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A STUDY OF UV FLUORESCENCE EMISSION OF PAINTING MATERIALS

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Ever since the 1920s, paintings have been investigated under a UV light source to reveal the visible fluorescence emission of the various materials present on their outer layers. However, to date, the study of UV induced visible fluorescence emission of artworks it is still in a quite early stage. The emission results to be dependent on many factors: on the UV light source, on the pigment/dye, on the binding medium, on the varnish layer (if there), on their interaction, and on their aging. In order to form a first database with a significant number of entries, we analyzed about 220 samples of painting materials and acquired both their reflectance and fluorescence spectra in the visible range. The acquisitions have been performed on samples consisting of egg tempera or linseed oil pictorial layers, binding mediums and varnishes, spread on a gypsum and rabbit glue preparatory layer, previously deposited on a wooden substrate. The samples were prepared in 1994 by conservators of the Opificio delle Pietre Dure of Florence, Italy. Some of the acquired spectra have then been compared with

corresponding spectra of the same samples, as acquired in 1995, during a similar measurement campaign.

Introduction

In our opinion, UV induced visible fluorescence analysis still has unexploited potentials in several important topics in the field of study and conservation of artworks. It might in principle contribute to the characterisation of materials used by the painter, to the evaluation of the artwork conservation state, to the identification of retouches and previous restoration interventions, as well as to the control of possible conservation treatments. It has moreover the all-important characteristic in this field, of being a non-invasive technique, if the UV dosage is controlled and maintained within safe boundaries.

The acquisition of information about an object is obtained without coming into physical contact with it, but by exploiting the fact that the materials employed reflect, absorb and emit electromagnetic radiation in ways that depend on their molecular composition and shape. For more than hundred years, paintings have been examined under UV light. Traditionally, this diagnostic method is mainly used to observe the whole painting surface, reveal the presence of old varnish layers, and determine whether the fluorescent haze on it is even or not, where a lack of fluorescence may indicate retouched or newly repainted areas. However with further effort, UV induced visible fluorescence, by its own nature, could tell us more on the actual chemical composition and conservation of the painting's materials.

The acquisition of spectra of UV induced visible fluorescence on selected spots, is documented in literature [2], although a large database, to our knowledge has never been attempted. With building a database of the UV fluorescence emission of the most popular materials used through the years, we aim at providing a useful tool for adding information to the systematic studies which are performed on pigments, media and varnishes. Moreover we believe it could result useful for the comparison and understanding of the UV fluorescence imaging data observed under direct UV light analysis by the conservators and/or acquired with a multi-spectral imaging device.

1. UV Fluorescence basic principles

Fluorescence generally occurs when a fluorescent molecule (fluorophore) resonantly absorbs electromagnetic radiation that promotes it to an excited electronic state. Subsequent radiative relaxation of the excited states results in emission of light, where some of the excitation energy is lost through heat or vibration, and part is emitted at longer wavelengths, compared to the excitation radiation. Unlike in phosphorescence, where light is emitted only while the

radiation stimulus is present. For a given fluorophore, the . [1]fluorescence intensity is directly proportional to the intensity of the radiation received. Fluorophores can be identified and quantified on the basis of their excitation and emission properties. Different materials may exhibit different colours and intensities of fluorescence, while they look identical when observed in day-light conditions.

2. UV fluorescence applications for conservation: state of the art and opportunities

Due to the relatively short wavelength of the UV radiation, and therefore to the limited capacity of this radiation to penetrate, fluorescence emission of artworks depends on the contribution external layers, namely on the (semi)transparent varnishes, on the painted layers, i.e. the colouring agent, or pigment, and the binding medium, and on their chemical interactions. Traditionally, fluorescence photographs are mostly used to identify different varnishes and over-paintings. Occasionally, fluorescence colours are considered to give some indications of the pigments used, but are seen as a supplementary rather than a primary technique for identifying materials. Currently, professionals in the conservation area mostly acquire fluorescence images of paintings using one or more UV discharge lamps as illumination sources, a photo camera with a high sensitivity film as detector, and long exposure times [1]. However, this method only gives qualitative indications. It does not allow a spectral signature evaluation, nor a correct colorimetric measurement. Digital (multi-spectral) images promise a higher colour accuracy. In order to classify these images of the painted surface and discriminate the materials on them, it would be necessary to have a database of spectral and colorimetric characteristics of fluorescence emitted by materials commonly used by artists and conservators In this way it would be possible to replace with quantitative data the current empirical identification of materials through perceived colours of fluorescence.

3. Spectra acquisition methodology

Reflectance and fluorescence spectra were acquired in two different sessions but both using a SpectraScan PR704 (PhotoResearch, CA USA). This spectroradiometer works in the 380-780 nm range with 2 nm resolution. Two sets of lamps have been used to provide the correct

[1] For simplicity, hereafter we will use the word "fluorescence" as synonymous of "UV induced visible fluorescence".

illumination. The UVa radiation was supplied by two lamps consisting each of four UVa long-arc-pressure mercury fluorescence tubes (PLS-9 Philips, The Netherlands), placed in an aluminum box where the box window was covered with a filter meant to block visible light. As a matter of fact, the emission spectrum of these lamps covers also the visible and the near infrared spectral ranges. It is therefore better to use filters to cover the lamp reflector window to cut off the not-UVa radiation which could otherwise hinder the fluorescence radiation, which is typically a weak signal. The best results were obtained with the filter DUG11 (Schott, Germany). The DUG11 filter has a peak transmittance of about 0.7 at approx. 340 nm, FWHM of 70 nm, and transmittance of less than $5x10^{-6}$ in the spectral range between 400 nm and 800 nm (see Fig. 3). Moreover the SpectraScan was equipped with a filter, a KV418 (Schott, Germany), that cuts the radiation under 418 nm, in order to limit the residual lamp light from the fluorescence emission. The fluorescence spectra of the samples were acquired in a dark room in order to limit the influence of stray light. In order to remove the residual stray light reflection, on the fluorescence emission acquisition, an un-mixing correction was also performed "a posteriori", as described in section 5.

