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On the use of photobleaching to reduce fluorescence background in Raman spectroscopy to improve the reliability of pigment identification on painted textiles•

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Subjecting a specimen of red lead watercolour paint on silk to photobleaching was demonstrated to be a simple means by which to engineer a reduction in the magnitude of the fluorescent background that was approximately exponential with time, with a corresponding improvement in the signal to noise ratio of the Raman spectrum, thus rendering the characteristic peaks more easily visible and allowing more confident identification of the pigment. However, relative heights of the Raman peaks obtained from the sample were seen to alter progressively as a result of irradiation, indicating that some component of the sample was undergoing degradation that may result in longer-term damage to a fragile historic artefact.

It was also shown that crystals of the lead monoxide pigment massicot were present in the samples of red lead on a painted silk artefact dating from 1750. It is concluded that this was either due to deliberate mixing of pigments by the artist, contrary to historic records, or as a result of the roasting techniques used to create red lead pigments at the time and not due to thermal degradation of the pigment during Raman analysis.

KEYWORDS: pigments; photobleaching; degradation; painted textiles.

INTRODUCTION

Raman microspectroscopy has long enjoyed a reputation as the technique of choice for the non-destructive analysis of pigments on a wide range of historic and archaeological artefacts including the analysis of pigments on manuscripts,[1–15] polychromes,[16,17] icons,[18] Roman intaglios,[19] wall paintings, murals and frescoes,[20–31] wall coverings,[32,33] postage stamps,[34] funerary artefacts,[35] and fine art.[36–38] We have already reported its use in pigment identification on painted textiles;[39] however, these artefacts often give spectra in which the characteristic peaks are masked by a large fluorescent background, preventing unambiguous identification of pigments in some cases. Extended photobleaching has been suggested as a means by which such fluorescent background might be reduced.[3] The question arises as to whether this technique is both effective and safe for use on fragile historic artefacts such as painted textiles.

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BACKGROUND

Paint techniques have been used on a wide range of textiles such as upholstery and furnishing fabrics, costumes for theatre and normal wear, trades union banners, religious and military colours, tapestry cartoons and stained cloths (cheap imitations of tapestries) or even painted areas used to fill in missing areas of damaged tapestries[39] over time. Raman spectroscopy is valuable to textile historians as a non-destructive technique for pigment identification on painted textiles as it increases scholarship through enhanced knowledge of the original materials used, thereby elucidating the original appearance of an artefact, especially if fading has degraded the pigment. It may also enable authentication of artefacts by identifying the presence of a synthetic pigment whose first date of manufacture is known or provide evidence for an artefact's provenance. For textile conservators, pigment identification may help determine the appropriate restoration or cleaning processes for the artefacts and identify later additions that should be removed if it is decided to return an artefact to its original appearance.[40] Moreover, developing an overall picture of the deterioration process is facilitated through knowledge of the pigments employed and the dates of

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the textile. If these are identified, better predictions could be made about future degradation pathways of textiles, and treatments could be better developed to accommodate these.[40] Finally, health and safety considerations dictate that conservators handling fragments from friable samples during conservation treatments must take appropriate precautions should artefacts contain toxic materials such as lead, arsenic and mercury, as was common in early pigments.[39]

The large background fluorescence often seen on Raman spectra from painted textiles is typically considered to be due to surface contamination (soiling) or due to the organic binders used in painting the textiles, though it may be an inherent aspect of aged samples[41] and may be indicative of the age of the sample as has been found in certain glass artefacts.[42] Many other factors, including storage conditions, may also be significant in determining the magnitude of the fluorescence. Photobleaching has been reported as a means by which background fluorescence can be reduced in Raman spectroscopy[3] while laser ablation of a surface varnish film has been shown to reduce fluorescence on fine art by removing the overlying varnish layer[43] though this latter technique cannot be considered to be non-destructive and may be inappropriate for more fragile artefacts.

This work therefore has two aims:

(1) To undertake a systematic study of the effect of photobleaching on the magnitude of the background fluorescence and the quality of the spectrum obtained, as defined by the signal to noise ratio.
(2) To consider whether the process of photobleaching can be considered to be entirely benign when applied to fragile historic artefacts.

We are unaware of any study that has sought to consider the appropriateness of the photobleaching technique in the context of fragile historic textiles.

