

FAST AND NON-INVASIVE IDENTIFICATION OF BINDING MEDIA IN EASEL PAINTINGS BY A PORTABLE HYPERSPECTRAL SENSOR

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Abstract

A portable hyperspectral sensor collects reflectance spectra in the 350-2500 nm range, allowing the identification of not only inorganic but also organic materials. In this paper, a non-invasive and non-destructive characterization of pictorial binders was used. The results show that their main spectral differences are located between 1200-2400 nm. It is also indicated that when a binder is mixed with colorants, some modifications occur in respect to its original spectrum. In addition, the first derivative transformation of original spectra allows an easier identification of these organic binders, where the most discriminating regions are 1160-1250 nm and 1660-1800 nm.

Keywords

Organic binders, Reflectance spectroscopy, *In situ* analysis.

Introduction

Characterization of materials has been acknowledged as one of the most significant steps in conservation of artworks since it plays an essential role in understanding chemical and physical composition, as well as the state of conservation. In addition, it also provides useful advice to conservators for further conservation intervention. Thanks to the fast pace of scientific and technologic development, currently there are many analytical techniques for material identification that can be used both in labs and in-situ. These include both non-invasive and invasive techniques. In-situ non-invasive analysis is an approach of the utmost importance in this field because sampling is not always possible and, in any case, it should be minimized as much as possible. Besides, the general knowledge acquired from non-invasive analysis makes possible the identification of areas where the sampling for invasive in-depth analyses is necessary to get additional information (e.g. when an overpainting hides the original pictorial layer).

Reflectance spectroscopy is a well-established technique for acquiring compositional information of artworks thanks to its high degree of selectivity of colorants. Compared with other analytical spectroscopic techniques, reflection spectroscopic analysis commonly requires no sample preparation, is less time-consuming and more cost-effective. FORS (Fiber Optic Reflectance Spectroscopy) analysis, first used by Bacci (1995) as an innovative analytical method for material identification, has been proved to be a very popular method in pre-

liminary investigation for material identification of artworks (Bacci, 1992; Aceto, Agostino, Fenoglio, & Picollo, 2013). Most of the research performed so far with this technique used it for pigment identification (Dupuis, Elias, & Simonot, 2002; Aceto *et al.*, 2014). Those instruments mainly work in the 250-1100 nm range and in some extended ranges to even longer wavelength, but the most used regions are focused on the UV and visible regions (Aceto *et al.*, 2012; Cosentino, 2014a).

In the present study, a portable hyperspectral sensor was employed for material identification. Hyperspectral sensors are spectroradiometers widely used in the geological field (Ben-Dar, Inbar, & Chen, 1997; Ramakrishnan & Bharti, 2015; Clark, 1999). They are designed to acquire continuous reflectance spectra in the 350-2500 nm range, which allows the identification of both inorganic and organic materials in artworks. The shape of the spectral curves in the visible range provides useful information to differentiate the color of materials. On the contrary, the spectral absorptions in the Visible and Near InfraRed (VNIR) regions are a function of different electronic and vibration processes of molecules (Gaffey, 1986). Besides, hydroxyl, which is often a part of the crystal structure of a mineral and closely related to organic materials, is often infrared active. H₂O and CO₃²⁻ can also be identified in the 900-2500 nm range because of the overtones and combinations bands (Clark, 1990). For the artworks, the earliest application of the hyperspectral sensor that was used as a non-invasive technique for in situ characterization of stone surface alterations (to better understand the alterations) was done on the façade of the Santa Maria Novella church (Camaiti, 2008). Later, Ricciardi, Pallipurath, & Rose (2013) and Maynez-Rojas, Casanova-Gonzales, & Ruvalcaba-Sil (2017) used them on paintings but mainly for color discrimination; moreover, the discussion of spectral range is limited to wavelength shorter than 1700 nm.