Both for the fluorescence and for the reflectance acquisitions, each lamp was placed, so to form an angle of 45° with the normal to the painting's surface in order to minimise the radiation specularly reflected by the surface and redirected towards the detector. The illumination source for the reflectance acquisition was an halogen lamp (Flexilux 3000, Schölly Fiberoptic GMBH). Also is this case the lamp's fibres were placed at 45°, symmetrically with respect to the sample, to illuminate the investigated area. The reflectance spectra were recorded in the 380-780nm range. A Spectralon ™ (Labsphere Inc, Great Britain) (a pressed $BaSO_4$ 99% diffuse reflectance target) was used as reference. The acquisition time of each single spectrum was of about a few hundred ms for the reflectance spectra and of about 60 sec for the fluorescence spectra. The acquisition area was about 1,5x1 cm^2.

4. The pictorial material samples

The specimens were prepared in 1994 by conservators of the Opificio delle Pietre Dure of Florence (OPD) and were stored since then in a dust-free place. The main purpose in making this sample collection, was to re-create materials and their combinations as found in painted works and/or confirmed by "historical" treatises. It was deemed important not to limit the research solely to "traditional" materials but also to include contemporary painting materials, as well as those most often used in restoration. The pictorial materials were applied to small

wood panels prepared with a traditional ground of gypsum and rabbit skin glue. The materials varied in order to include pure pigments layers, film forming materials and mixtures layers. Moreover the same pigments/dyes were prepared with two binding media: either egg-tempera or stand-oil. All the materials used in the preparation of the sample collection were previously tested at the Scientific Laboratory of the OPD using IR spectrophotometric analysis.

Materials and samples preparation scheme

Support: 30x200x15 mm wooden panels (plywood)

Preparation: rabbit skin glue dissolved in H_2O (1:16 ratio) and gilders gypsum added until the glue reached saturation.

Imprimitura: rabbit skin glue dissolved in H_2O (1:32 ratio).

Pigments & dyes: purchased as powders from Zecchi - Colori (Firenze).

Binding media

Egg tempera: 50% egg yolk, 25% egg white, 25% vinegar.

Linseed oil (stand oil) purchased from Zecchi - Colori (Firenze).

More about the sample collection is to be found in [4]. The samples we analysed are all those contained in Table 1 (where the chemical formula can be found), both with egg tempera and linseed oil as binding medium, plus the mixture of all pigments either with *Lead Tin White* and *Ivory Black* in egg tempera. Moreover, we acquired reflectance and fluorescence spectra of 40 film forming agents. Results on binders and varnishes are reported in fig. 8 and 9.

5. Correcting and un-mixing the spectra

We processed the observed spectra to separate the fluorescence emission from the component due to stray light, and to compensate for the attenuation of the KV418 filter placed in front of the spectroradiometer optics.

We basically needed to subtract, from the acquired spectra, the part of radiation that was not due to fluorescence emission but to the stray light reflection, and we have to divide this result by the transmission curve of the filter. This was realized according to the following equation:

$$UV_{fluo}(\lambda) = \frac{UV_{measured}(\lambda) - R(\lambda) \times UV_{ref}(\lambda)}{\tau_{KV418}(\lambda)} \qquad \text{eq. (1)}$$

where $UV_{fluo}(\lambda)$, $UV_{measured}(\lambda)$ and $UV_{ref}(\lambda)$ represent, respectively, the computed fluorescence emission spectra of the specimen, the spectra measured by the spectroradiometer focussed on it and acquired under UV illumination, the spectra measured by the spectroradiometer focussed on the reference standard under UV illumination and placed in the

same geometrical condition as for the sample. $\tau_{KV418}(\lambda)$ represents the transmission curve of the filter. The reflectance $R(\lambda)$ is given by:

$$R(\lambda) = \frac{W_{measured}(\lambda)}{W_{ref}(\lambda)} \qquad \text{eq. (2)}$$

where $W_{measured}(\lambda)$ and $W_{ref}(\lambda)$ represent the spectra obtained respectively from the specimen illuminated with white light, and from the reference standard, placed in the same geometrical conditions, and with white light shined on it. In fig.1 an example of the effect of the correction procedure is shown. The spectra have been smoothed with a 5 points FFT method.

6. Some intriguing comparisons

A thorough analysis of the reasons for the fluorescence emissions registered, is beyond the scope of this paper. Definitive answers on the evolution with age of fluorescence radiation, are also not to be expected among the results of this work. We would like here mainly to introduce, together with some preliminary results, some suggestions for future work.

Discriminating power of combined reflectance fluorescence spectral analysis

The most striking example is perhaps that of white pigments which show close reflectance curves both in egg tempera and linseed oil, but very different fluorescence activated spectra, see fig. 3. The same holds e.g. for *Madder Lake* and *Carmine*, see. fig. 2.

