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State of the art: non-invasive interrogation of textiles in museum collections

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Abstract. Heritage professionals are keen to see analytical science applied in support of the long-term preservation and access of our heritage. While textiles and other organic artefacts can be characterised by laboratory-based, non-destructive microanalytical techniques, rapid analytical methodology which is non-invasive, requires no sampling and can be performed on-site conveys particular advantages. Here we present an overview of our recent studies which have been directed at developing near infrared (NIR) spectroscopy as such a non-invasive collections management tool to assist textile curators and conservators. We review some of our earlier studies on FTIR and Raman spectroscopy, which were the prelude to our recent venture, before recounting our experiences with the NIR technique in the textile heritage context. It is our contention that a NIR spectrometer will become an essential part of the conservation scientist's tool-kit for interrogating textiles directly, enabling the identification of modern fabrics that inform decisions on storage and display, and the non-inteventive, routine condition assessment of aged textiles such as historic silks, informing the collections management strategy.

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Using X-ray microanalysis in a scanning electron microscope, while studying metal thread samples taken from a 15th century Rhenish tapestry, colleagues at the Textile Conservation Centre were able prove that gold coloured threads on the superfrontal of the altar cloth were brass-coated copper and therefore must have been added a few centuries after the original construction [1]. Besides offering means of authentication or expanding on an object's biography, analytical science also has a crucial part to play in the preservation and continued enjoyment of our cultural heritage. Decisions on the handling, treatment, display and storage of an artefact should be informed by knowledge of the composition of the object and the condition of the materials, as well as an understanding of their behaviour. The role analytical science has to play can be evidenced by many examples, including our recent work on the linen canvas of the *HMS Victory* Trafalgar sail and the split silk of the Shackleton Ensign [2].

In preparation for the bicentennial of the Battle of Trafalgar and the exhibition of the sail, for instance, we needed to obtain a quantitative estimate of the performance of the sailcloth. At the time we had little option but to undertake destructive mechanical testing of loose yarns from damaged areas. The results

showed the reduced tenacity of the linen and together with other mechanical test data suggested the need for cautious handling during conservation cleaning, and indicated the risk of free hanging display [3]; consequently the sail was displayed lying flat. While the removal of long yarns for testing was ethically acceptable in this case, non-destructive methodology that could have reported on the condition of the integral areas of the canvas would have been invaluable. Of course, such an approach is obligatory for the majority of heritage artefacts, as sampling must be minimal to avoid disfigurement and the integrity of the object must be maintained.

Prompted in particular by our work on historic sailcloth and silks, the theme of our research of late has been the development of non-destructive microanalytical techniques for the characterisation of historic textiles. Bearing in mind the need for on-site collections management tools, our most recent focus has turned to non-invasive technology, and especially near infrared spectroscopy, which might permit the direct interrogation of objects without the need for sampling. This paper briefly reviews some of our previous work, explains something of the basis of NIR spectroscopy, outlining applications in other related fields, and then presents an overview of our recent experience in non-invasive analysis within the textile heritage context. We conclude that near infrared spectroscopy has significant potential, together with other complementary portable techniques, to enhance heritage collections management. It offers the solution to representative sampling and routine monitoring of textiles, which can be difficult to justify for non-destructive, albeit micro sampling, methods. Our intention here is to highlight the potential of the technique with the aim of fostering its wider application in the field of organic heritage characterisation; for details of our methodology we refer the reader to the publications cited in the list of references.

Non-destructive and non-invasive vibrational spectroscopic analysis

In subsequent instrumental analyses of the yarns from the Trafalgar sail, we were able to observe characteristic Raman spectra which gave a qualitative indication of the deteriorated state of the linen canvas [4]. Good quality spectra were recorded using an irradiating laser wavelength of 1064 nm to reduce fluorescence interference, and a low laser power, to avoid sample degradation, with repetitive scanning. We identified two particular signatures of ageing: the reduced intensity of a band at 120 cm^{-1}, probably a consequence of disruption of the tertiary structure of the cellulose biopolymer; and an altered intensity ratio for bands at 1097 and 1122 cm^{-1}, which arise from the symmetric and anti-symmetric glycosidic (COC) stretching modes of the cellulose chains, coupled with the glucopyranose ring breathing mode, indicative of hydrolytic fission of the cellulose chain.