However, due to the complexity of the targeted materials, such as painted surfaces, the interpretation of the reflectance spectra is not always easy. Consequently, the identification of organic materials on painted surfaces by this non-invasive technique is underestimated. To better “decode” the multi-component reflectance spectra, a standardized database of raw reflectance in full range is urgently needed. Currently, heritage scientists generally used a referred standard spectral database from the United States Geological Survey Clark *et al.* (2003) and Istituto di Fisica Applicata Nello Carrara (IFAC’s database) (IFAC, 2011) for pigment identification. Reflectance spectra of organic materials as pure materials, like binding media, are seldom discussed and compared. Cosentino created a database of reflectance spectra of historical pigments in powder and mixed with different binders within the 200-900 nm spectral region (Cosentino 2014b). However, he cannot treat as standards the pure compounds because not only the range is relatively limited, but also the spectra were collected after the pigments were mixed with egg yolk and applied on parchment and paper. Besides the raw spectra, their derivative transformation can also make a difference in characterization of organic materials. Spectra after derivative transformation may swing greater amplitude than the original one; more importantly, peaks of overlapping bands become more distinguishable to separate out Aberásturi, Jiménez, Jiménez, & Arias (2001). However, higher derivative order as second or even higher increases the noise of the spectra (Tsai & Philpot, 1998). Thus, the first derivative transformation is selected in our research. Dooley *et al.* (2017) discussed the discrimination between oil and alkyd resins in paintings through their first derivative spectra.

In this paper, we report and compare the reflectance spectra, and their first derivatives, of the three most common binding media in easel paintings (linseed oil, animal glue, and egg yolk) in the 350-2500 nm full range by a hyperspectral sensor, along with the discussion

on their discrimination when they are mixed with different pigments. Particular attention is paid to the 1200-2400 nm spectral range. The results obtained show that this portable hyperspectral sensor can be used for the identification of pictorial binders both as pure material and as mixtures.

Methods and Materials

The hyperspectral sensor employed in this study was an Analytical Spectral Devices FieldSpec FR Pro 3, a portable high-resolution spectroradiometer (Figure 1). It is designed to acquire Visible, Near Infrared (VNIR: 350–1000 nm) and Short-Wave Infrared (SWIR: 1000-2500 nm) punctual reflectance spectra in 0.2 s per spectrum. The sampling interval is 1.4 nm and 2 nm for the VNIR and SWIR spectral regions, respectively. The VNIR spectrometer has a standard spectral resolution (full-width at half maximum of a single emission line) of 3 nm at around 700 nm; while in the SWIR region the spectral resolution is 10-12 nm from 1400 to 2100 nm. Spectra were collected and then processed using ASD's RS3 and ViewSpec Pro 6.0 software to eliminate spectral noise. The instrument is equipped with a contact reflectance probe (fixed geometry of illumination and shot with a spot analysis of about 1.5 cm²) that provides an internal light source, so spectra can be acquired without involving the typically used ambient solar illumination like in the applications of remote sensing.



Figure 1 - Portable hyperspectral sensor, ASD FieldSpec FR Pro 3.

The spectra collection of binding media as pure materials was carried out at first on a Teflon support. Animal glue was dissolved in water (10% w/w), deposited on a substrate to form a film and tested after total drying. The egg yolk was dried and then put on the support as a thin layer, while polymerized linseed oil was prepared on a glass slide by deposition of liquid oil in 2010 and then exposing it for 6 years to indoor light at room temperature and humidity. The commercial canvas from Zecchi (Florence, Italy) was divided into small squares of 10*10 cm² size as support for easel painting mock-ups. The canvas had already been grounded with a layer of gypsum and linseed oil. Four pigments were selected: synthetic

cinnabar, yellow ochre, azurite, and lapis lazuli, which are traditional colorants, also used in the ancient times (Cennini, 1991), and representative of different colors and chemical composition. Binders were mixed with these pigments and then evenly painted by brushes on the canvas in accordance with the methodology described by Cennino Cennini (1991). Typically, for the tempera painting samples, the amount of pigment and egg yolk was almost the same. During the mixing, some water was added into the mixture to decrease the viscosity for easy application. Meanwhile, in the case of a mixture of pigment and animal glue or linseed oil, the average amount of the pigments was almost twice the amount of the binder.

Reflectance spectra were collected with the Contact Probe by direct contact with each material or painted surface. In order to eliminate the influence of the ground layer and the canvas support on the reflectance spectra, a background was first collected on unpainted surfaces.

The spectrum of each compound, used as standard, was the average spectrum of 10 acquisitions collected on the same studied surface of each material. The spectra were then processed as collected and as their first derivative by ViewSpec Pro 6.0 software. The parameters for comparison are: reflectance maximum/absorption maximum and inflection points, the latter corresponding to maxima or minima in the first derivative spectrum (Bacci, Magrini, Picollo, & Vervat, 2009).