Analysis based on the chemical composition

One of the most popular pigment in traditional paintings, the mineral lazurite, or *Lapis lazuli*, is known to show fluorescence. During our survey we found the one prepared with lapis lazuli to be indeed among the most fluorescent samples. Both specimens prepared with egg tempera and linseed oil exhibited a peak emission in the blue region, respectively at 456 nm and about 480 nm. The artificial paint ultramarine, which has the same chemical composition as natural lapis lazuli, showed a similar spectrum, however the intensity was slightly less, and the peaks were at 444 nm for tempera and about 470 nm in oil. *Realgar* and *Orpiment* are also very similar in colour and chemical composition. They are both natural pigments which contain arsenium (**As**). This could also explain why the show the same shape of fluorescence emission spectra, but a different intensity. Similar considerations could be made for pigments containing cobalt (**Co**), like *Smalt*, *Cerulean blue* and *Cobalt blue* (see fig. 4b). The difference in emission intensity could be due to the oxidation number of respectively Al_2O_3 e

SnO_2. *Smalt* contains also K_2O, where potassium has the lowest oxidation among them. Transitional metal ions are notorious for their fluorescence quenching abilities, and actually most of the compounds containing copper (**Cu**), manganese (**Mn**), and titanium (**Ti**) like: *Natural Siena earth*, *Burnt Siena earth*, *Umber earth, Burnt umber earth*, *Azurite*, *Malachite*, *Verdigris* and *Titanium white* do exhibit a very low fluorescence signal or no fluorescence at all. In particular TiO_2 is known as ultraviolet-absorbing pigment with high fluorescence quenching properties. Pigments containing iron like *Yellow ochre* and *Mars yellow* both contain **Fe III** and have (see fig.5a) a similar behaviour. The spectra present peaks at about the same wavelengths, and an absorption peak at about 400 nm which seems to be characteristic of ochre, and in general of pigments containing iron. If we analyse the *Red ochre* spectrum, we observe that the peak at 550 nm is disappeared (see fig.5b). Also *Red ochre* contains **Fe III** but no **OH** group.

Changes with binding medium

When the medium used is different, in general there are changes in the reflectance exhibited by the specimens, however the largest differences are in the fluorescence emission. Most of the inorganic pigments/dyes do not exhibit much fluorescence as powders. While it is know that linseed oil, dammar and mastic are examples that not only yellow, like many organic materials with age, but also develop fluorescence (and in [2] we find that, since the yellowing implies absorption in blue range, also the fluorescence emission becomes generally more yellowish). We registered that in some cases, like for the *Orpiment* (fig. 6a), the main change seems to be in the intensity of the fluorescence emission. This is not, nevertheless, a uniform trend. For *Cobalt blue* and *Vermillion*, the shape of the fluorescence spectra changes significantly, retaining however some of the most prominent absorption and emission peaks. We could generally say that there is a tendency for the fluorescence emission peaks to increase in intensity and to shift the main peaks towards longer wavelengths (fig. 8). One of the oldest organic fluorescent pigments to be found is natural *Madder Lake*, which is based on alizarin (fig. 7) from the root of the perennial plant Rubia tinctorum. The curve of the fluorescence activated by 400 nm incident light has been published in literature [3] with a peak in the 580 nm region. The fluorescence emission that we registered from the specimen of madder lake paint in egg tempera, has two peaks, one around 420 nm and the other around 620 nm (see. fig. 6b). The fluorescence emission of the corresponding specimen of genuine madder lake paint in linseed oil, has also two peaks, where the first peak is shifted of about 20

nm to higher wavelength range. We believe that the first peak be actually linked to the binder, (and in fact a peak in the 420-440 nm is common for almost all specimens), while the second one, around 620 nm, would be the one typical of natural *Madder*.

Binding media analysis

Some of the most interesting results have been obtained for the binding media specimens and their comparisons with white pigments. Among the binders, the reflectance curves observed are very similar as far as main peaks are concerned, however they present varying intensity mainly in the blue-green range (see. fig 9a). As far as the fluorescence emission is concerned, *Cooked linseed oil*, *Poppy-seed oil*, *Black oil* and Copal resin present a perfect superposition in the 500-550 nm range, and share the 500 nm peak with *Egg yolk*. The specimens prepared with *Lean tempera* and *Arabic Gum*, present a less intense fluorescence and a very similar behaviour (see. fig 9).. *Stand-oil* is again different, and has a quite distinct curve. *Lead white* in egg tempera shows a striking similarity with *Egg yolk*, while *Lead white* in linseed oil has similarities in the 475-560 range with *Cooked linseed oil* and not with *Stand-oil* (see. fig 10).

Varnishes analysis

Comparing the varnishes spectra, we can observe two main groups of curves. *Egg white*, *Damar resin*, *Mastic* and *Sandrac* belong to the first group, while in the other group there are all the varnished realized mixing various compounds with *Stand-oil*, which is observed to have predominant influence. The only specimen which presents a fluorescence emission with a behaviour similar to *Stand-oil* is *Shellac* (see. fig 9).

1995 Measurement campaign

In 1995 a similar measurement campaign was conducted [5], however on a limited collection of samples. In particular only egg tempera data were investigated, and only pure pigment layers. However, it was still possible to compare the reflectance behaviours and fluorescence emissions of these materials and to observe the changes occurred over 9 years. The UV light source in 1995 was a set of 2 lamps, each composed by 4 Madzafluor TFWN fluorescent tubes 60 cm long placed in a aluminium case, without any additional filter, while for the reflectance measurements a fibre halogen lamp Schott Mod. KL 1500 Electronic was used. As detector the same SpectraScan photoradiometer was used, only with an additional filter, 690 Ealing, to strongly reduce the stray light emitted by the fluorescent tubes in the 680-780 nm range. The same acquisition procedure as that described for the more recent campaign was

followed. Observing the samples' data we gathered, we can conclude that the fluorescence emission increased in intensity both for low and high emission paints. In some cases also the spectrum shape changed considerably. We report the *Madder Lake* and *Carmine* spectra, (see fig. 2) which have, and had, similar reflectance spectra, thus could be mistaken when only observing the reflectance colour, but present definitively different fluorescence behaviours.

