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TEXTILE MATERIALS: REFERENCE STANDARDS FOR CHARACTERIZATION USING MULTISPECTRAL ANALYSES

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ABSTRACT

The main objective of the research carried out by the Textile Conservation Sector at the Opificio delle Pietre Dure in Florence, Italy, was to verify the actual potential of optical investigation as means for identifying dyes on ancient textiles, and thus, to avoid taking micro-samples. For this we used samples of new fabrics dyed following procedures described in a number of fifteenth-century documents (es. PLICTO), and we systematically studied their different responses in electromagnetic radiation spectral bands. Soon, additional samples will be integrated into the small group of samples dyed in the 1980s, thereby expanding our database, which is still in its initial phases. The first group of samples consists primarily in silk and wool fabrics mounted on non-acid card stock. The multispectral techniques employed in the research are: visible light (VIS), Infrared reflectography (circa 950nm) imaged in false-colour infrared, UV fluorescence and reflected UV, also imaged in false-colour UV. All the images obtained with these methods were calibrated according to known reflectance standards and using reference colour profiles. The results obtained for the various samples proved to be quite interesting and provide the stimulus to define a more articulated research project in the future. Multispectral imaging techniques were supported with FORS (Fibre Optics Reflectance Spectra), which proved to be a complementary verification technique. The research technique was developed so that it could be used in the future, in a cost-contained environment and employing correct methodologies, by all laboratories and so that results could be compared with existing databases. Further, it has already been widely demonstrated, especially in the field of painting conservation, [1] that this type of diagnostic can provide important information for scientific and art historical documentation.

OBJECTIVES OF THE RESEARCH

The scientific examination of the materials in a work of art, both constituent and those resulting from past restoration interventions, their interactions and behaviour over time, is an essential element in the conservation of an object, and is an indispensable prelude to every restoration treatment. With increasing frequency, scientific innovations are being employed in the field of textile conservation for studying execution techniques and for the analysis of the conditions of an object. Textile materials, often fragile and except for a few cases not traditionally investigated, lend themselves to non-invasive investigation techniques rather than micro-destructive techniques. We are faced with various elements when we work to extract two essential pieces of data that characterize a textile piece (or any other object): dating and place of manufacture. These elements have already been addressed by CIETA in Lyon, France, during the 1950s. [2] They must evaluate the width, the selvages, the design repeat, the placement of the decorative elements and their relationship with the weave, the different types of spun yarns and their function within the weave, the material each one is made from, the twist method and the type of substance used in the dyeing. Without resorting to invasive analyses such as C14, it is clear that numerous pieces of information are required before being able to date a textile piece and to hypothesize a possible provenance. Here, we will address the dyed yarns that make up the textile, often analysed using HLPC, a micro-

destructive technique requiring at least $\geq$ 0.2 mg of material. In undertaking a study as complex as dyes and dyeing, specific and comparative analyses are fundamentally important in supporting hypotheses that would otherwise be only plausible. Any conjecture made during the observational stage of an object remains simply conjecture until it is supported or refuted by rigorous scientific answers.

Under this premise, the laboratories at the Opificio delle Pietre Dure carried out investigations on various textile artworks using multispectral imaging. The initial results of these investigations were the main stimuli for further study employing rigorous scientific methods. The first objective of this work is to identify dyestuffs using only optical analytical methods to verify the actual potentiality of the methods. These investigations were as limited as possible, because even though they are defined as non-invasive, they might be considered damaging in a more in-depth, future analysis. Physical analysis allows us to acquire an extensive understanding of the work and obtain a plotting of the different materials, which could also be a support element in better identifying eventual future areas for individual sampling. This approach requires cross analyses in order to be able to verify the results. The acquisition of images using multispectral methods, precisely calibrated with a computerized data management, greatly facilitates and has many advantages in the creation of a database. Among these advantages are the ease in disseminating information, the inalterability of the information, immediate comparison with multiple results, and the possibility of repeating the investigation in time, thereby facilitating ageing studies and monitoring of the conservation conditions of the materials. The behaviour of the materials under investigation can, in many cases, also bring about the identification of organic components, their possible compositions and differentiation of original materials from those added during the object's lifetime.