Results and Discussion

Characterization of Pure Substances

The reflectance spectra of egg yolk, animal glue and linseed oil, as collected in the full range, are shown in Figure 2a, while the wavelengths of the most characteristic peaks of each binder are listed in Table 1. As color is not discriminating for their identification, the differences detected in the visible range are not discussed here, but the main dissimilarities for distinguishing different binders are searched and found in the region between 1200 and 2400 nm.

Binding media	Experimental Wavelength (nm)
Egg yolk	1211, 1441, 1507, 1727, 1762, 1942, 2058, 2177, 2309, 2347
Animal glue	878, 1182, 1447, 1506, 1730, 1946, 2049, 2177, 2286, 2347
Linseed oil	1206, 1441, 1727, 1754, 1932, 2132, 2304, 2347

Table 1 - Experimental wavelengths (nm) of three binding media. Each peak can be assigned to overtones and combinations bands of specific chemical bonds (Vagnini, 2009).

The most evident variances among the three binders are marked in Figure 2b, when the region in question is zoomed. Since egg yolk contains mainly albumins with low quantities of fats, its raw spectrum contains features of both lipidic and proteinaceous components. It is noticed that the differences among oil, egg yolk, and animal glue are well visible in three regions (1150-1250 nm, 1650-1800 nm, and 2250-2400 nm) (shadow areas in Figure 2b), where the reflectance peaks are assigned to overtones and combination bands of C-H bonds. Due to the presence of lipidic contents in egg yolk and linseed oil, the spectral features of these two binders are more comparable than that of animal glue. However, although the wavelength of the reflectance peaks in egg yolk and linseed oil are sometimes

very similar, their absorption intensity varies greatly, i.e. the same peaks in egg yolk are sharper and stronger than in linseed oil. For example, the first overtone of CH₂ stretching mode is observed at around 1730 nm for both these two binders, whereas the peak intensity is stronger for egg yolk than for linseed oil. On the other hand, the differences in the wavelength (and peak intensity) of the overtones and combination bands of C-H bonds between animal glue and the other two binders are more evident. For example, the second overtone of methylene stretching is located at about 1210 nm for egg yolk, while for animal glue it is shifted, being at around 1182 nm.

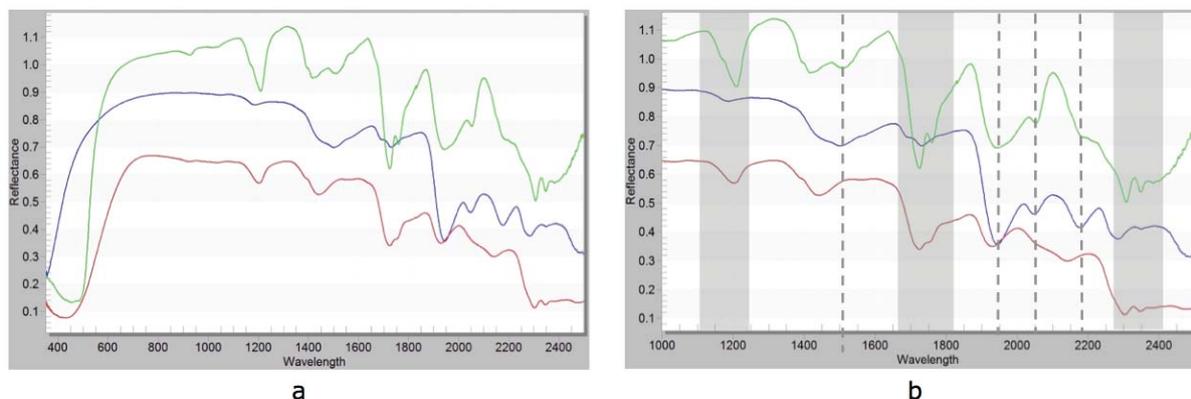


Figure 2 - Reflectance spectra of three binding media: in full spectral range (a) and in 1200-2400 nm region marked with the most evident variances (b). Green = egg oil; blue = animal glue; red = linseed oil.