Conclusions

We presented the methodology and some of the results of the acquisition of reflectance and fluorescence spectra of about 220 specimens of egg tempera or linseed oil pictorial layers, binding mediums and varnishes. The samples were prepared in 1994 by conservators of the Opificio delle Pietre Dure of Florence, Italy.

Acknowledgments

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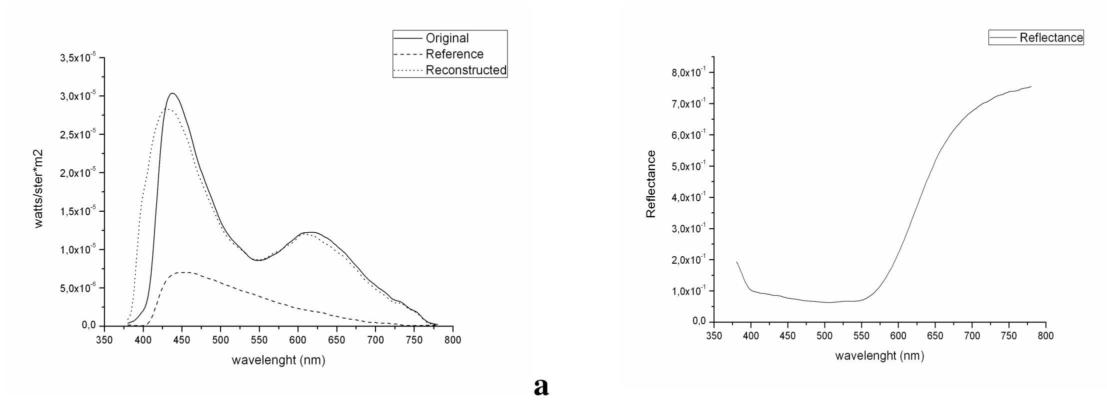


Fig. 1 An example of the result of the correction processing on the fluorescence emission of a specimen. The original acquired signal (a) is corrected, knowing the specimen reflectance (b) and the stray light reflected by the reference standard

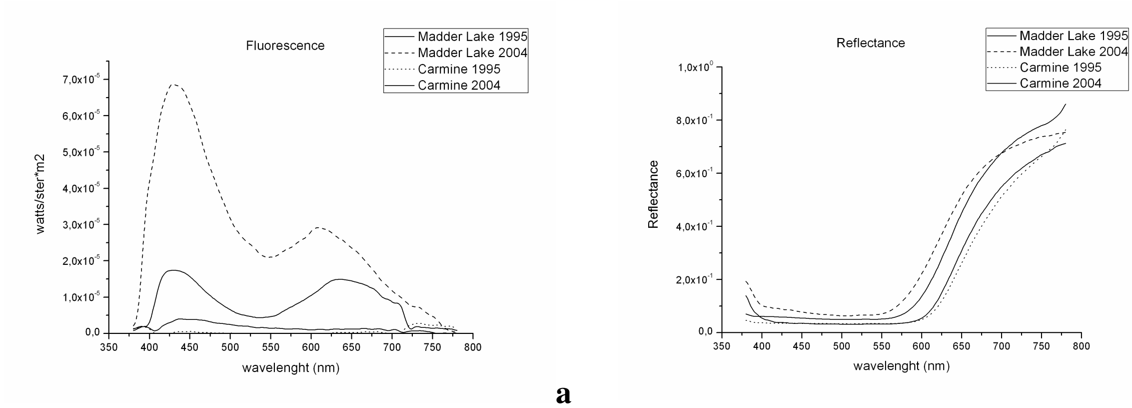


Fig 2 Fluorescence and reflectance spectra of Madder Lake and Carmine in egg tempera in 1995 and 2004

13m Linseed Oil	*14m Chalk CaCO3*	21m Indigo+Lapis Lazuli C16H10O2N2 + Na6-8Al6Si6O24S2-4	24m Azurite + Lead-tin Yellow 2CuCO3*Cu(OH)2 + Pb2SnO4	27m Indian Yellow C19H16O11Mg*5H2O	30m Burnt Umber Earth Fe2O3*nH2O+MnO2+(Al2O3*SiO2)*2H2O Fe:Mn=40:15
13l Egg Tempera	*14l Titanium White + Ivory Black [(Ca3(Po4)2]+CaCO3+C 10:2*	21l Lapis Lazuli + Carmine Na6-8Al6Si6O24S2-4 + C22H20O13	24l Indigo+Orpiment C16H10O2N2 + As2S3	27l Mars Yellow Fe2O3*H2O+Al2O3	30l Raw Umber Earth Fe2O3*nH2O+MnO2+(Al2O3*SiO2)*2H2O Fe:Mn=40:15
	14h Titanium White + Ivory Black [(Ca3(Po4)2]+CaCO3 +C 10:1	21h Cerulean Blue CoO*SnO2	24h Verdigris + Lead-tin Yellow	27h Yellow Ochre Fe(OH)3	30h Red Ochre Fe2O3
	14g Titanium White TiO2	21g Ultramarine Blue, artificial Na3Ca(Al3Si3O12)S	24g Veronese Green Cu3(AsO4)2*4H2O)	27g Row Sienna Earth Fe2O3*nH2O+MnO2+(Al2O3*SiO2)*2H2O Fe:Mn=60:1	30g Bole Al2O3*SiO2+Fe2O3
	14f Zinc White + Ivory Black [(Ca3(Po4)2]+CaCO3 +C 10:2	21f Prussian Blue Fe4(Fe(CN)6)3	24f Viridian Cr2O3*2H2O	27f Realgar As2S2	30f Burnt Sienna Earth Fe2O3*nH2O+MnO2+(Al2O3*SiO2)*2H2O Fe:Mn=60:1
	14e Zinc White + Ivory Black [(Ca3(Po4)2]+CaCO3 +C 10:1	21e Deep Cobalt Blue CoAl2O4	24e Chromium Oxide Green Cr2O3	27e Lead-tin Yellow Pb2SnO4/PbSn2SiO7	30e Madder C14H8O4 & C14H8O5
	14d Zinc White ZnO	21d Smalt SiO2,K2O,Al2O3, CoO	24d Light Cobalt Green CoO+ZnO	27d Orpimente As2S3	30d Carmine C22H20O13
	14c Lead White + Ivory Black [(Ca3(Po4)2]+CaCO3 +C 10:2	21c Lapis Lazuli Na3Ca(Al3Si3O12)S	24c Verdigris Cu(CH3COO)2*H2O	27c Cadmium Yellow CdS	30c Red Lead Pb3O4
	14b Lead White + Ivory Black [(Ca3(Po4)2]+CaCO3 +C 10:1	21b Indigo C16H10O2N2	24b Green Earth Fe(II),Mg,K,Al2O3*SiO2	27b Litharge PbO	30b Vermilion HgS
	14a Lead White PbCO3*Pb(OH)2	21a Azurite 2CuCO3*Cu(OH)2	24a Malachite CuCo3*Cu(OH)2	27a Naples Yellow Pb3(SbO4)	30a Cadmium Red CdS(Se)

