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Quantitative chemical near-infrared hyperspectral imaging of Islamic paper

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Abstract

Heritage objects are well known for their compositional inhomogeneity due to materials and processes used in their production. Hyperspectral imaging is gaining importance in the field of heritage conservation by expanding spectroscopy to the examination of an entire surface of an object. This paper focuses on the application of near-infrared hyperspectral imaging to the characterisation of Islamic paper using a pushbroom HSI scanner in the 1000–2500 nm range to collect hyperspectral datacubes. A calibration target was devised using 105 samples from the well-characterised reference Islamic paper collection of the UCL Institute for Sustainable Heritage. Two material properties of Islamic paper were of interest: starch sizing and degree of polymerisation (DP). In addition to the developed discrimination and regression models using multivariate data analysis methods, a quantitative chemical map of the DP of an Islamic paper was generated as a case study for improved visualisation of the inhomogeneity of material properties, of value to researchers and conservators. As a case study, this research shows the wealth of valuable chemical information that near-infrared hyperspectral imaging could provide for diverse heritage applications in the future.

Keywords

Spectral imaging; Near infrared; Chemical mapping; Multivariate data analysis

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Introduction

Understanding the material properties of heritage objects is vital for their successful conservation. Despite extensive research so far, spatially resolved quantitative chemical analysis of heritage materials is not straightforward (Strlič and Kolar 2005, Oriola et al. 2014). Many studies have been conducted to study cellulosic collections using near-infrared (NIR) spectroscopy as a non-destructive analytical technique in combination with multivariate data analysis for the purpose of material characterisation (Oriola et al. 2014, Mahgoub et al. 2016, SurveNIR 2008). However, such studies have so far focussed on spot analysis while degradation processes progress in a heterogeneous manner with possibly significant local differences due to the use of different materials and additives, such as primer and paint layer on canvases and sizing and finishing layers in the papermaking process (Trafela et al. 2007, Oriola et al. 2014). Development of a methodology that would enable the distribution of the chemical composition of a whole artwork to be mapped is of great importance (Kubik 2007, Wang and Paliwal 2007, ElMasry and Sun 2010).

Over the last decade, spectral imaging systems that simultaneously record spectral and spatial information, used in remote sensing, medicine, forensics and food engineering, have advanced dramatically with the evolution of technology and sensors. This research quickly resonated in the field of heritage conservation (Lu and Chen 1999, Fischer and Kakouli 2006, Chang 2007, Kubik 2007, ElMasry and Sun 2010, Liang 2012).

Spectral imaging technology, including hyperspectral imaging (HSI) and multispectral imaging (MSI), has widened the possibilities of imaging and material characterisation (Fischer and Kakouli 2006), which has the potential to improve knowledge of the distribution of material properties while investigating an entire object (Lu and Chen 1999, Wang and Paliwal 2007, Liang 2012, Dooley et al. 2013).

Most of the applications of spectral imaging in the field of cultural heritage have focussed on qualitative investigation due to the complexity of the objects and lack of standard materials for calibration. The three-dimensional dataset (hypercube) resulting from HSI in the NIR region contains two spatial dimensions and one spectral dimension, which can be used to study physical characteristics as well as chemical composition, i.e. the state of an object (Lawrence et al. 2003, ElMasry and Sun 2010, Yao and Lewis 2010). Due to the complexity of the data, multivariate regression is required to extract quantitative information from this spectral region (ElMasry and Sun 2010, Dooley et al. 2013). In 2011, a 2D quantitative chemical map of iron gall ink on paper was developed to visualise the degradation and material properties for the first time (Cséfalvayová et al. 2011); however, the need remains to study the effect of different measurement conditions and calibration parameters, e.g. the spectral distribution of lighting and its intensity, or surface morphology, as well as to ascertain calibration stability. In other words, focus needs to be on the metrology of quantitative chemical mapping based on hyperspectral imaging. This is an active area of research that requires further studies (Fischer and Kakouli 2006, Dooley et al. 2013).