A common strand of our research is the quest to identify age-related condition markers for historic textiles in order to inform the work of curators and conservators, and we have preferred to capitalize on our expertise in the application of vibrational spectroscopy [5]. As illustrated above for Raman spectroscopy, these techniques can report on both the microstructure and chemistry of the organic polymers which compose textile fibres, from specimens of yarn just a few millimetres in length. Micro samples will also afford useful data from direct mid-infrared interrogation in attenuated total reflection Fourier transform infrared (ATR-FTIR) spectroscopy.

We have, for example, been able to characterise ancient silk excavated from the 8th century Buddhist Famensi temple at Xi'an, China, by subjecting a few short fibres to polarized ATR spectroscopy [6]. Silk is notorious for its catastrophic deterioration. The silk fibroin crystallite alignment becomes disoriented as silk ages. The relative extent of this disorientation can be quantitatively determined from the spectra. For a set of artificially aged silks, the associated spectral disorienta-

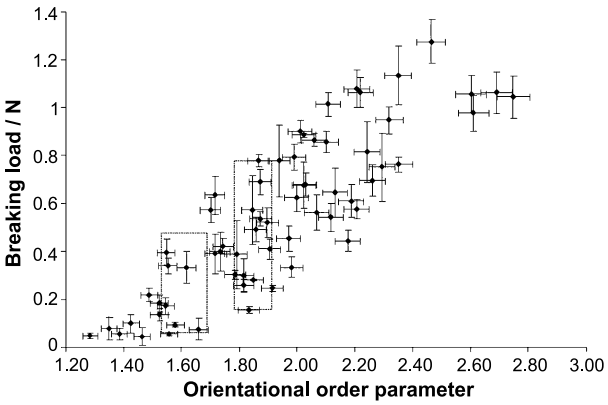

Fig. 1. A plot of the polarized FTIR-ATR spectrally derived fibroin crystallite orientational order parameter against the breaking load for a variety of artificially aged silks. The dotted boxes show the envelopes for the spectral parameters of two sets of archaeological silks from the Famensi temple, Xi'an

tion parameter was shown to correlate well with the mechanical performance of the cloth [7]. With reference to this model data, it was then possible to estimate the physical condition of the Famensi artefacts, which would inform the preservation strategy (Fig. 1).

The laboratory-based techniques in both of these particular examples may be referred to as non-destructive. They are indispensable for studies on models and precious micro samples. However, in terms of the ethics of sampling it is difficult to justify their general use in the analysis of heritage artefacts. The time it would take to fully characterise a single large object, like the Trafalgar sail, or a group of textile objects such as might comprise a museum collection is an additional concern. In these instances, ideally what is needed is a complementary rapid, non-invasive approach that will allow the direct interrogation of artefacts using portable equipment on-site, circumventing the need to remove samples for subsequent study in the laboratory.

Whole artefact analysis using standard Raman or ATR protocol is quite limited. A collection of fans at the Musée Galliéra in Paris have been subjected to Raman analysis and semi-synthetic cellulose-based polymers and additives such as camphor identified [8]. However, Paris and Coupry noted that the height of the spectrometer compartment restricted the size of the objects that could be studied, though use of a microscope attachment offers some further versatility [9]. Some ATR sample accessories can allow ready access for un-mounted, flat material, but the method is still regarded as an invasive technique due to the need for intimate contact between the sample and the ATR element, which for solids necessitates compression [10–12].