Table 1 Name and chemical composition of the pigments examined

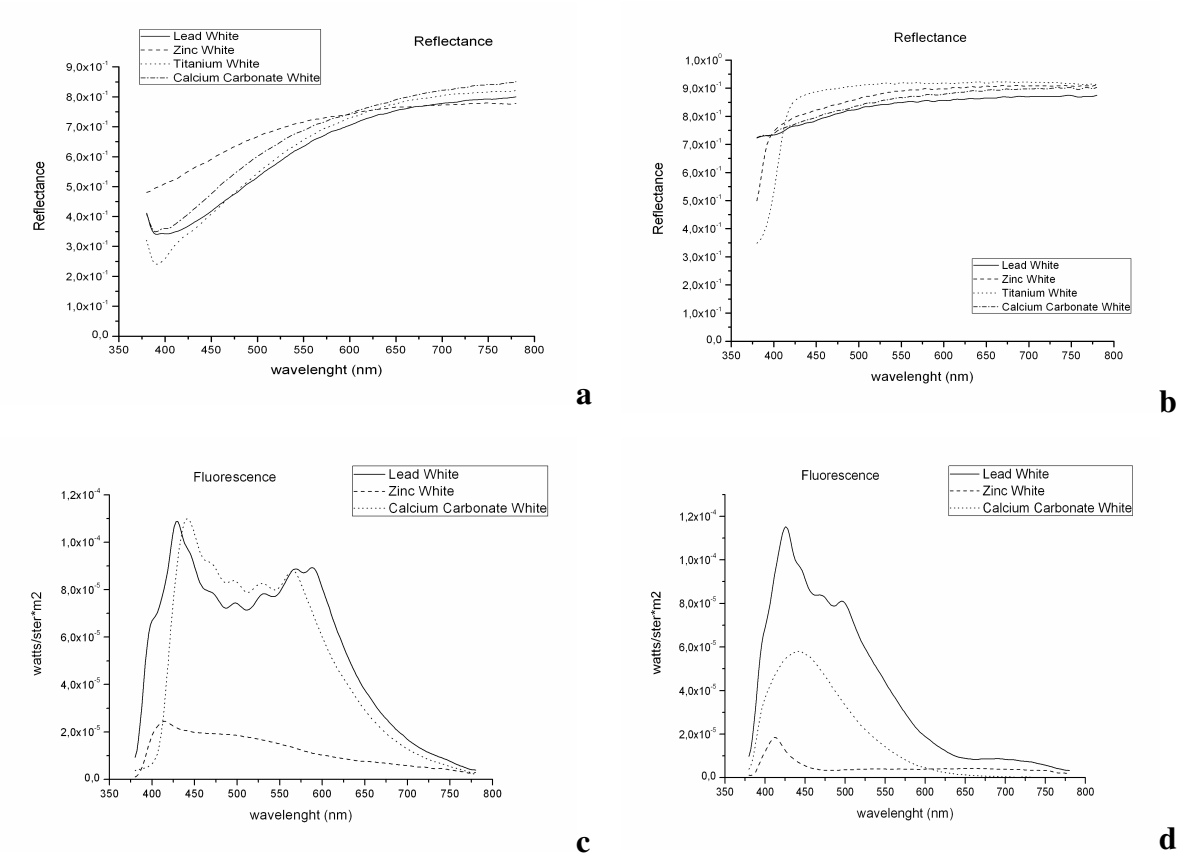
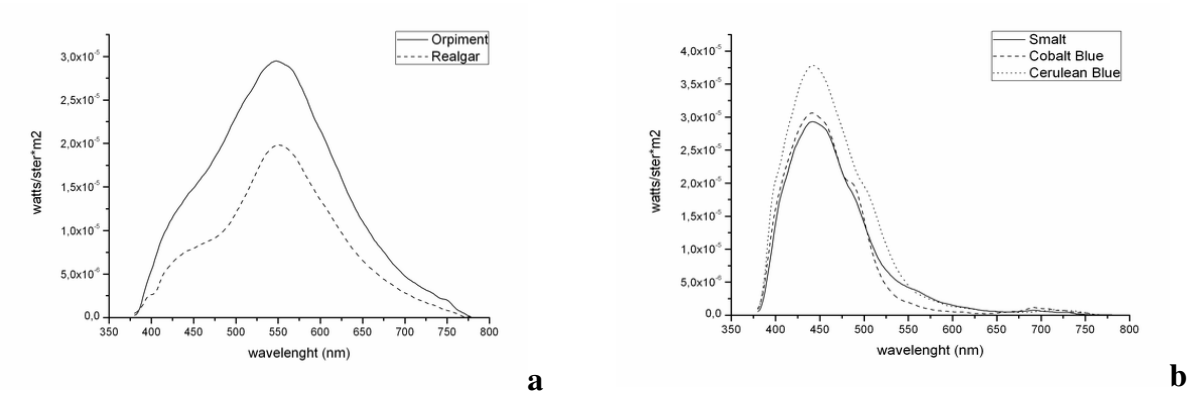