In view of this, this paper explores the potential for mapping the chemical composition of Islamic paper, specifically starch sizing and the cellulose degree of polymerisation (DP). While starch is a characteristic component of Islamic paper (Mahgoub et al. 2016), the cellulose DP provides essential information about an object's conservation condition. Together, these

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parameters provide a better understanding of the material and provide a measure of change in collections through imaging.

For the quantitative model, a calibration target was prepared using 105 samples and imaged using a pushbroom HSI scanner in the 1000–2500 nm range, while the collected datacubes were analysed using multivariate classification and regression methods.

Materials and Methods

Islamic paper calibration target

A calibration target (Figure 1) was prepared using 105 samples (Table 1) from the well-characterised reference Islamic paper collection of the UCL Institute for Sustainable Heritage (Mahgoub et al. 2016). The target was imaged, analysed and used to build a calibration method using multivariate analysis methods as described below. Different objects from the same reference collection were also used to validate the method and to build quantitative chemical maps.

Figure 1. Calibration target with 105 characterised samples of Islamic paper.



Table 1. The 105 Islamic paper calibration target samples, showing the distribution of samples containing starch (yellow cells).

	11	10	9	8	7	6	5	4	3	2	1	
1	56-3	55	45-3	42	36	26	6	5	3	56-1	45-2	1
2	131	125-1	121	114	108	97	91	85	84	83	74	2
3	43	18	10	9	8	29-2	127-2	133-2	17	44	125-2	3
4	126	116	115-1	113	107	100	98	72-4	69	49	29-1	4
5	2	129-2	129-1	16-1	25	35	37	115-3	133-1	128	127-1	5
6	109	38	39	40	41	47	48	63	65	75-T1	76	6
7	34	33	89	110	111	117	118	123	124	130	120	7
8	75-1	27-1	72-2	101	96	82-1	73-2	53	57	50	46	8
9	95	72-1	62-1	52	31	30-1	28-2	23	11-1	4	62-2	9
10						72-3	24	132	106	104	103	10
11												11

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The presence of starch in the samples used in the calibration target was previously identified using the iodine test (Isenberg 1967, Baker 1991). The DP of cellulose in paper was determined using the viscometric standard method and the Mark-Houwink-Sakurada equation (Evans and Wallis 1987, BS ISO 5351:2010).

Hyperspectral imaging system and acquisition parameters

A pushbroom HSI scanner (GILDEN Photonics, Figure 2) was used to collect hypercubes in the range of 1000–2500 nm with a spectral resolution of 6.3 nm using a mirror scanning setup. The scanner is based on a line spectrograph (Specim, ImSpector N25E) with a 30- μm slit connected to a mercury-cadmium-telluride (MCT) camera with a spatial resolution of ~ 0.8 px/mm. The maximum frame rate of the camera is 100 fps with an F/2.0 fixed aperture and fixed scanning distance of ~ 110 cm. It has a 2D detector array which simultaneously acquires one spatial dimension (x-direction: 320 pixels) and one spectral dimension (256 wavelength channels) along the direction of the scanning stage (y-direction). The current setup of the scanner allows the scanning stage to accommodate objects up to a maximum of A3 size ($\sim 30 \times 40$ cm). The object is illuminated by a line of halogen lamps (at 250 or 500 W) at an approximately 30° angle and at ~ 18 cm distance. Lights were warmed up for ~ 1 h before data acquisition to allow them to stabilise. In all measurements, a spectral flattening filter was used in front of the lens and Whatman filter paper no. 1 was used as background. Proprietary software (SpectraSENS) provided by the manufacturer was used to control the whole process of acquisition and calibration. The system was spectrally and spatially calibrated. Table 2 shows all the acquisition parameters as used during the measurements.

Figure 2. Hyperspectral imaging scanner used in this study.