REFERENCE STANDARDS AND FORMULATION CRITERIA

In order to meet our objectives, we obtained new yarns that we dyed using methods described in a number of documentary sources on ancient textiles from the fifteenth century (es. Plichto). [3] Our aim was to systematically study the interaction of the electromagnetic waves in the visible and the near-ultraviolet spectra. The dyed samples were the result of the research and experimentation for a 1984 diploma thesis at the School of Higher Education at the OPD. [4] This small nucleus of samples will soon be integrated into a broader range of samples that will permit this initial databank to be expanded. In the research carried out in the 1980s, the choice of materials was limited to protein-based yarns (wool and silk), mounted on non-acid conservation cards and a few natural vegetable and animal dyes fairly easily obtained in Italy, assuring us of provenance and preparation standards. In mapping out the route to take in order to create a scientific base reliable for our research, we must also take note of possible obstacles that may be intrinsic to the materials themselves, in addition to external factors that can disrupt our progression. When considering **ancient materials** such as wool and silk (organic materials) we must also take into account the ancient techniques and treatments for their preparation, from cleaning to spinning, finishing and dyeing, which can include washes in lye-ash soaps or boiling agents, sulphur fumigation, mineral or vegetal weighting agents, [5] natural colorants where the mordant can often alter the beginning dye colour. Dyes are subdivided according to the dyeing method into three main groups:

- direct or substantive dyestuffs such as saffron, turmeric and safflower (dyer's thistle);
- mordant dyes such as madder, kermes, cochineal (Mexican, Armenian, Polish), safflower, weld, turmeric, and orchil;
- vat dyes such as woad, indigo and Tyrian purple.

In addition to these elements, there are also properties unrelated to processing procedures that are intrinsic to each fibre (ageing, colour fading, photo-deterioration, atmospheric pollutants), as well as extrinsic factors (surface dirt, spotting). In terms of **new materials**, industrial processing procedures often make use of surfactants, finishing products, size and bleaches, visual whiteners and other substances often affecting the various operations on textiles due to their processing and finishing. Even if some of these substances can be easily removed from the textile by washing in water and neutral surfactants or by using enzymes, the use of reference samples must be carefully evaluated. It should be remembered that even the RH (relative humidity) parameter can be meaningful in colorimetric evaluation (CIELab) when there are substantial variations with respect to the normal standards for conservation due to the hygroscopic nature of textile materials.

EXPERIMENTATION: IMAGE RECORDING EQUIPMENT AND METHODS

The multispectral investigations carried out on textile samples and on the case study specimens were characterized by a bandwidth range of 360 nm to 1000 nm. The images were recorded using a Hasselblad 500 CM camera equipped with a Leaf Aptus 65s digital back, one shot, 16 bits, with a 28 MP resolution and a CCD size of 44mm x 33 mm. This type of hardware was chosen because it was compatible with the need to use a commercially available back with a broad spectral range. A 80 mm Hasselblad lens was chosen for its low absorption in the ultraviolet range despite the multicoating layers on the lenses. The CCD sensor was modified by removing the athermic filter in order to reach a sensitivity up to circa 1000 nm.

The images were recorded in various spectral bands, from ultraviolet to near-infrared, using the following methods:

- Reflected UV: two black lights with a maximum emission peak at 360 nm. The bandwidth was selected using a Kodak 18A filter. This type of emission recording does not require complete darkness as for UV fluorescence, but when working in ambient light an athermic filter must be used on the CCD in order to block the IR because the Kodak 18A presents a transparency band in the near-infrared zone.
- UV fluorescence: the emission recordings were made using a pair of black lights equipped with Schott DUG 11 UV excitation filters, using a KV 418 Schott as cut-off filter. The images recorded in fluorescence required, obviously, total darkness.
- Visible: tungsten lamps with an athermic filter on the CCD were used.
- Near-infrared: again, two tungsten lamps were used with a Kodak Wratten No. 87 filter.
- False-colour ultraviolet: this was obtained by combining the images recorded in the visible and in the reflected ultraviolet spectra. In this case, the RGB electronic image was obtained by selecting the green and blue component in the visible spectrum for the R and G channels respectively, with the reflected UV in channel B.
- False-colour infrared: this was obtained by combining the images recorded in the visible and the near-infrared spectra. In this case, the RGB electronic image was obtained by selecting the infrared component for the R channel, and the red and green components in the visible spectrum for the G and B channels respectively.
- Image calibration: calibration is the crucial point when creating a database and in obtaining repeatable and reliable numeric data that will serve as comparison for the different images acquired using the same technique. For this phase, samples of Spectralon® with reflectance percentages in the utilized range (from 360 nm to 1000 nm) were used.

During the preliminary phase of the image recordings a chromatic calibration was done and the gamma curve for correct contrast in the images was determined. For this operation eight

Spectralon® samples were used with reflectance factors of 2%, 5%, 10%, 20%, 40%, 60%, 80%, 99%.

Reflectances for the reference standards were measured with a spectrophotometer and the Lab values were obtained. These parameters proved to be fundamental in the comparisons between the various images using the Adode Photoshop CS3 program.

During the first phase, multispectral recordings were made on specifically prepared samples using pure and mixed colours on wool and silk to create a multispectral database that will allow for comparisons of the image recordings on real case specimens. The comparisons with the six multispectral images made on each sample provide interesting results because they permit broad discrimination among the different dyes and identification of the dyestuffs that were used. The results of these observations were also compared with the data obtained on the same samples using the FORS technique (Fiber Optics Reflectance Spectra). The comparison between only the a* and b* chromatic components, excluding the L component indicator of the intensity of the sample, allowed us to identify the same dye used in textiles with different saturations, which can be attributed to dyebaths having different concentrations.

CASE STUDY

Type: Religious vestment, chasuble [6]; Measurements: cm 135 x 76; Subject: relic of Saint Firmus; Technique: cut voided red velvet for the main body of the piece; Date: 15th century; Production: Florence, Venice?; Embroidery: fine red silk cloth with couched gold embroidery and various types of embroidery stitches in polychrome silk thread backed by natural linen fabric (cross and column); Date: 14th – 15th century, Material: linen for the lining and the backing of the embroidery, red silk for the velvet, polychrome silk thread and gold thread; Provenance: Church of S.S. Fermo e Rustico, Verona, Italy.

Chasuble front

Chasuble back

The chasuble was uncovered with the relics on the high altar of the church of S.S. Fermo e Rustico on 15 July 2005 on the occasion of the 17th Centennial of the Saints' martyrdom (304-2004). It was discovered underneath a lead box housing the wooden box with the bones of the two titular martyrs. The Renaissance chasuble is made of figured velvet, with an embroidered

area centred on the front depicting the Madonna and Child with Saints (Firmus and Rusticus?), which retains reminiscences of the late-Gothic according to Frattaroli [7]. The back of the chasuble has a strip of red taffeta bearing a full-figure of the crucified Christ along with circular and poly-lobed motifs worked in gold thread and polychrome silk threads. The cross is worked in gold threads directly on the ground taffeta. Towards the lower portion of the central column on the back there is an embroidered coat-of-arms, worked in polychrome silk thread. The attribution of the arms is still under study.

Fig. 1 Chasuble front: position of the points chosen for readings on the dyes.

IDENTIFIED DYESTUFFS		
	FORS TECHIQUE	MULTISPECTRAL ANALYSES
Point 1	COCHINEAL	COCHIINEAL
Point 2	INDIGO	INDIGO
Point 3	INDIGO + SAFFLOWER	INDIGO + SAFFLOWER

Table of the sample points and zones, and the dyestuffs identified using the FORS technique and using multispectral analysis.