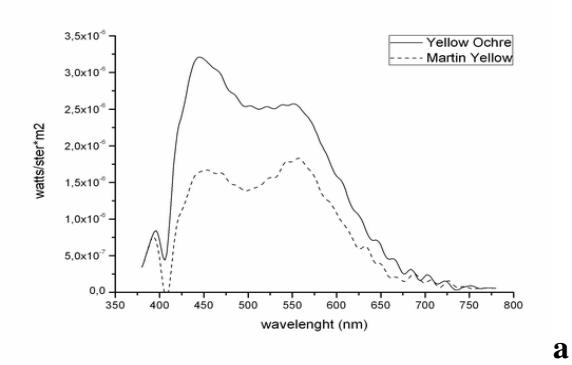
Fig. 3 (a) and (b) Reflectance spectra of white pigments in egg tempera and linseed oil, and corresponding fluorescence curves



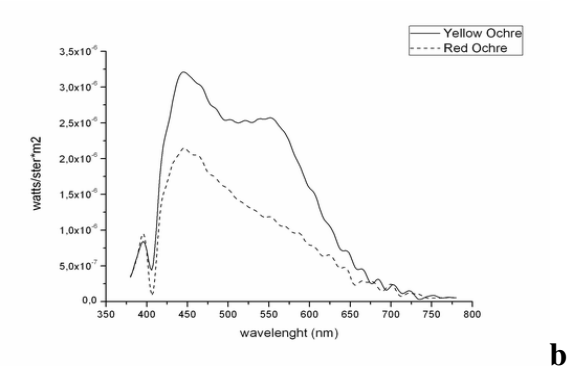
		Peaks	
Pigment		**nm**	**W/sr*nm*m^2**
Orpimeno	As2S3	548	2,95E-05
Realgar	As2S5	550	1,98E-05

		Peaks	
Pigment		**nm**	**W/sr*nm*m^2**
Smalt	CoO*(SiO2, K2O, AlO3)	442	2,93E-05
Cobalt blue	CoO*Al2O3	442	3,06E-05
Cerulean blue	CoO*SnO2	442	3,78E-05

Fig 4 (a) Fluorescence spectra of Orpiment and Realgar, with relative peaks data. (b) fluorescence spectra of Smalt, Cobalt blue and Cerulean blue, with corresponding peaks data



a

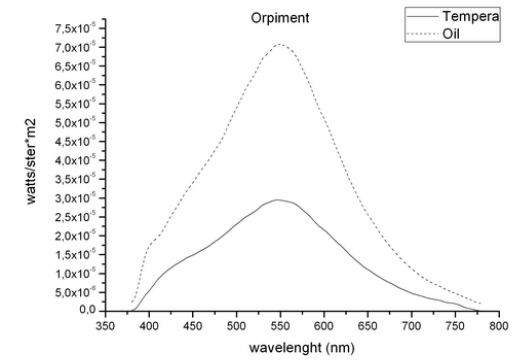


b

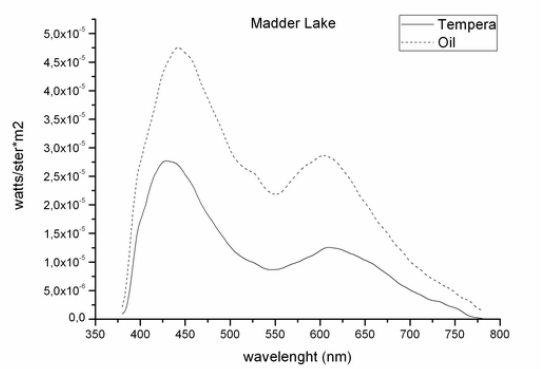
Peaks			
Pigment		nm	W/nm*m^2*sr
Yellow ochre	Fe(OH)3	446	3,21E-06
		552	2,57E-06
Mars Yellow	Fe2O3*H2O+ AlO3	456	1,67E-06
		558	1,83E-06

Peaks			
Pigment		nm	W/nm*m^2*sr
Yellow ochre	Fe(OH)3	446	3,21E-06
		552	2,57E-06
Red ochre	Fe2O3	446	2,15E-06

Fig. 5 Comparison of fluorescence spectra of specimens containing Fe (III)



a



b

Fig. 6 Comparison of fluorescence spectra of the same pigment in egg tempera and oil

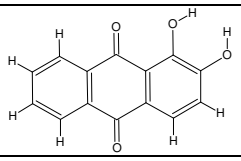
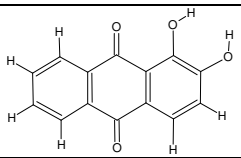
Alizirin $C_{14}H_8O_4$	1,2-diidrossi-9,10-antrachinone
2D structure	
Purporin $C_{14}H_8O_5$	1,2,4-triidrossi-9,10-antrachinone
2D structure	

Fig 7 2D structure of alizarin and pupurin molecules, contained in genuine Madder Lake