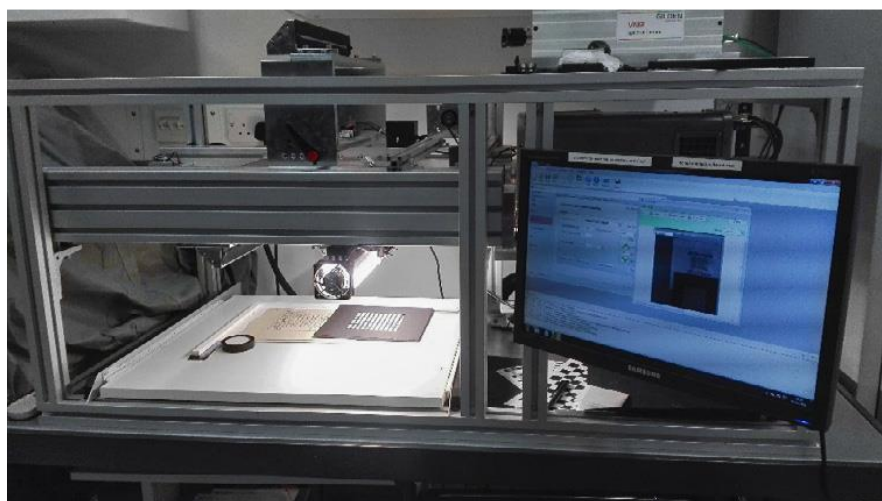


Table 2. Hyperspectral imaging scanner acquisition parameters.

Lens	30 mm	Aperture	F/2.0
Exposure	6 ms	Binning	None
Scan speed	54.9 mm/s	Gain	1
Lights	500 Watt	Spectral range	1000-2500 nm

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Hyperspectral data analysis

HSI hypercubes were acquired using the SpectraSENS software. All the data were acquired in raw format and then converted into reflectance using a Spectralon reference standard and dark current detector as calibration references.

Two different multivariate data analysis methods were used to analyse the hyperspectral datacubes: partial least squares (PLS) regression for the determination of the DP of cellulose in paper, and principal component analysis with a linear discriminant analysis (PCDA) to identify the presence of starch. Data in the spectral range of 1450–2350 nm were used in the calculations.

PCDA is a supervised classification (Blanco and Villarroya 2002, Næs et al. 2002, Stuart 2007, Miller and Miller 2010) and it was used to develop a discrimination model to determine the presence of starch sizing using all the samples (45 with and 60 without starch) in the Islamic paper target. Principal component analysis (PCA) is first applied to reduce variable dimensions before discrimination, followed by linear discriminant analysis (LDA) performed on the selected PC scores.

PLS (Næs et al. 2002, Brereton 2009, Miller and Miller 2010, Brereton and Lloyd 2014) was used to build a calibration regression model for the determination of the DP. Using the reference laboratory method (BS ISO 5351:2010), only 57 samples of the Islamic paper target were measured, as lignin-containing samples cannot be dissolved in the cupriethylenediamine solution.

Different spectral pre-processing methods (Manley 2014) were tested to optimise the quality of calibration. Standard normal variate (SNV) and Savitzky-Golay methods were used.

Validation was performed using the leave-one-out cross-validation method (LOOCV). The root-mean-square error of cross-validation (RMSECV) and the correlation coefficient R_{cv} were calculated to evaluate the models (Næs et al. 2002) using these equations:

$$RMSECV = \sqrt{\frac{\sum(y_i - y_i^{cv})^2}{n}}, \quad R_{cv} = \frac{\sum(y_i - y_m)(y_i^{cv} - y_m^{cv})}{\sqrt{\sum(y_i - y_m)^2 \sum(y_i^{cv} - y_m^{cv})^2}}$$

Where y_i contains the known values of DP for each sample, y_i^{cv} contains the DP values that are estimated by cross-validation and n is the total number of the samples. y_m and y_m^{cv} are the mean of the known and estimated DP values respectively.

Matlab with the aid of PLS toolbox library from Eigenvector was used to process the datacubes, develop models and build chemical maps.