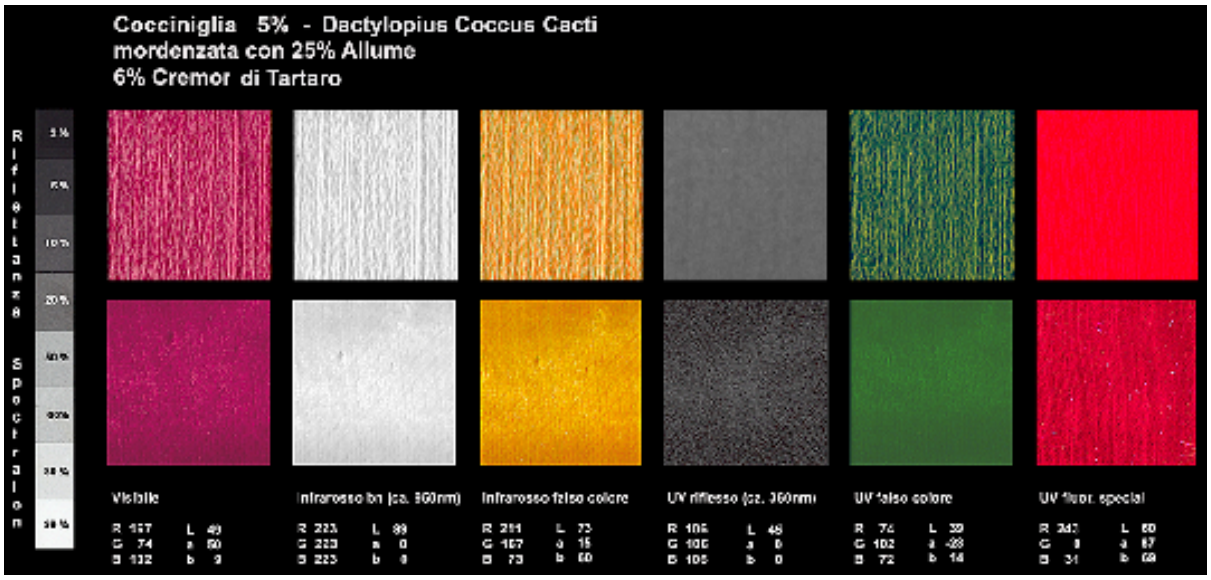

Fig. 2 Multispectral recordings for the cochineal reference and zone 1 (visible, BN infrared, false-colour infrared, reflected UV, false-colour UV, UV fluorescence)