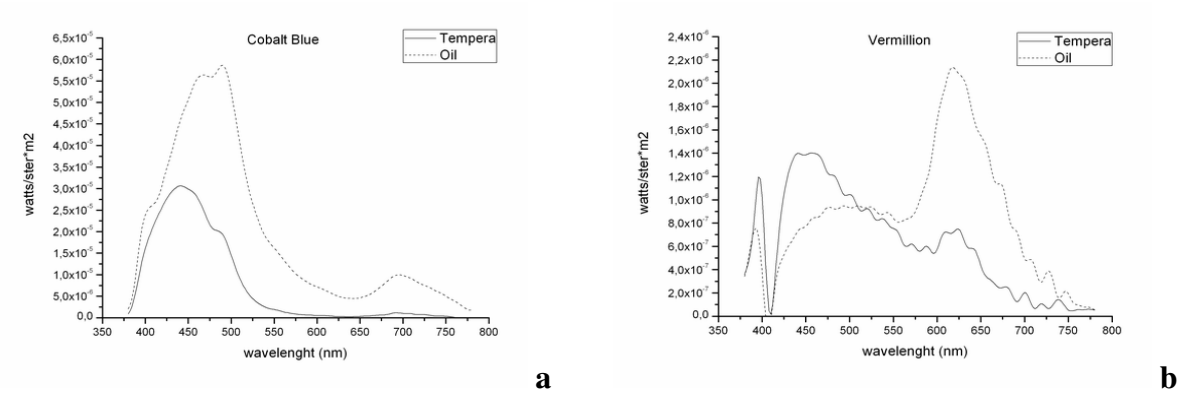


Fig 8 Comparison of fluorescence spectra of the same pigment in egg tempera and oil