In the heritage context, non-invasive techniques have been researched and developed for application to inorganic components (e.g. [13]). For instance, X-ray fluorescence spectroscopy proved valuable in identifying mercury treated fur hats [14], and portable Raman spectroscopy showed its worth in differentiating gemstones [15] and pigments on miniatures [16], as well as characterising ceramic lustres [17]. There have been fewer reports of the *in situ* interrogation of organic heritage materials by portable vibrational spectroscopic techniques. While fibre optic and opto-mechanical accessories are available that allow the remote probing of artefacts in reflection [18], Raman spectroscopy remains subject to poor scattering by organics (necessitating increased irradiation with consequent risk of thermal damage) and fluorescence interference, and mid-FTIR spectroscopy generally affords anomalous spectra which are a mix of spectral and diffuse reflectance [12]. Thickett et al. have recently reported on the identification of cellulose nitrate lacquers on silver objects via a chalcogenide fibre optic probe [19], but the examination of thin coatings by FTIR transflection represents an ideal case. Anyway, the chalcogenide fibres are brittle and furthermore absorb in the region 2200–2050 cm^{-1} reducing their practical use *in situ* [20, 21].

We have therefore focused on portable near infrared spectroscopy, which is immediately a rather more innocent and versatile procedure, applicable not only to textiles but a variety of organic materials.

NIR spectroscopy

The near infrared region of the electromagnetic spectrum (4000–13,000 cm^{-1}, 2500–780 nm) lies between the visible region and the mid-infrared. NIR spectra result from the absorption of photons which excite complex vibrations in molecules, giving rise to combinations and overtones of the fundamental vibrations found in mid-IR (MIR) spectroscopy. Due to their relatively high frequency and anharmonic nature, vibrations of X–H bonds are the most responsive in the NIR; so for a proteinaceous silk fabric, for example, the spectral pattern is dominated by bands arising from OH (absorbed water, serine), NH (peptide) and CH (peptide, alanine, serine, etc.) containing moieties of the protein fibroin (Fig. 2).

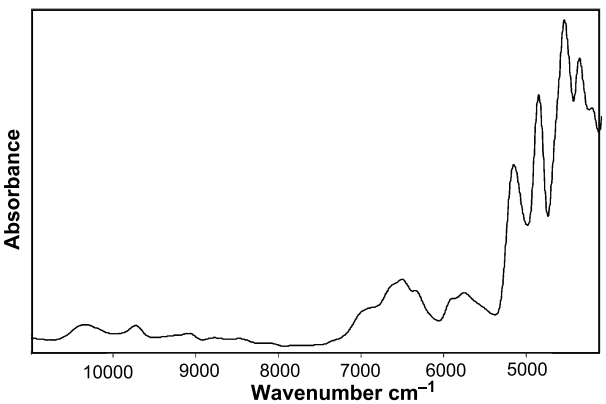

Fig. 2. The NIR absorbance spectrum of silk (relative humidity (RH) 45%): 10500–9000 cm^{-1} OH and NH second overtone; 7100–6300 cm^{-1} OH overtone and amide NH overtone and combination bands; 5900–5700 cm^{-1} CH overtone; 5170 cm^{-1} OH combination; 5000–4500 cm^{-1} amide NH combination; 4500–4000 cm^{-1} CH combination

In comparison with the spectral bands seen in MIR spectroscopy, the NIR spectrum is less well resolved, owing to the large number of combination and overtone transitions possible for each fundamental vibration; this complexity can preclude simple band assignments, though the spectral patterns are usually sufficiently characteristic for materials identification [12]. Conversely, NIR absorption coefficients are lower, so that spectral intensities are reduced. This has implications for the analysis of bulk materials, which may absorb MIR radiation completely [22]. NIR reflectance spectroscopy, the more appropriate for *in situ* analyses, will then report on material up to a millimetre or so below the surface and so is ideal for textiles.

Diffuse reflectance near infrared spectra can be acquired rapidly and through a remote silica-glass fibre optic probe, which is relatively flexible and allows efficient transmission of the radiation, facilitating remote interrogation of 3D objects. This removes the need for sampling, ensuring truly non-interventive analysis.

Such advantages have enabled the technique to be used successfully within the food industry, agriculture, polymer manufacturing and plastics recycling since the 1970s (e.g. [23]). From its ability to provide simple materials identification to more complex performance correlations, NIR has also found a range of applications within the textile industry, from quantification of textile blends for consumer information [24–26] to quality control of processing methods such as fibre retting [27] and fibre sizing [28, 29].