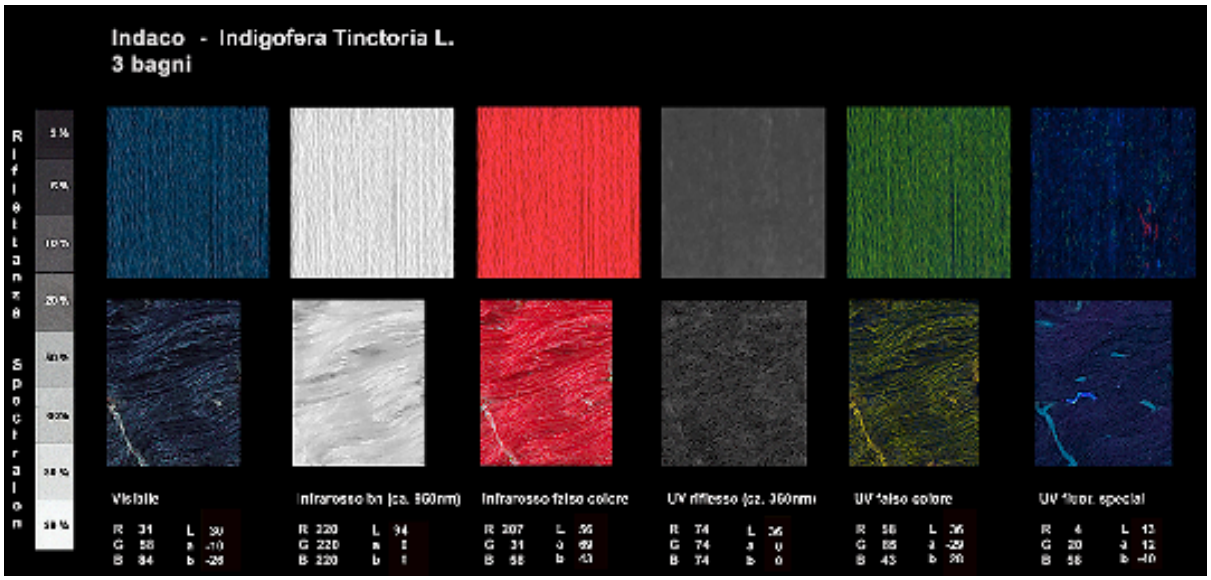

Fig. 3 Multispectral recordings for the indigo or woad reference and zone 2 (visible, BN infrared, false-colour infrared, reflected UV, false-colour UV, UV fluorescence)

Fig. 4 Multispectral recordings for the safflower + indigo reference and zone 3 (visible, BN infrared, false-colour infrared, reflected UV, false-colour UV, UV fluorescence)

CONSIDERATIONS REGARDING READING THE MULTISPECTRAL IMAGES

The readings obtained form the multispectral images [8] and the FORS (Fiber Optics Reflectance Spectra) [9] indicate the presence of some specific dyes (point 1, cochineal; point 2 indigo or woad; point 3 safflower + indigo or woad). When these dyestuffs are placed in geographical and historical coordinates of their origin they provide useful data, and combined with data some from documented commercial trade, help to better understand how they were used for the chasuble. We do know that for the **colour blue** (vat dyes) woad (Isatis tinctoria L.) was used. This dye is obtained from a biennial, and sometimes perennial, plant belonging to the Cruciferae family, common to all of Europe, including frigid zones. The main dye is extracted from the leaves, and is the same as indigo. Dyeing with woad has ancient origins and we know that during the Middle Ages (Twelfth century) it was cultivated and prepared for dyeing even in Spain. In the Fifteenth century, there is an increase in the sources, tied mainly to restriction or guidelines for dyeing in the large centres (among these Lucca and Florence). In Italy, woad was grown mainly in Umbria (Nocera and Gualdo), from which the latter takes its name. Interest in using woad wanes as a result of the European importation of large quantities of indigo from India and America. Up until the mid-1800s, indigo was used as the basis of tincture of indigo, called ‘piede’ in Italian. Indigo is thought to be one of the most ancient and widely-used dyes in the world. Indigofera tinctoria L. is the most important species among the indigo dyestuff plants and it is native to India where it has been used from earliest times (‘Veda’ documents from Fifteenth to Fifth centuries BC) [10] The plant belongs to the Fabiaceae family and is a tropical plant. The dyestuff is extracted from the leaves of the plant. The first references to indigo as a dyestuff in the Western world are in the 1305 Capitolare dei tintori (Dyers Capitularies) from Venice. These date from only a few years before Marco Polo described the method for preparing indigo and its tincture, which he had observed in India. Through the Middle Ages, its introduction into Europe was impeded in order to protect, it is said, the woad production, which was an important aspect in the economics of many countries. Indigo did not encounter any obstacles in Venice, perhaps due to the fact that the city did not have agricultural difficulties, and the Venetian dyers readily used it. It is quite difficult, however, to establish which of the two plants was used to dye the embroidery threads. We must keep in mind that even though the indigotin molecule is the

main dyestuff found in both Indigofera tinctoria and Isiatis tinctoria, and thus theoretically the same, the concentration of dyestuff in the Indigofera tinctoria is ten times greater.