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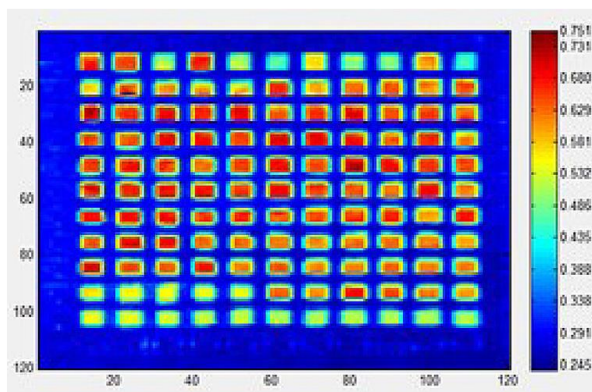
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Results and Discussion

Sample Selection

To identify the sample pixels in the calibration target, an average image was calculated from the datacube. Then, based on the values, a threshold was selected to differentiate between sample and non-sample pixels (Figure 3). Each sample square is $\sim 7 \times 7$ pixels (1 cm^2). Representative spectra were collected from the middle of each sample (ROI – region of interest – of 3×3 pixels) and used in the calibration model.

Figure 3. An average image of the calibration target calculated over the spectral dimension for each pixel. The colour scale shows the distribution of the average values, the minimum average value (blue) assigned to holder pixels and maximum values (red) representing sample pixels.



NIR chemical imaging: Data analysis

Starch Presence

All the Islamic paper calibration target samples were used to build the calibration model for the discrimination between samples with (1) and without starch (0). The distribution of samples in the target is provided in Table 1. PCDA was used for starch discrimination and validation success was expressed as a proportion of the correctly identified samples.

The average spectra (ROI = 3×3 for each sample) were pre-treated with SNV in the range of 1450–2350 nm. In PCA, the selection of the number of PCs was based on the results obtained from the application of LDA to 1–25 PCs, and the number leading to the highest % correctness on cross-validation was selected.

The proportion of successfully identified samples (83.6% of the 105 calibration samples, 9 PCs) shows an excellent potential to use the HSI technique for discrimination. For comparison, the LDA model (Mahgoub et al. 2016) developed using a handheld NIR spectrometer (1600–2400 nm, spectral resolution 8 nm) was somewhat better with 94% of correctly identified samples (138 samples in the calibration dataset). Although different illumination methods, backgrounds for calibration and measurements were used, as well as different spectral ranges, these differences should have been accounted for in the process of calibration.

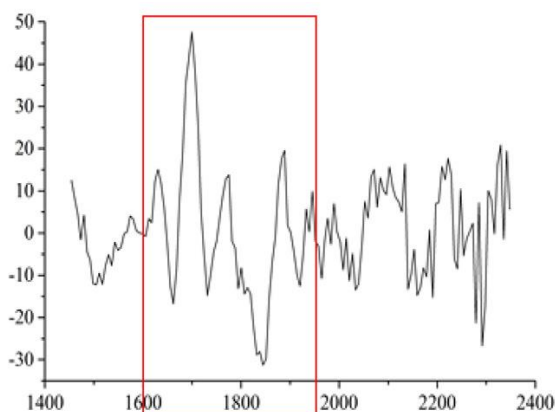
In order to explore the wavelengths that contributed most to the model, the weights for each wavelength in the discriminant function were calculated by combining the coefficients of the

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LDA model with the loadings from the PCA (Figure 4). The spectral range of 1600–1950 nm appears to have the most pronounced influence. This will be studied further in future work.

Figure 4. Weighted PCA loading vectors based on LDA coefficients, indicating the wavelengths with the highest contribution (1600–1950 nm) to the starch discrimination model.



Degree of polymerisation

Only 57 samples were used to build the regression model to predict the DP for unknown samples, as not all samples could be measured using the viscometric method. However, the size of the dataset still conformed to the relevant ASTM E1655 standard (ASTM 2000). The distribution of the DP values for the samples is presented in Figure 5.

For the PLS model, the spectra were pre-treated using the Savitzky-Golay filter (1st derivative). Different polynomials (2, 3 and 4) and filtering window sizes (5–55 points) were tested to allow selection of the optimal parameters for the model. Equally, different numbers of PLS factors/latent variables (1–25) were set and the calibration model was calculated for each combination, following which the optimal factor number was selected based on the model predictive quality as expressed by small RMSECV (prediction bias) and high R_{cv} .