Direct NIR spectral analysis has allowed the development of rapid methodology for the distinction of different textile fibre blends. For example, Cleve et al. were then able to characterise mixtures of cotton, polyester, viscose, silk, wool, polyacrylonitrile and cellulose acetate [24]. In a related context, Kumagi et al. [30] have reported on the clear discrimination of individual synthetic polymers such as polyethylene, polypropylene and polyvinyl chloride. Ghosh has successfully used NIR spectroscopy to determine cotton maturity; immature cotton fibre can be problematic to dye. Regression calibration provided an excellent correlation between the NIR spectra and quantification of surface defects using light microscopy [31, 32]. A simple means to assess the commercial value of raw wool has been proposed by Larsen and Kinnison [33]. Fibre diameter and grease content are the two most important factors, determining the eventual use of the wool fibre and affecting the spinning process. Both characteristics have previously been determined through time consuming microscopic analyses and wet chemical processes, but again, the information can be extracted from the spectra following facile near infrared analysis. This last example also illustrates the statistical approach, in this case multiple linear regression, which is typically taken in the processing of near infrared data. The need for this arises partly from the complexity of the NIR spectra, and the often minor differences between data sets [34].

Near infrared spectroscopy has singular potential for interrogating the condition of organic artefacts, especially in association with univariate and multivariate spectral analysis [24, 35]. Despite its relative maturity though, it is only very recently that reports have begun to appear showing the value of NIR spectroscopy to conservation science. So far others have investigated its application to the characterisation of wood [36–41], paper [42–45], gelatin size [46], and polyurethane foams [47]. Our recent survey of textile conservators and curators in the U.K., in which we sought to determine end-user requirements which could be satisfied by on-site instrumental analysis, highlighted a need for modern materials characterisation at collections and the routine condition assessment of historic silks. After initial studies on linen [48], this then persuaded us to concentrate our research efforts on these two areas of textile heritage, having in mind the eventual goal of on-site application of the near infrared technique.

Modern materials in collections

Since their development in the late 19th Century, synthetic polymers have moved steadily into almost every area of life, and as a consequence, into a growing number of museum collections. However, the degradation mechanisms and preservation of these modern materials in the museum environment are less well understood, with differing production methods, additives and treatments likely to imbue varying and complex routes of deterioration. Five types of semi-synthetic and synthetic polymers are already of major concern in collections with regards to instability: cellulose acetate, cellulose nitrate, polyurethane, polyvinylchloride and synthetic rubber [49].

An area of particular interest to us is that of contemporary textiles. Synthetic polymers have of course been used extensively for clothing as textile fibres,

interlinings and surface coatings, and as fastenings and adornments. Over time their deterioration is inevitable, but it may be possible to minimise the ageing rates by careful control of the environment, and to further avoid damage to adjacent susceptible materials from reactive gaseous by-products release, topics of growing concern to conservators and curators. The degradation and stabilisation of these polymers vary through the classes, so it is first crucial to identify the materials held in collections. The nature of textile artefacts constrains sampling and conventional light microscopy, which can readily distinguish natural fibres, does not easily distinguish the synthetic polymer classes. Fibre processing methods such as extrusion do not convey polymer-specific morphological features, in addition, by their very nature, materials such as interlinings and padding are not readily accessible for sampling. The *in situ* application of NIR spectroscopy offers the solution to these problems, with the promise of fast identification of modern textiles and hence providing collection managers with the information required for a greater understanding of their collections, enabling informed long-term action plans and more immediate remedial conservation plans to be implemented.

On-site characterisation of contemporary textile collections

Our research in this area has been directed at the identification of textile materials within the collection context, which offers challenges beyond those which have been resolved for other applications such as quality control and recycling. So far we have compiled a spectral library of well-defined materials and tested our methodology on three site visits; at this point approximately 250 reference samples have been obtained from textile and polymeric materials manufacturers, covering the major classes of polymers found within textile collections. The optimum acquisition protocol was determined for on-site application and used in recording spectra of the reference standards [50].

The experimental work has utilised a Perkin Elmer Spectrum One Fourier-transform near infrared spectrometer (FT-NIR), with an Axiom fibre optic probe, employing a scan range of 4000–12,000 cm^{-1}, resolution of 8 cm^{-1} and scan accumulation of 64. The background reference was Spectralon®. All data were collected in absorbance. Spectral processing was carried out with Thermo Galactic Grams AI version 7, and spectral matching with the Spectral ID add-on. The first derivative least squares algorithm was found to give the search of the reference set with the best quality match of an unknown.