For the **colour yellow** (direct or substantive dyes), safflower, Carthamus tinctorius, a spice used also as a dye, has been long-known even the most remote areas of the orient and Central Asia (Forbes 1964). It is also cultivated in northern Africa and southern Europe, and in Italy it is found mostly in the Romagna region. It is used principally for dyeing red, but in Indonesia it is used to dye both yellow and red. It should be pointed out that the main dyestuff, carthamin, a scarlet red colour, is insoluble in water, while the yellows from safflower, the other two pigments found in the safflower florets, are water soluble. Thus, when dyeing both wool and silk yellow using safflower, it is necessary to use a mordant, specifically alum (Schweppe 1992). When using safflower for dyeing red no mordants are used, even though there have been some reported cases when alum was used to dye silk red. In our case, further confirmation of the presence of safflower together with the indigo can be obtained from PIXE (Particle Induced X-ray Emission) analysis to verify the presence of aluminium (providing the zone being analyzed has sufficient amount of detectable mordant) and thus establish if the intent was to use yellow or red with the indigo. Based on the tonality of the colour we are observing, it seems probable that the safflower was used as red and not yellow. Again in this case, our database gives the same reflectances and the fluorescence as on the wool. On silk, however, in addition to a diminished chromatic intensity, there is a difference in the fluorescence, most likely caused by the treatments made to the silk.

We must point out that in the case of the **colour red** (mordant dye) our database does not yet contain four other dyes having similar active principles, kermesic and carminic acids. The five dyestuffs in question are: **kermes**, extracted from Coccus illicis or Kermococcus from where the Roman name coccus or vermiculum granum is derived, as well as Kermes vermilio. It is one of the oldest dyes, possibly in use since the Neolithic. **Polish cochineal**, produced by the scale insect Porphyrophora polonica is also referred to as St John's blood due to the fact that the harvesting of the insect commenced on the Saint's feast day, the 24th of June; it has been used since at least the 6th Century AD. The use of this colorant on textiles extended throughout Northern-Central Europe and was used as an alternative to kermes, largely available around the Mediterranean. **Armenian cochineal** comes from Armenia and Azerbaijan and is produced by the insect Porphyrophora hamelii. This dye has been cited by Armenian chroniclers since the 5th Century AD and it is one of the least costly dyes. **Indian lac** is obtained from various insects such as the Kerria lacca or Kerria chinensis, species indigenous to India and Southeast Asia. It was used in India to dye silk since 1500 BC. In the thirteenth century, it was introduced in Europe in Spain and in Provence, and proved to be fairly stable for also dyeing wool. In Italy the use of the lac dye, precipitated using alum, also appears in some sources. **Mexican cochineal**, Dactylopius coccus (Coccus cacti L. 1758, Costa 1835, Pseudococcus cacti Brumeister 1839) became available in Europe after the discovery of the New World. It was imported from Central America and quickly replaced the use of kermes. Its use in Italy was met with great resistance, and in 1574 laws in the Veneto area prohibiting its use remained on the books, even though in practical terms the dye had been used for some time. [11]

Investigations using HPLC techniques (High Performance Liquid Chromatography) underscore the differences among the various components of the dyes using the named component, such as dcII, which has not yet been identified. The problem in this type of analysis is the quantity (more than 0.2 mg) necessary to make an analysis, often not possible to do because of the problems relating to the sampling of the original material (Wouters

1997), without considering the very high costs that the application requires for a precise analysis.

