-destructive analyses.

| Sample name and description | | sample size (mg) | |
|---|---|---|---|
| Top layer (silk damask): warp, double thrown silk, blue | D2 | < 0.1 mg |  |
| Top layer (silk damask): weft, double thrown silk, dark blue | D3, D5 | < 0.1 mg, 0.3 mg |  |
| Top lining: weft, double thrown cellulose fibre, blue | Fa1 | 0.5 mg |  |
| Inner lining: linen, pale blue | Fb1 | < 0.1 mg |  |
| Braid for bows and buttons: double thrown silk, blue | Ob2 | <0.1 mg |  |
| Background for shoulders and collar: blue wool | Ml1 | < 0.1 mg |  |
| Braid upon the *kikkō*: double thrown silk, purple Oa1 0.6 mg | Oa1 | 0.6 mg |  |

Table 1: Description of samples collected for micro-destructive analyses.

### 2.5 Description of historical samples and sample treatment

The historical samples were collected from the areas highlighted in Figure 2 and described in Table 1. They were treated following a two step procedure, in order to allow for recovering both labile and mordant dyes[11]. The first step entailed a solvent extraction with dimethylsolfoxide (400 µL DMSO) in an ultrasonic bath at 60 °C for 1 hour. The second step entailed the addition of 400 µL of $H_2O$/MeOH/HCl solution (1:1:2) to the residue; the extraction took place at 60 °C for 1 hour in an ultrasonic bath. The extracts were admixed, filtered, evaporated under a gentle stream of $N_2$, and finally reconstituted in 20 µL of DMSO.

## 3 Results and discussion

The preliminary results obtained by applying non destructive techniques allowed for assessing the stratigraphy of the *manchira* and identifying the five layers used to build up its structure, as depicted in Figure 3[8].

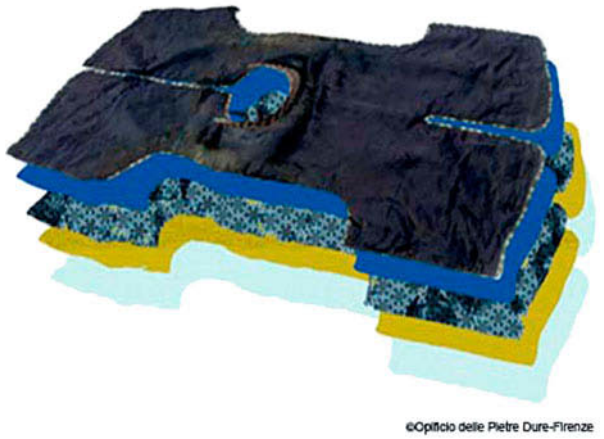


Figure 3: Assembly of the *manchira* on the basis of the results obtained by non-destructive techniques[8].

The first layer from the inside is made up of a light blue linen fabric; the second one is an interlining made of undyed hemp. The typology of fibers was identified on the basis of the SEM observation and on the evaluation of their average diameter[9,12]. For the first layer, the average diameter of the fibers was 12.97 µm (compatible with linen, whose diameter ranges from 10 to 40 µm); for the second layer, the average diameter of the fibers was 34.54 µm (compatible with hemp, whose diameter ranges from 15 to 50 µm)[12]. The third layer, which is not detectable by visual inspection of the object, was previously characterized by X-ray radiography[8] and consists in a metal armour formed by small iron (possibly steel) rings linked with hexagonal plates of treated leather and probably covered in lacquer. The fourth layer, which is visible under the thorns on the surface, is made of a cellulose fibre and is dyed in a dark blue shade. The top layer consists in a silk damask. The bottom of the collar and straps are lined in wool, while the braids are made of blue dyed silk. The whole structure is assembled by a beautiful, dyed leather border.