Other variances marked with dashed lines are also essential for distinguishing these binders. At around 1507 nm, the first overtone band of N-H stretching mode is identified in both egg yolk and animal glue as a shoulder, which cannot be found in linseed oil. A similar circumstance occurs at 2177 nm where the first overtone band of carbonyl stretching mode (Amide I + Amide II) is observed. Linseed oil, however, has a combination band of C-O stretching and C-H stretching peak at about 2132 nm in the closer region as a shoulder. Another important characteristic signal of the two proteinaceous materials, found at about 2050 nm, is the combination band of N-H stretching and bending. All the peaks attributed to proteins are stronger in animal glue than in egg yolk; this may be in accordance with the fact that glue is completely composed of proteins, while egg yolk also contains oils. The last dashed line locates at around 1950 nm, where the three binders show a very strong peak, but it is discriminating only for linseed oil. In fact, the second overtone of the C=O stretching of esters in oils occurs at 1932 nm, whereas the combination band of O-H stretching and bending in proteins are seen at about 1945 nm.

As discussed above for pure binding media, the wavelength of the peaks or the spectral feature in well-defined spectral ranges can be considered discriminating elements for the identification of these binders even when they are mixed with pigments (i.e. in painted layers). However, shifts in wavelength in respect to pure materials are expected when a matrix with pigments is made. Possible shifts of peaks and changes in signal intensities will be discussed as follows.

Characterization of Binding Media in Mixtures

To better understand the influence caused by the mixing with pigments, spectra were collected on the easel painting mock-ups. Figure 3 shows the reflectance spectra of pure egg

yolk and its mixture with synthetic cinnabar in the 1200-2400 nm range. Since cinnabar has no absorption feature in the NIR-SWIR region, the spectrum of the painted surface is expected to be similar to that of pure egg yolk. Actually, egg yolk is well identified from its distinctive features, but three changes in respect to the pure egg spectrum are observed. One is the disappearing of the shoulder at 1507 nm. The second is the shift of the broad peak at about 1943 nm to 1922 nm, due to the combination of the O-H stretching and bending mode, along with the broadening of the peak itself. The last change occurs at about 2050 nm, where the combination band of N-H stretching and bending shifts to 20 nm longer.

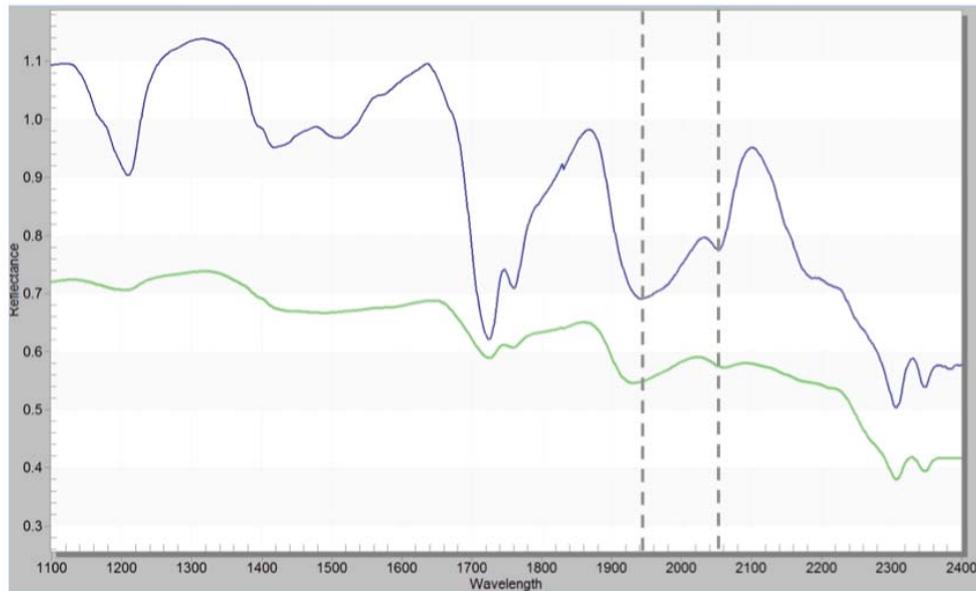


Figure 3 - Reflectance spectra of egg yolk mixed with cinnabar (green) in 1200-2400 nm region. Egg yolk (blue) is also reported for comparison purposes.