EXPERIMENTAL

Figure 1 shows part of the textile sample taken from the Karen Finch Reference Collection at the Textile Conservation Centre, Winchester. It originally formed part of a painted silk chair cover indicated as belonging to the Doge Paolo Renier around 1750.[44] Preliminary microscopic analysis indicated that there was a single paint layer with no ground or varnish over the paint and no size on the fabric to prepare the surface. The pigment appeared to be suspended in water-soluble egg tempera (it is a watercolour). A small (approx 1 cm^2) sample

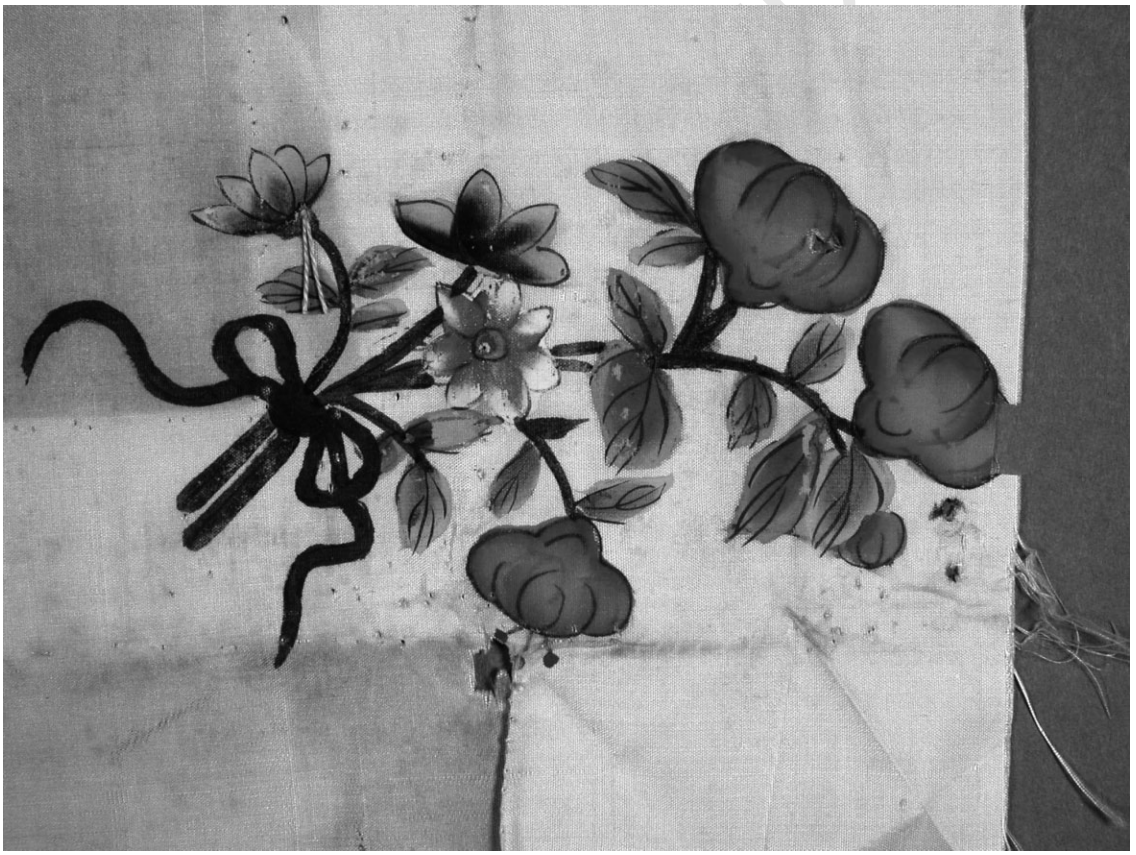

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Figure 1. Image of flowers from the painted Chinese silk chair covering, courtesy of the Karen Finch Reference Collection at the Textile Conservation Centre, Winchester.

was removed for analysis. No further sample preparation was undertaken.

Raman spectroscopy was applied to a highly fluorescent red lead pigment sample on a painted textile. The experimental system used incorporated a Leica microscope with 50× lens coupled to a Renishaw Raman RM1000 spectrometer with a 785-nm diode laser of maximum laser power 25 mW, providing a spot size of 1 μm^2 at the sample. The instrument has a Peltier cooled charge-coupled device (CCD) detector. The Raman spectrometer was set up in confocal mode as per the manufacturer's recommendations to reduce the background fluorescence from underlying substrate layers.

An initial Raman spectrum of the red lead was taken of ten accumulations of 30 s each. The magnitude of the fluorescence was such that a laser power of 25% power (6 mW) was chosen in order to prevent saturation of the detector. The same point on the sample was then photobleached for 1 h and a spectrum taken under conditions identical to the first. The process was repeated twice with irradiation periods of 0.5 h each time followed by spectra collection in each case to ascertain the effect of repeated photobleaching. red lead was chosen as it is known to be stable under irradiation with longer wavelengths.[45]

RESULTS AND DISCUSSION

Figure 2 shows a typical series of spectra obtained from a particular point on the red lead sample at various times during the photobleaching process. For the spectra shown, photobleaching was undertaken at 100% power and no offset was applied to the ordinate axis in this presentation. The original spectrum, (uppermost trace) exhibits considerable background fluorescence. The next lower trace indicates clearly that 1 h photobleaching at 100% power removed the rising background such that the peaks are seen against a flat background at 250 000 counts. The lowest two traces, after half an hour's additional photobleaching each, demonstrate that continued irradiation lowers the level of the background.