Once the assembly of the *manchira* and the source of the fibres were characterised, open questions still remained about the dyestuffs employed to yield the dark colour of the outer layer and the purple and green details of the collar and straps. In particular, the study aimed at understanding the reason for the poor state of conservation of the dark damask for restoration purposes, and at identifying the nature of the purple colour on the collar and straps, which may yield information on the recipes used for dyeing, and thus on the dating and price of the object. The results obtained by the inspection under the optical microscope, the study of cross sections of the threads embedded in suitable resins under UV and visible light and the interpretation of HPLC-DAD chromatograms will be discussed in the following paragraphs depending on the different investigated areas of the *manchira*.

### 3.1 The damask (top layer)

The OM observation of the warp showed a bad conservation condition of the thread (see Figure 4 B), which appeared crystalline and coarse (see Figures 4 C, D and E), and the blue colour appeared evenly dis-

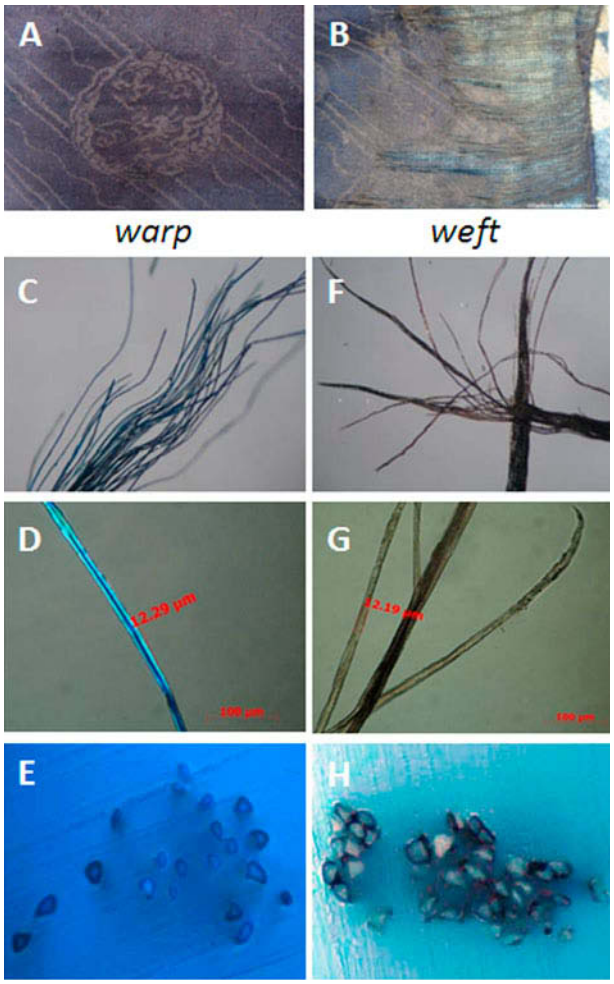


Figure 4: Pattern of the silk damask (A) and degraded area on the right shoulder (B). Images under the stereomicroscope of the warp (C) and the weft (F), Vis light; D, G: longitudinal sections of the warp and weft, respectively, acquired under the optical microscope in transmission, 20x; E, H: cross sections of the warp and the weft, respectively, acquired under the optical microscope, UV light, 50x.

tributed on the surface of the threads. The OM observation of the weft showed an advanced degradation of the fibrils, which appeared crystallized and characterized by micro-fractures (see Figures 4 F, G and H ). The dark colour appeared oddly distributed, and the surface was covered by a significant amount of particulate[9].

Two samples were collected from the weft and the warp of the silk damask, and were subjected to HPLC-DAD analysis. The chromatograms obtained from the two extracts gave different results: for the warp, indigotin was the detected dyeing molecule (Figure 5), and the chromatographic profile matched with the one obtained by analysing the indigo dyed *ex-novo*. In the chromatogram obtained for the weft, quercetin-glycosides were detected along with indigotin (Figure 6).