The same behaviors are also found when egg yolk is mixed with other pigments, regardless of the type and color of pigments, as well as whether the pigment has absorption peaks in these regions. An example is shown in Figure 4, when the two blue pigments (azurite and lapis lazuli) were mixed with egg yolk, azurite has some absorption peaks in the detected spectral range.

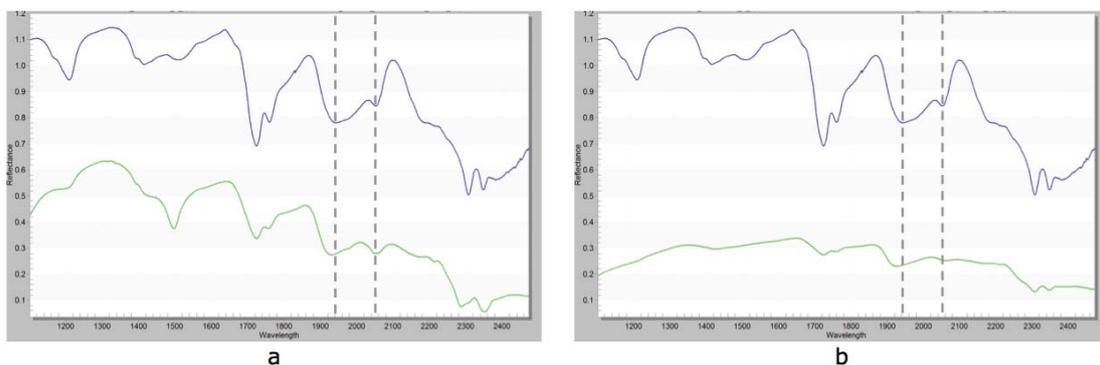


Figure 4 - Reflectance spectra in 1200-2400 nm region of egg yolk with azurite (green) (a) and lapis lazuli (green) (b). Egg yolk (blue) is also reported for comparison.

When these binders, i.e. egg yolk, animal glue and linseed oil, are mixed with the same pigment, their reflectance spectra often show features comparable to those of pure binders, and their identification is possible. Figure 5 illustrates the reflectance spectra in the 1200-2400 nm region of yellow ochre in the three different mixtures.

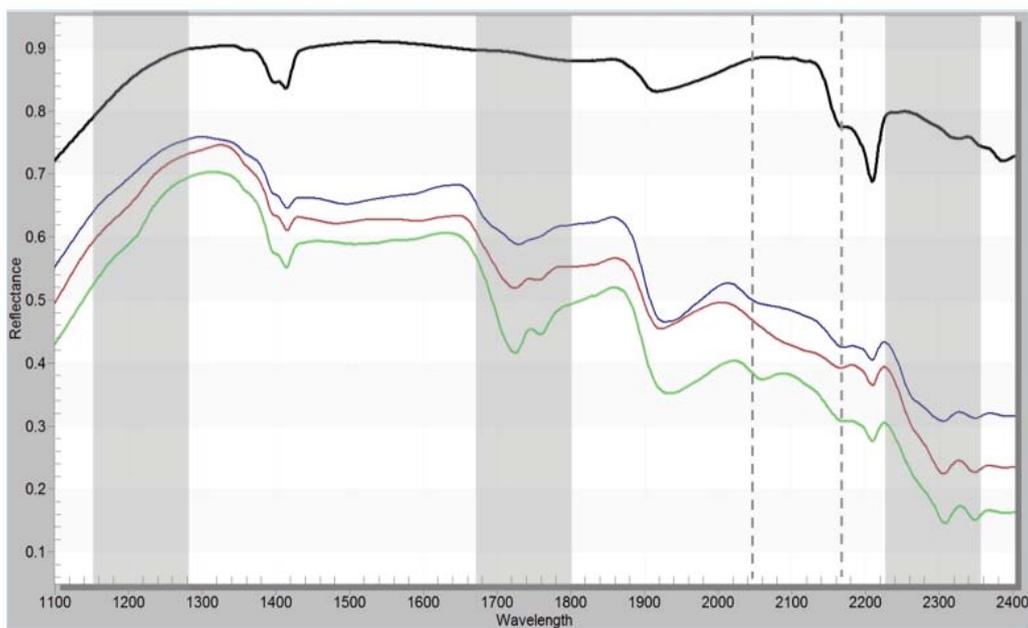


Figure 5 -Reflectance spectra of three binding media mixed with yellow ochre.