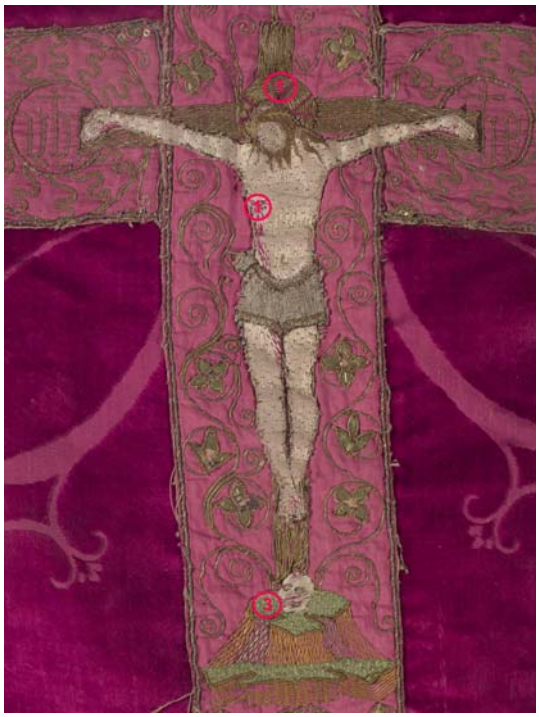

Fig. 5 Chasuble. Detail of some points on the back that show the red colour used in the halo (1) and for Christ's blood (1), and a green used for the base of Golgotha (3). In these instances, the same dyes used on the front of the chasuble were found.(vedi tabella)

Our database contains the Mexican cochineal dye Dactylopius coccus (Costa), premordanted with other mordants such as alum, cream of tartar (potassium bitartrate), and iron sulphate on both wool and silk. In the sampled points, we were able to identify the recipe on wool containing 5% cochineal, premordented with 25% alum and 6% cream of tartar. Instead for silk, the material in our chasuble, greater similarity is seen in the percentage from our database at 10% with the same mordants. Even though this percentage is higher than that for wool, we still do not attain the saturation level. We should signal the continual differences in tonality, with the same percentages, between wool and silk, a tendency also observed for other types of dyes in our database. In those instances where wool is more saturated, the silk always shows a lesser degree of saturation. Specifically for the chasuble, this means that if we compare the zones with the points chosen for silk with the reference samples they are less intense and less saturated than the wool reference samples. For this reason, comparisons with the more similar wool references are made in Figs. 2, 3, and 4. Given this situation, we need to think about the percentages used for dyeing silk that must obviously change with respect to the percentages used for wool; that is, in this case they must be greater in order to obtain the same intensity as our chasuble. Unfortunately, we still do not know if the multispectral analyses and the FORS applied to the other dyes give identical results, and therefore at this time, we can not date the voided velvet in our chasuble to a period after 1518, the point in history when Mexican cochineal is believed to have begun being used. In terms of historical-artistic information it would be highly important to know if this type of velvet, stylistically dating to the fifteenth century, is from a later period, confirming the hypothesis that this velvet-type was reproduced in numerous Italian centres using design-types and technical characteristics maintained unaltered over the centuries.

CONCLUSION

The attributions that emerge from the various types of samples are certainly interesting and supportive of continued research, and even though some vegetable and animal dyes can be used interchangeably to obtain different colours using different techniques and mordants, the building up of a databank based on analytical and documentary data is fundamental. We should point out that by preparing our recipes using firm sources we have obtained very interesting indications on mixtures such as safflower and indigo. Currently, we can not, therefore, definitively define the appearance of a specific dye, even though in some of the cases we verified, the analytical techniques, such as XRF (X-ray fluorescence analysis) advantageous because of its transportability, can furnish hard data on the use of specific mordants, for example iron or copper. Other mordants such as potassium alum (double sulphate of potassium and aluminium) can be identified with PIXE (Particle Induced X-ray Emission) technique, but for now, this requires the object to be transported and adapted to this method.

The multispectral investigations are organized in such as way as to be carried out, following a correct methodology, by individual laboratories and in comparison to existing databases. Additionally, it has already been widely shown that this type of diagnostic can and has provided important information for documentary purposes, especially for painted works of art. Just as for other types of investigations, however, it is only through experience and the implementation of the databanks that the best results are achieved.