We may conclude that the warp was dyed with an indigoid plant, such as *Indigofera tinctoria* or *Polygonum tinctorium*, also used for the preparation of the *ex-novo*[13,14,15]. The presence of quercetin-glycosides suggested the presence of a flavonoid dye, such as *Sophora japonica*, a yellow Japanese dye[13,16]. It is interesting to note that SEM analyses[9] showed the presence of a relevant amount of iron in the weft threads (data not shown). These analytical data, merged with the study of Japanese historical recipes used for dyeing in black, suggest the hypothesis that the weft was double dyed, with an indigoid vat dye an then with *Sophora japonica* on an iron mordant. In particular, such recipe is mentioned for obtaining black shades for precious textiles, reserved for the Emperor and his court[17,18,19]. Moreover, the presence of iron as a mordant may explain the bad state of conservation of the dark damask, with respect to the other threads in the *manchira*[13,21].

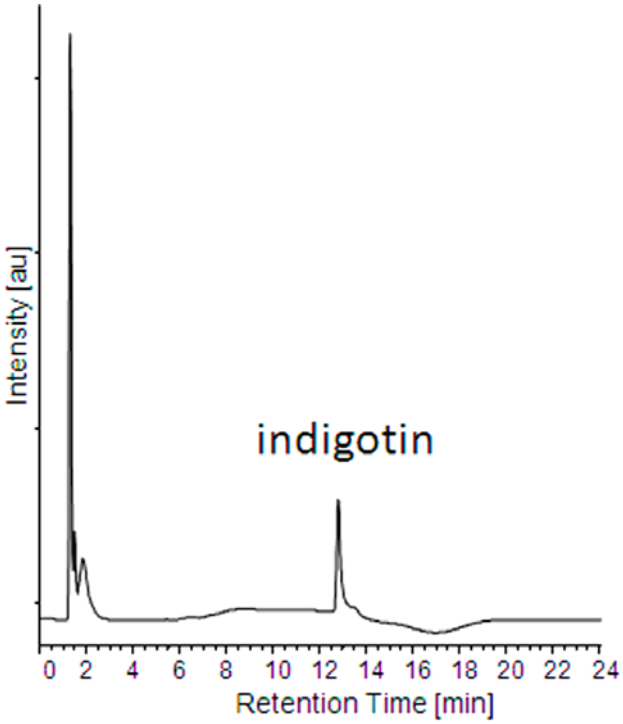


Figure 5: HPLC-DAD chromatogram of the DMSO extract of the warp of the silk damask, at 620 nm (sample D2).

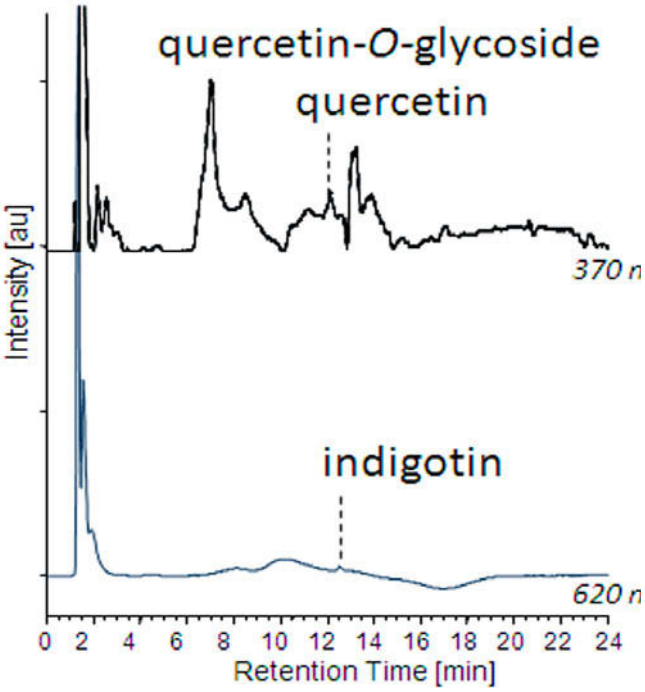


Figure 6: HPLC-DAD chromatograms of the extracts of the weft of the silk damask (sample D5): DMSO extract, at 620 nm (blue line) and extract after mild methanolysis, at 370 nm (black line).