The optimal calibration model gives an RMSECV = 318 and R_{cv} = 0.70 using 13 factors (n = 57, 3rd polynomial, window = 7 points; Figure 6). Comparing this model to the PLS model developed using NIR spectrometry for the determination of the DP for Islamic paper (n = 45, RMSECV = 298, R = 0.88; Mahgoub et al. 2016) and for European paper (n = 86, SEE = 161, R = 0.99; Trafela et al. 2007), the model performs in a similar manner, taking the differences in spectral resolution and different sizes of datasets into account, in addition to the different pre-processing methods used. It is possible that better results could be obtained with a bigger dataset in the future.

Figure 5. Distribution of the DP values for 57 Islamic paper samples in the calibration target.

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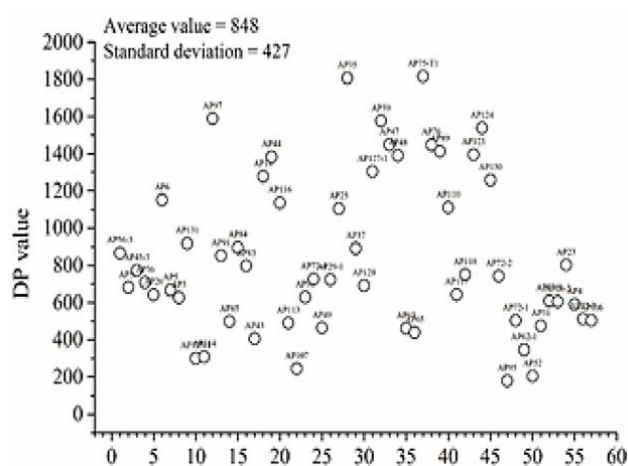
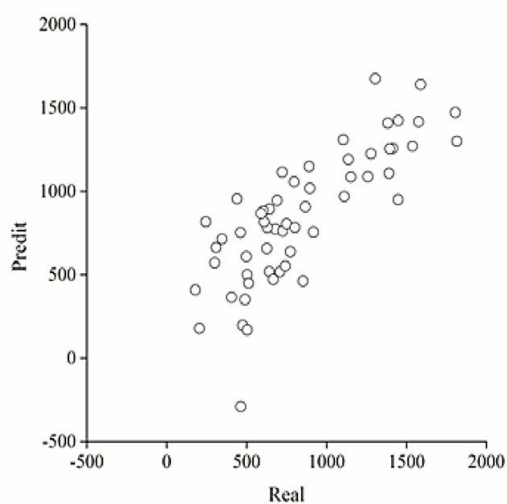


Figure 6. The results of the developed PLS regression model for the determination of DP of cellulose in paper using 57 Islamic paper samples. The plot correlates predicted with actual DP measurements.