As we have already reported, our system, in combination with a trolley and flexible clamp to hold the probe, has proved particularly versatile for on-site use, and has allowed the spectrum of an area of fabric just a few millimetres across to be acquired in less than one minute. So, for example, we have been able to identify readily the five components of a small hand-

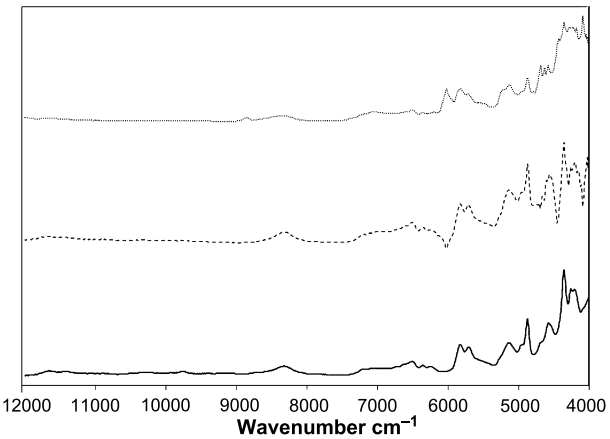

Fig. 3. Offset NIR absorbance spectra of the green netting before subtraction (·····), after a spectral subtraction of a polyester reference (- - - -) and a reference spectrum polyamide (——)

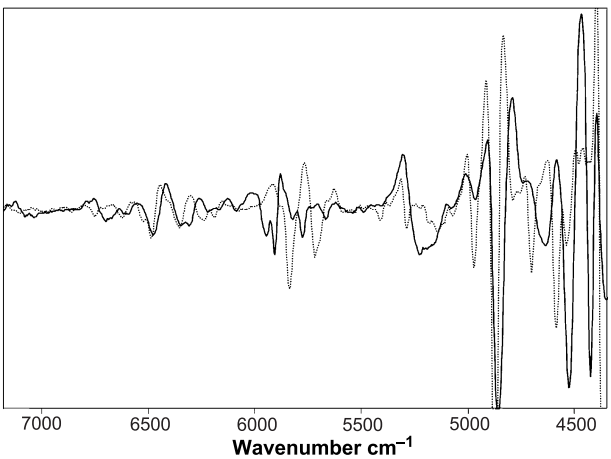

Fig. 4. Second derivatives of the near infrared spectra of a ''parachute'' dress from a regional collection (·····) and silk fabric (———). The subtle differences between the synthetic polyamide and natural silk are the more easily visualized in the processed spectra

bag, including the polyester ground fabric, the viscose embroidery threads, the PVC sequins and polyester bead decorations, and the polyamide netting on which the sequins were applied [51]. The last assignment attested to the value of spectral subtraction as the polyester fabric dominated the spectrum (Fig. 3).

A visit to a regional collection allowed us to rectify a mis-assigned item, a slip which was originally worn as an undergarment to a wedding gown and catalogued as silk [51]. During the Second World War rationing limited the availability of luxury fabrics and these were often salvaged from elsewhere. The fabric was purported to have been surplus parachute silk but our NIR results showed the garment to be polyamide. In this instance the distinction of the natural and synthetic polyamides is the more easily visualised in the processed spectra; the second derivatives help to emphasise the minor differences (Fig. 2).

Besides providing curatorial staff with accurate details of construction and manufacture, we have also been able to show that NIR can readily identify problematic materials and so help aid informed storage and display [52]. The deteriorating and sticky surface coating on a pair of shoes in the contemporary collection of the Victoria and Albert Museum was found to be polyurethane polyester, while the NIR spectra of the degrading designs on a set of T-shirts matched that of the PVC reference.

These exemplars show how such analyses could provide invaluable support to contemporary heritage textiles management. Furthermore, the studies are not only non-invasive but also rapid. In a particularly demanding situation, for example, we were able to complete the characterization of over 300 items within two days [53]. Beyond this more straightforward materials identification, NIR spectroscopy also holds the promise of a means to routine condition monitoring of textiles and other organic heritage, as outlined below for silk.