### 3.2 The blue linings and the blue braids

Two samples from the inner pale blue lining and the top blue lining (Fa1 and Fb1, respectively) were analyzed by HPLC-DAD and, as expected, revealed the presence of indigotin, thus supporting the hypothesis that they were dyed with indigo. One sample collected from one blue braid (Ob2) was also analyzed by HPLC-DAD and revealed the presence of indigotin, thus suggesting also in this case the presence of an indigo dye.

### 3.3 The kikkō

The analysis of the *kikkō* entailed the characterization of the green watch chain along the hexagonal borders, of the purple braids in their centre, and of the background wool. Due to the excellent state of conservation of the green threads, no sample was taken from the watch chain, and only OM observation and FORS analyses were undertaken. It was possible to sample some fibres from the purplish silk in the centre of the hexagons and from the blue wool, and to observe them under the OM (see Figures 7 C, D and E for the purple silk, and F, G and H for the blue wool). The state of conservation of the sample threads appeared generally poor, with wide crystalline areas and diffuse particulate. A small sample of the blue wool constituting the background of the fabric on the shoulders and collar (Ml1) was also collected.

The results of FORS analysis applied to the green yarn are shown in Figure 8 A, and the reflectance minima are listed in Table 2. The comparison between the acquired spectrum and the database of the reference spectra allowed us to reference silk threads dyed with safflower yellow and indigo (see Figure 8 A). The application of complimentary non destructive techniques, such as multispectral imaging, are in progress in order to confirm this hypothesis[20].

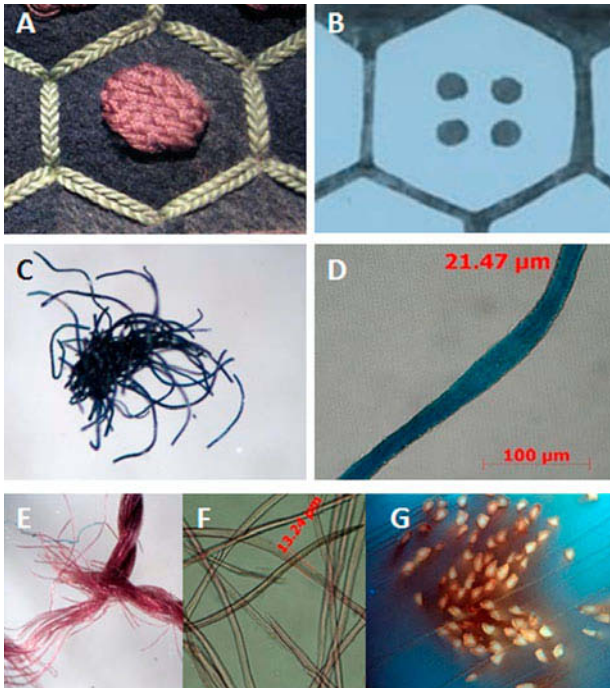


Figure 7: A: one of the *kikkō*; B: X-Ray radiography of one of the *kikkō*; C: sample collected from the blue wool (Ml1); D: longitudinal section: optical microscope, transmission, 20x; E: sample collected from the purplish area of the *kikkō* (Oa1); F: longitudinal section: optical microscope, transmission, 20x; G: cross section: optical microscope, UV, 50x.