Quantitative NIR chemical map of Islamic paper

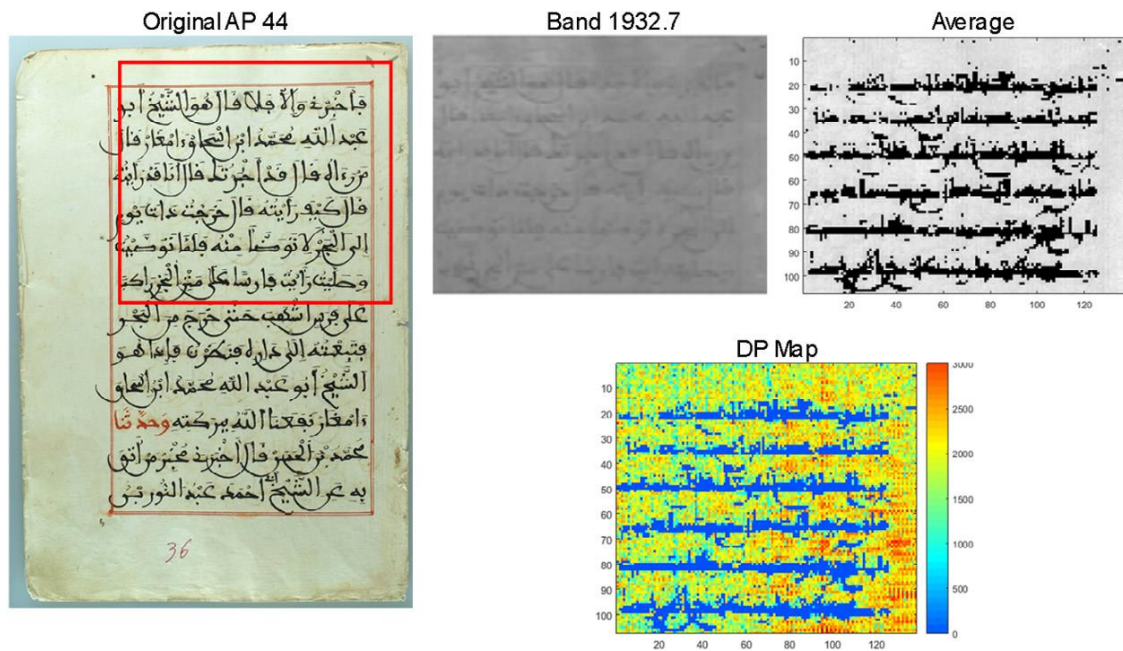
The main advantages of HSI in the NIR region over NIR spectroscopy is the ability to produce chemical images which allow visualisation of the spatial distribution of chemical composition in non-homogeneous samples (Manley 2014).

Using the developed PLS regression model to determine the DP of cellulose in Islamic paper, a quantitative DP map was generated for one document (AP 44) from the reference sample set (Figure 7). The figure shows the predicted DP values for each pixel in the image. The pixels with ink were excluded as a different regression model should be used for these. The actual measured DP of the document, determined using viscometry, is 1380.

Figure 7. Image of an Islamic paper (AP 44) from the UCL Institute for Sustainable Heritage Reference Collection. From left to right: visible image, image at 1933 nm, average image with ink pixels excluded, and the preliminary DP map.

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A visually fairly homogeneous object was selected specifically to establish a variation in the modelled DP pixel-by-pixel. A level of what appears to be noise can be observed in the predictions, which requires further detailed evaluation and will be part of our future work. It is unlikely that this is chemical noise, as while differences in DP can be substantial across a page, there is usually a slow gradient from the spine of a book towards its margins (as could be observed in the image as well); however, significant differences within areas represented by the size of a pixel (1.2 mm/px) in Figure 7 are highly unlikely in well-pulped and homogeneous papers. Further research is necessary to evaluate if this is due to spectral noise or variations in paper surface morphology leading to minor differences in spectra.

While the map opens new and exciting areas of further research into quantitative imaging, it also provides conservators and researchers with the possibility to visualise deterioration, which makes the tool suitable for evidence-based conservation decisions, e.g. identification of areas that require preferential treatment.

Conclusion and Further work

In this study, NIR hyperspectral imaging (1000–2500 nm) was explored for non-destructive characterisation of Islamic paper, due to its potential to provide spatially and spectrally resolved information for a whole surface of an investigated object. A custom-made target composed of 105 Islamic paper samples enabled the development of calibration models. A discrimination model was developed to identify starch as an important characteristic of Islamic paper and a PLS regression model was developed for quantitative mapping of the DP of cellulose in paper, which could provide visual cues as to the current conservation condition of the document.

The quantitative imaging method shows excellent potential in comparison with point-based spectrometry. In future work, the influence of further experimental parameters will be tested to provide a fully evaluated and robust quantitative imaging method for a series of material properties.

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Acknowledgments

The authors gratefully acknowledge the financial support of the EPSRC Centre for Doctoral Training in Science and Engineering in Arts, Heritage and Archaeology (SEAHA), and EU Horizon 2020 project NANORESTART.

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