Condition monitoring of historic silks

Silk has been prized as a luxurious fabric since its first production in ancient China over five millennia ago. All over the world, historic silk fabrics are frequently part of museum collections, seen in ceremonial dress, military banners, tapestries, upholstery and so on. Unfortunately, the silk fibroin is subject to ready deterioration, promoted by a variety of endogenous and exogenous factors such as finishing agents, light and moisture. As a consequence many silk artefacts are somewhat fragile and problematic to handle, conserve and display.

An indirect method of assessing the condition of a fabric would be of particular value, informing conservation and curatorial decisions, and aiding in the prioritization of objects for conservation and in specifying safe display lifetimes. Appropriate non-destructive techniques have been developed which require just micro samples [7, 54], but rapid on-site, non-invasive monitoring would offer significant additional benefits. This would mean, for example, that a tapestry might not need to be taken down from display for characterization, and that data could be obtained for silk threads across the whole of the object. The NIR technique could once again offer the solution if it is possible to identify a spectroscopic marker of condition.

We were aware from previous work that moisture sorption by natural textiles is a condition-related parameter [5], and furthermore that it is possible to determine the water content of a variety of products by monitoring the water absorptions in the near infrared [55]. We therefore decided to first test this univariate approach [56].

Studies on artificially aged model silks

The intensity of the water combination band at 5170 cm^{-1} in the near infrared spectrum of silk (e.g. Fig. 2) shows the typical behaviour of a hygroscopic

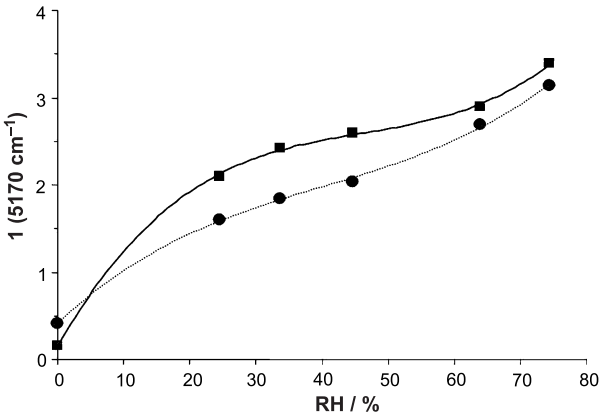

Fig. 5. Plots of the normalised intensities of the water combination bands, I (5170 cm^{-1}) for unaged (-■-) and humidity aged (···●···) silks versus relative humidity (RH). The size of the symbols is indicative of the standard error amongst the set of 24 replicate NIR spectra at each data point. Third order polynomial trendlines generated in MS Excel are included to aid visualization

natural fibre consisting of a semi-crystalline hydrophilic polymer aggregate (Fig. 5). The intensity is diminished for a silk model artificially aged at high humidity, equivalent to a few hundred years natural exposure, suggesting reduced sorption. However, while qualitatively useful, we have found that this does not provide a readily quantifiable indirect measure of condition with sufficiently clear distinction for the wider variety of aged silks subject to thermal and light ageing [56].

Next we wondered whether dynamic studies would be more revealing. Tsuchikawa et al. monitoring the process with NIR spectroscopy, have noted an altered deuterium exchange rate for archaeological wood, compared to new wood, immersed in D_2O [41]. In similar studies carried out under a D_2O saturated atmosphere, we have seen the water combination (5170 cm^{-1}) and overtone bands (7000 cm^{-1}) from silk rapidly declining in intensity in a period of minutes (Fig. 6), while the amide NH bands in the accessible amorphous regions of silk fibroin (6686, 6581 cm^{-1}) show evidence of slightly slower but still rapid exchange. The NH moieties in the crystalline zones (6486, 6352 cm^{-1}) reduce in intensity much more slowly. However, the data for aged silks is very similar and does not correlate with the degree of degradation as measured by the tensile strength of the fabrics [56].