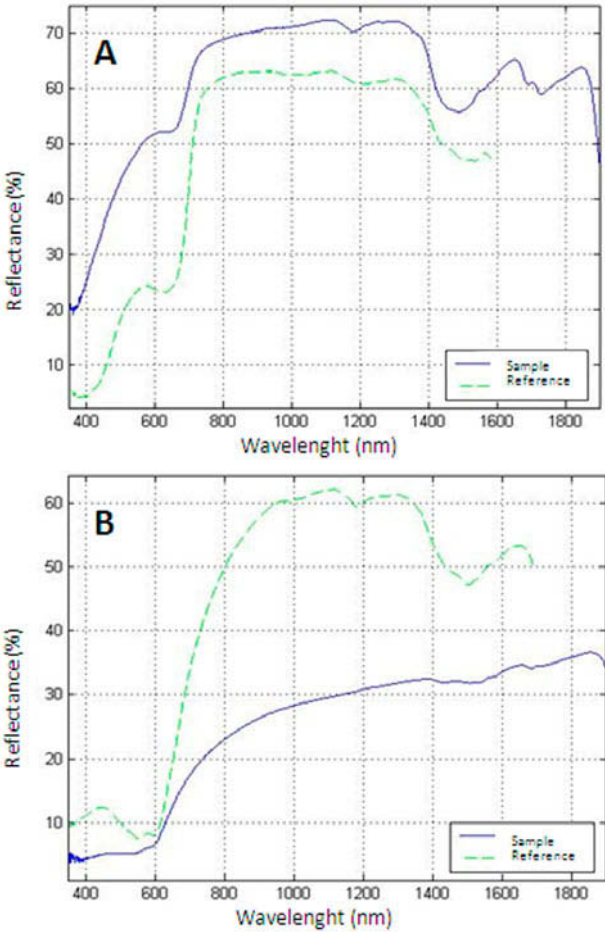


Figure 8: A: FORS analysis of the green border of the *kikkō* (blue line), compared to the one obtained by the analysis of a reference silk thread dyed with indigo + yellow safflower (green dotted line); B: FORS analysis of the purplish inner part of the *kikkō* (blue line), compared to the one obtained by the analysis of a reference silk thread dyed with alkanet (green dotted line).

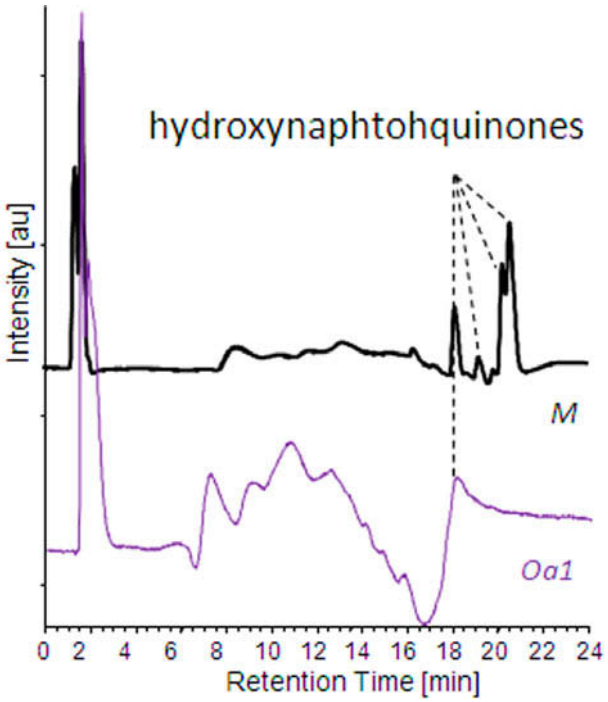


Figure 9: HPLC-DAD chromatograms at 490 nm of the DMSO extract of the purplish thread in the *kikkō* (sample Oa1) and of the DMSO extract of the *ex-novo* fabric dyed with *murasaki* (*M*).

| Sample | % $R_{min}$ (nm) |
|---|---|
| Green border of the kikko | 650, 1175, 1500, 1680, 1750 |
| Reference yarn dyed with indigo + yellow safflower | 640, 1180, 1500 |
| Purplish inner part of the kikko | 550, 595, 1440, 1540 |
| Reference yarn dyed with alkanna | 555, 1180, 1500 |

Table 2: Reflectance (%) minima highlighted in the FORS spectra shown in Figure 8 A and B.