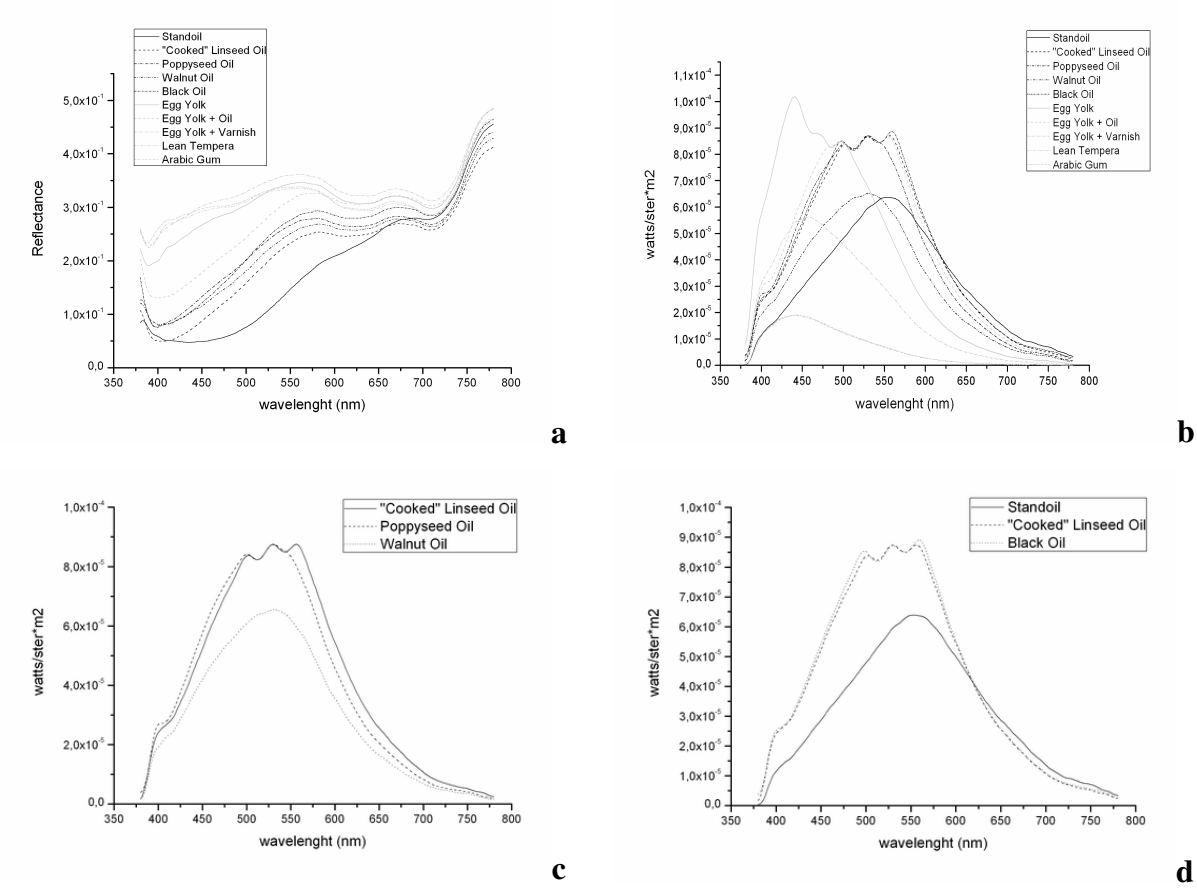


Fig 9 Reflectance and fluorescence spectra of binding media specimens

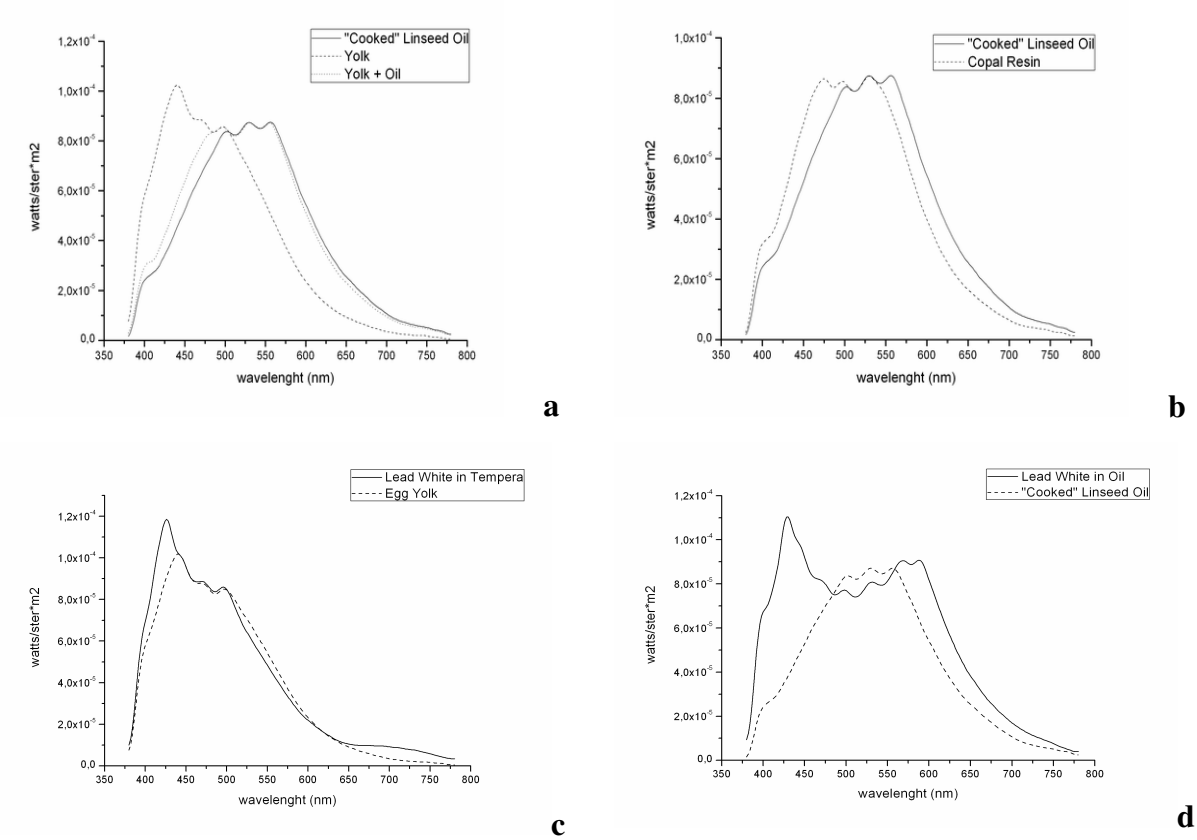


Fig 10 Comparison of fluorescence spectra of binding media and *Lead white* pigment

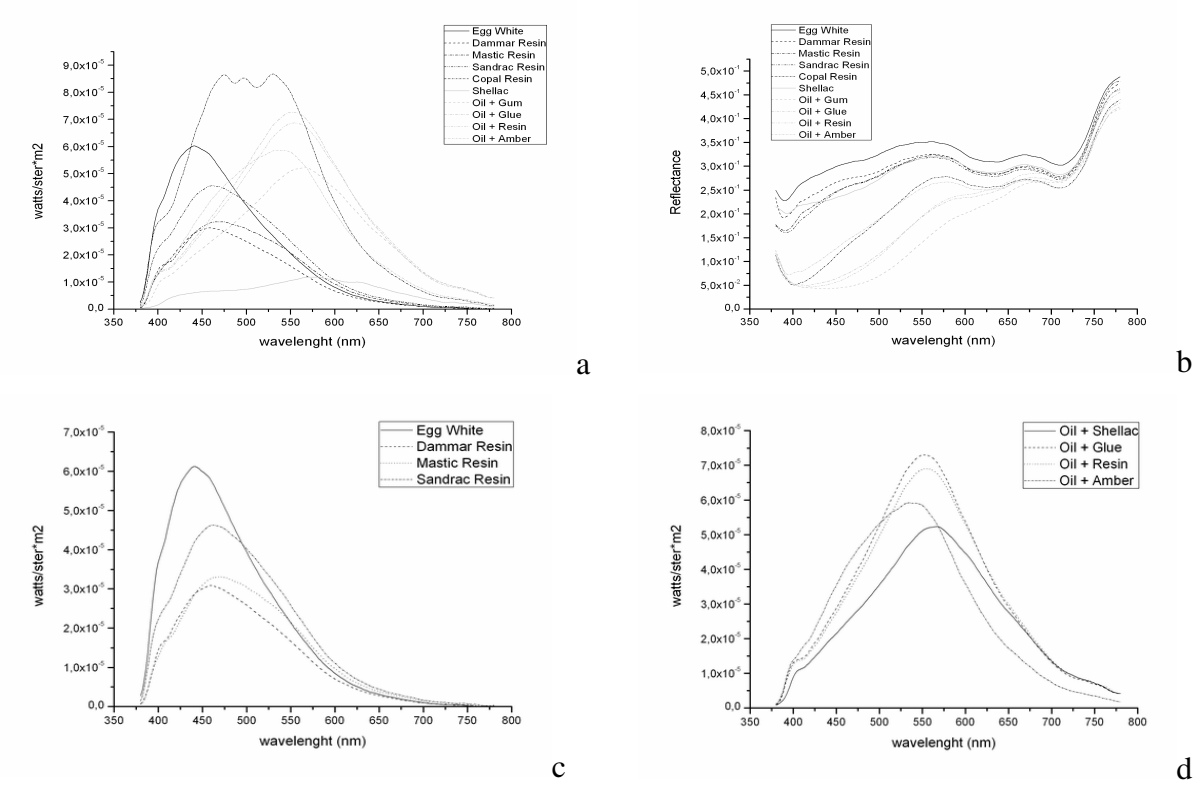


Fig 11 Reflectance (b) and fluorescence (a) spectra of varnish specimens

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