Others have found correlations between NIR spectra and mechanical properties through a multivariate spectral data analysis, e.g. this has presented rapid methodology for the assessment of wood in the timber industry (e.g. [57]), and the same approach is now being developed for the characterization of paper in archives and libraries under the EU Survenir project [42]. In view of the seemingly subtle differences between the spectra of new and aged silk, other than in the regions of the water bands, we were somewhat skeptical as to the success of this more complex chemometric analysis. However, the results have proved extremely encouraging. As shown in Fig. 7, for example, which includes data for model silks subjected to artificial ageing under extremes of heat, light and humidity, NIR spectroscopy does appear to produce good predictive correlations with primary condition parameters.

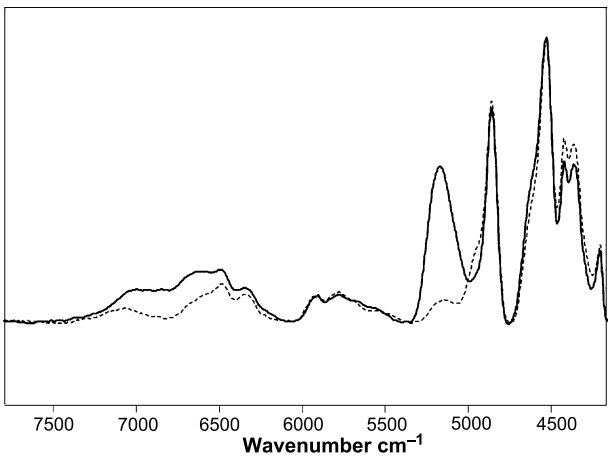

Fig. 6. NIR absorbance spectra of unaged silk following deuterium exchange under a D_2O saturated atmosphere, at 0 (——) and 0.5 (- - - -) h. Spectra are offset and baseline corrected to allow the changes to be readily visualized

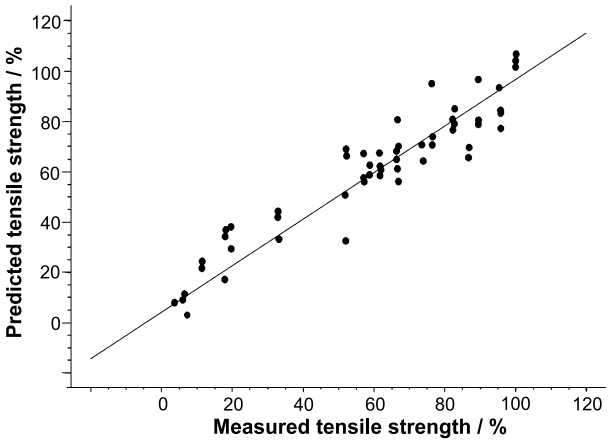

Fig. 7. A full spectral multivariate analysis was performed on a data set of 60 NIR spectra from heat, light and humidity aged silks using the Unscrambler 9.6 package (Camo) following multiplicative scatter correction. Partial least squares regression against the % residual tensile strength gave a correlation coefficient 0.95 and a root mean square error of prediction 9.8

We are currently extending this research with the aim of creating a tool for on-site use, leading to better informed preservation and improved access of silk collections.

Conclusions

There is an enthusiasm amongst conservators and curators for the application of analytical scientific tools to enhance heritage collections management. While laboratory-based, non-destructive techniques are invaluable for studies on models and precious micro samples, they have limitations, especially with regard to the comprehensive and representative analysis of large objects and entire collections. Rapid analytical methodology which is non-invasive and can

be performed on-site presents particular advantages. Amongst the small range of candidate techniques for the characterisation of textiles, near infrared spectroscopy appears to warrant serious consideration. Remote interrogation of artefacts is readily carried out with a fibre optic probe attached to a portable system. Modern materials within contemporary textile collections can be easily identified to aid in interpretation and inform decisions on storage and display. Our preliminary work on silk suggests that NIR has significant potential too for the condition assessment of the organic polymers associated with textiles, so further supporting the work of conservators and curators and ensuring the best preservation of our heritage. We hope that through sharing our experience in this overview of the textiles paradigm we will enhance awareness, both within the analytical science and heritage communities, of the exciting prospects for non-invasive investigations of our organic cultural heritage.

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