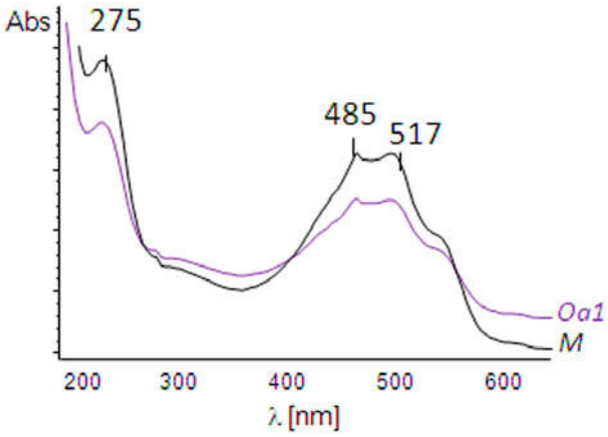


Figure 10: DAD spectra obtained for the peaks at 18 minutes for the chromatograms shown in Figure 9, assigned to hydroxy-naphthoquinones.

With regard to the results obtained by FORS on the purplish braid (see Figure 8 B and reflectance minima listed in Table 2), the database search did not yield relevant results, except for a match with a reference fabric dyed with alkanet (*Alkanna tinctoria* L.), which is not of Japanese origin. It is worth mentioning that the principal component in alkanet, alkannin, is an enantiomer of shikonnin, the principal component in gromwell (*Lithospermum erythrorhizon* Sieb. & Zucc.) or *murasaki* (obtained from *Lithospermum purpurocaeruleum*), which are present in Japan[13].

HPLC-DAD analysis was then undertaken in order to fully characterize the colouring materials of the purple silk and the blue wool. The analysis of sample Ml1 allowed us to detect indigotin, thus suggesting the use of an indigoid dye source. The HPLC-DAD chromatogram obtained by the analysis of the purple silk is shown in Figure 9 (purple line, *Oa1*). The chromatogram highlighted the presence of molecules whose UV-Vis spectrum matches with the one of alkannin and shikonnin (see Figure 10), which, as already mentioned, are two optical isomeric hydroxy-naphthoquinones responsible for the purple colour of alkanet and gromwell (*Lithospermum erythrorhizon* Sieb. & Zucc.) or *murasaki* (obtained from *Lithospermum purpurocaeruleum*), respectively[13,14]. The analytical results were compared with the ones obtained by analyzing reference specimens dyed with alkanet and *murasaki* (Figure 9, black line, *M*) and subjected to the same analytical procedure. Interestingly, the study of ancient recipes from Nara period (710-794 A.D.) suggested *murasaki* as a largely employed colouring material to obtain purple shades for highly valuable fabrics[18], thus confirming its possible use in the *manchira* under study.

## 4 Conclusions

The use of the combined analytical approach allowed us to assess the macro and micro state of preservation of the *manchira*, to identify the stratigraphy of the object and to characterize the materials and in particular to identify the dyestuffs used in its production. The non-destructive analytical techniques allowed for identifying the technology used in the assembling the *manchira*, and to highlight the main conservation issues.

The use of micro-destructive techniques allowed the restorers to highlight the peculiarities of the single threads, by identifying their structure and classify the provenance of the silk and linen threads. The analysis by HPLC-DAD proved fundamental in assessing the nature of the employed dyestuffs, leading to the identification of the used recipes and thus to the context of the production of the object. In particular, the identification of a double dyeing obtained by superimposing an indigoid dye to an iron-mordanted flavonoid dye (*Sophora japonica*) for the dark damask and of *murasaki* dye for the *kikkō* was crucial for linking the context of production of the *manchira* to high class purchasers[18]. The analytical results allowed to confirm the hypothesis based on the study of the iconography and the fashion of the *manchira* performed with the aid of a Japanese historian, and to date the production of the *manchira* to the first part of the XVII century[22].

Moreover, the identification of the dyestuff used in the black threads allowed the restorers to understand the reason for their poor conservation state[21], thus helping in planning their cleaning, consolidation and preventive conservation.

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