Black = pure yellow ochre; blue = yellow ochre mixed with animal glue;
red = yellow ochre mixed with linseed oil; green = yellow ochre mixed with egg yolk.

Similarly, the medium-weak peaks of animal glue and egg yolk around 1500 nm disappear in the spectra of the mixtures giving a similar spectral feature for all the three binding media. It is notable that the broad peak between 1930 and 1945 nm of these binders (i.e. 1942, 1946 and 1932 nm for egg yolk, animal glue and linseed oil, respectively) shifts for all binders to a shorter wavelength of about 15 nm, and linseed oil still shows the shortest wavelength. Another difference in mixtures between these spectra happens at around 2050 nm. Just like pure materials, yellow ochre with egg yolk has the same band at a longer wavelength, about 2070 nm, rather than 2045 nm, when mixed with animal glue. Whereas, the shoulder for linseed oil at around 2135 nm cannot be observed.

In fact, when pigments and binders have overlapping peaks, the identification of binders through the original reflectance spectra at some specific positions will be very hard. One example is the case of pigments containing O-H groups or/and H₂O molecules (e.g. azurite, malachite, yellow ochre). The peaks at about 1440 nm and 1945 nm, assigned to O-H overtone and combination bands, are no more discriminating. Hence, the peaks in the spectral range assigned to C-H bond vibrations are essential for the discrimination of these three organic materials (Figure 5), as discussed above.

Although the original reflectance spectra of pigment/binding medium mixtures can give us useful information for their identification, a more accurate differentiation can be achieved from their derivative spectra. Figure 6 shows the first derivative spectra of these three pure

binders. In these spectra the differences among the binders are increased and it is possible to take into consideration not only the wavelengths of maximum/minimum, but also the ratio of their intensity. Therefore, the discrimination of these organic materials is much improved.

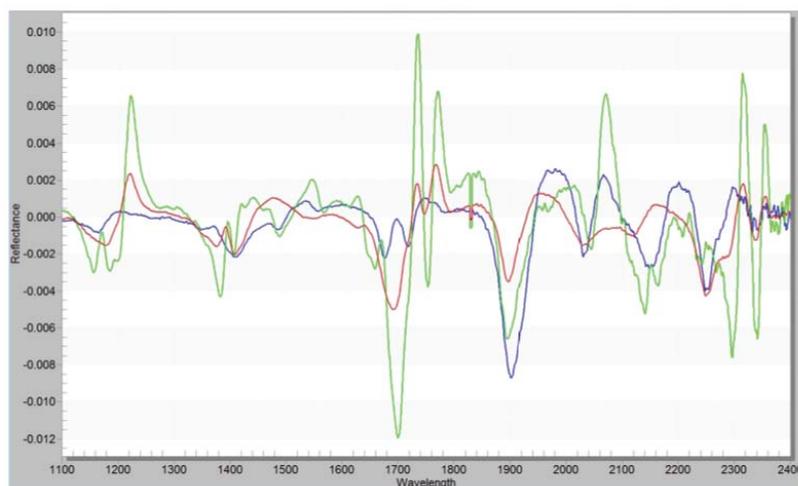


Figure 6 - First derivative spectra of three binding media in the full spectral range. Green = egg; blue = animal glue; red = linseed oil.

The most evident differentiations to identify egg yolk, linseed oil, and animal glue are located in two regions: from 1140 to 1260 nm, and from 1660 to 1800 nm (Figure 7). In the first region, animal glue has a negative medium peak around 1172 nm, while egg yolk has three ones, two negatives at 1190 and 1205 nm, and one strong and positive at 1222 nm. In the case of linseed oil, a similar peak at 1220 nm as in egg yolk is observed, but its intensity is much lower. The observation of the peak positions in the spectral range 1670-1725 nm gives evidence that egg yolk has a strong negative peak at about 1700 nm, while animal glue has a negative peak at 1670 nm, a positive one at 1700 nm, and another negative at 1725 nm (Figure 7b). Linseed oil has three negative peaks, where the one at the shortest wavelength (about 1670 nm) is the strongest. It is also well evident that the ratio of the reflectance at 1730 and 1775 nm differs for each binder. These two characteristic spectral ranges can also be used to discriminate the components in pigment/binder mixtures.