ENDNOTES

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   Aldovrandi A., Buzzegoli E., Keller A., Kunzelman D., "Il falso d'autore indagato con tecniche non invasive Rapporto preliminare sulle indagini svolte in Santa Maria della Scala di Siena durante la mostra 'Falsi d'autore', giugno – dicembre 2004" in OPD Restauro N° 17, Florence 2005, pp. 265- 272.
2. Buss C. "Problemi di datazione e attribuzione: approccio interdisciplinare" in Tessuti serici italiani 1450 – 1530, exhibition catalogue Leonardo a Milano – Milan 1983, p.17. (CIETA Centre International d'Etude de Textile Ancienne).
3. See the critical edition by F. Gargiolli, 1868 (L'Arte della seta in Firenze) in the Codice Riccardiano n. 25880; Rosetti Giovan Ventura " Plictho de l'Arte de Tintori che insegna a tenger pani telle bombasi et sede si per l'arte magiore come per la comune", Venice 1548.
4. Perrone Da Zara C. "Ricerca sperimentale sulla tecnica tintoria della lana e della seta nei tessili antichi", supervisors: Parrini V., Bacci G., Diploma thesis School of Higher Education, Opificio delle Pietre Dure, November 1984.
5. Weighting is done to silk fibres to compensate for the weight loss resulting from degumming (removal of the sericin gum), carried out before dyeing. It incorporates various mineral or organic substances into the fibre. Some of the commonly used mineral substances for weighting are iron, aluminium, zinc and tin salts (in the form of tin phosphates and silicates); the most commonly used types of organic substances are tannins.
6. We gratefully acknowledge the collaboration and the use of the images provided by the acting-superintendent Anna Maria Spiazzi and the director of procedure and preliminary investigation, Chiara Rigoni of the Superintendency PSAE for the Provinces of Verona

Vicenza and Rovigo. The Textile Sector of the OPD provided the preliminary conservation treatment project for the Saint Firmus relics.

7. Paola Frattaroli "Tessuto della pianeta" in Sancti nunc riversi sunt ora i santi son tornati, historical-artistic exhibition on the cult of Saints Firmus and Rusticus over the centuries, for the 17th Centennial of their martydom (304 – 2004); edited by Paolo Golinelli – Caterina Gemma Brenzoni, p.. 16-18. Università degli Studi di Verona.
8. We would like to thank Ezio Buzzegoli, conservator at the OPD, for refining the false-colour reflected ultraviolet methodology and for his suggestions during the analyses on other types of objects.
9. All the readings using FORS were made by Bruno Radicati (IFAC-CNR in Florence), and whose help and support on this contribution we gratefully acknowledge. The characteristics of the technique used are: FORS measurements were made using an Ocean Optics mod. HR2000 UV-VIS-NIR portable spectrometer with a detector consisting in a 1024 diode concave array (CCD) and 0.8 nm/pixel resolution and a spectral sensitivity range 200 nm to 1050 nm supplied by a 20 W internally stabilised halogen lamp with a colour temperature of approximately 3000 K. The measurement head is a hemispheric probe (project IFAC-CNR) with a recording diameter of circa 2 mm and 2x45°/0° geometry. The sample was lit using a fibre optic bundle connected with a connector positioned on top of the head; reflected radiation was recorded, again using fibre optics, with two connectors positioned at 45° with respect to the surface being investigated. The measurement technique of using only the Y fibre (180° geometry) for the zones where it was possible to measure only one or two threads.
10. The history of indigo begins in India during the pre-Vedic period. The only Vedic materials now in existence are in texts known as "Veda" in archaic Sanskrit that span ten centuries (15th to 5th centuries BC). In the post-Vedic period, from 500 BC to 300 BC, the word nîla, which means sky blue (indigo), is mentioned as a blue dye.
11. See Bensi P., "Tintura dei tessuti serici nei secoli XV e XVI. Nota storica e tecnica" in Tessuti serici italiani 1450 – 1530, exhibition catalogue Leonardo a Milano – Milan 1983 p. 40-41.

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7. Perrone da Zara C., ."Aspetti storici e tecnici della Tintura nel Medioevo e Rinascimento" Catalogo mostra "Gli arazzi della Sala dei Duecento" – Studi per il restauro – Ed. Panini Modena 1985 – Florence 20 April 30 June.

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