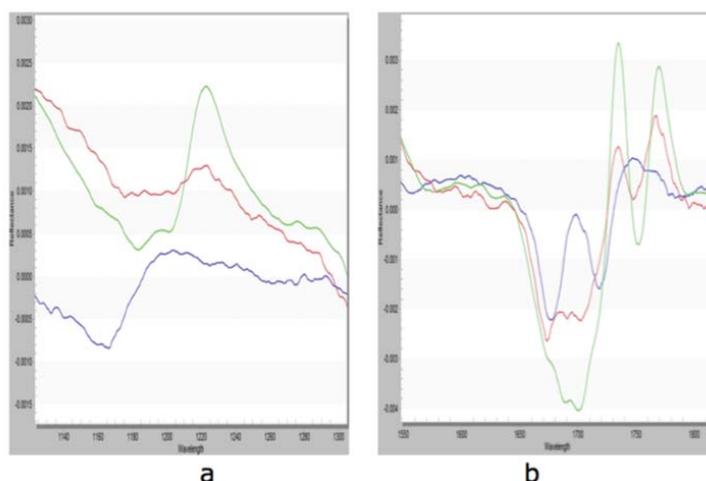


Figure 7 - First derivative of reflectance spectra of the three binders in the 1150-1250 (a) and 1670-1700 (b) nm spectral ranges. Blue = animal glue; Green = egg yolk; Red = linseed oil.

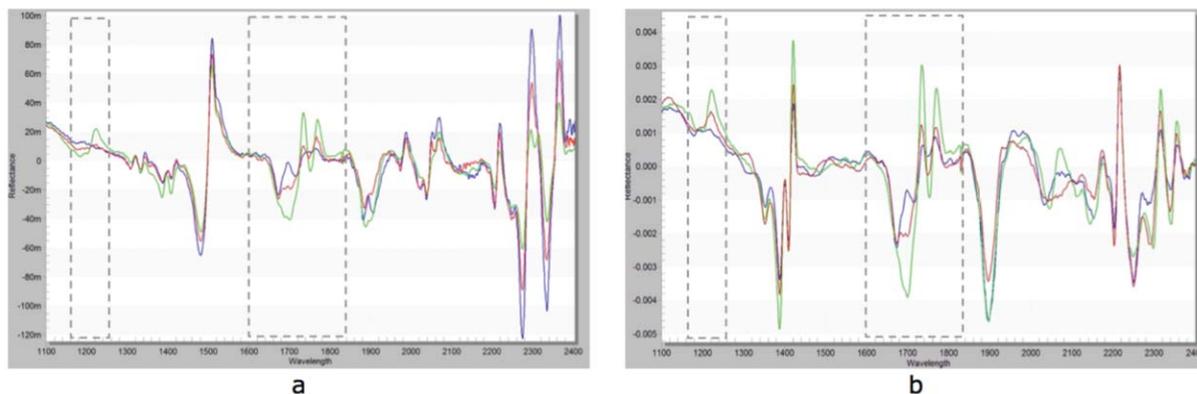


Figure 8 - Shows two examples of the application of the first derivative spectra to mixtures. In the examples, yellow ochre (Figure 8a) and azurite (Figure 8b) were mixed with the three binders. As expected, the two regions (dashed rectangular in the figures) are discriminating for the different binders.

Conclusion

It has been proved that the portable hyperspectral sensor is a powerful tool in identifying organic components, including the binding media, of easel paintings in a rapid and non-invasive way. Reflectance spectroscopy, particularly in the NIR-SWIR region, is useful for distinguishing egg yolk, animal glue, and linseed oil, both as pure material and mixed with pigments, just as in paintings. The interaction between pigments and binders makes the identification more difficult in some cases, but the unambiguous characterization can still be achieved by reprocessing the original spectra, i.e. through their first derivative. All the results obtained in this study show that this portable reflectance spectroscopy is valuable for the study of works of art in general, but also for conservation and restoration purposes. Indeed, in the case of conservation one possible advantage is the monitoring of the permanence or the degradation of a protective agent (e.g. the varnish), thus allowing a timely maintenance. While, in the case of restoration interventions, it makes possible a fast monitoring of the cleaning treatments which, contrary to that performed by UV lamps, allows the identification of the organic material discriminating the varnish (e.g. natural resins) from the binder of the pictorial layer (e.g. drying oil).

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