Analysis of the rate of reduction of the background level showed that the decrease was approximately exponential with time. The precise rate of decline was seen to be dependent upon the power at the sample; thus a similar reduction can be achieved on a more fragile sample if a lower power is used for a longer time. A similar exponential decrease of background fluorescence has been reported in a study of extended photobleaching on fluorescent samples of polyethylene.[41]

Further analysis of the signal to noise ratio of the spectrum showed an approximately 10-fold improvement in signal to noise after 2 h photobleaching at 100%, with the result that many more of the smaller peaks became visible.

Mathematical techniques have been proposed to remove the fluorescent background; however, such techniques do not improve the signal to noise ratio and therefore only make larger peaks more easily visible against the background and do not render smaller peaks more easily discernable against the inherent noise in the system.

Conversely, taking a spectrum over 2 h will improve the signal to noise ratio but will not remove the fluorescent background and, therefore, the small peaks will still not be

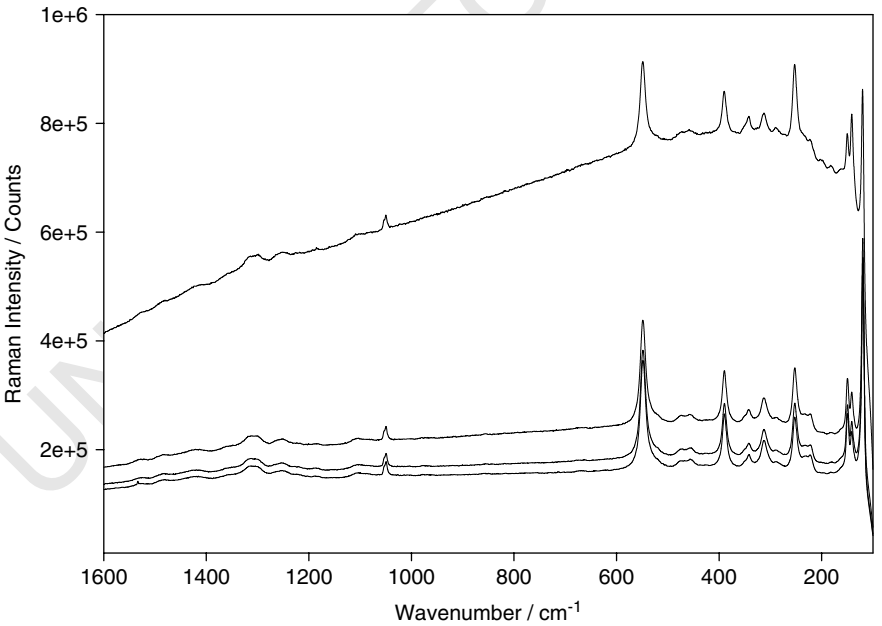

Figure 2. Raman spectra of red lead. The uppermost trace indicates the initial highly fluorescent spectrum. The next lower trace was obtained after 1 h irradiation at 100% power (25 mW). The lowest two traces were obtained after an additional 0.5 h irradiation each. No offset has been applied to these spectra.

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seen because of the *y*-axis scaling required to accommodate the fluorescent background.

Thus we would conclude, initially, that such photobleaching may be the ideal means by which the magnitude of the background fluorescence may be reduced and the signal to noise ratio improved, rendering the maximum number of characteristic peaks visible, thus enabling positive identification of an unknown pigment.

However, on closer inspection it is apparent that the relative peak heights of some peaks altered during the process. The peak heights were calculated from the absolute peak height above the local background. Consideration of the peak areas indicated that there was no broadening of the peaks; thus alteration of relative peak heights can be considered to be indicative of changes to the sample.

The heights of the peaks at 149 cm^{-1} and 548 cm^{-1} relative to the height of the 122 cm^{-1} peak above local background were unaffected. These peaks are characteristic of red lead. However, the heights of the peaks at 141 cm^{-1} and 252 cm^{-1} decreased significantly during the photobleaching process. The precise origin of these badly affected peaks is unknown. Comparison with a spectrum of silk indicated that they do not arise from the substrate. It is possible that they arise from the soiling now present on the textile – possibly the same soiling that is responsible for the background fluorescence – or are characteristic of the particular paint formulation used – the other additives included in the paint mix or the binder. Whatever their origin, the changing relative peaks heights could indicate that extended laser irradiation is changing some component of the artefact as a whole.

Thus it would seem that, although photobleaching has been considered not to have any effect on many pigments or materials, it may cause changes to other components within the artefact. In a study of the thermal degradation of paper-based artefacts resulting from the laser irradiation technique commonly used to clean such artefacts[46] the conclusion reached was that although visible and IR radiation do not induce direct photolysis of the cellulose, there may be photosensitised degradation induced by non-cellulosic components of the samples (metal ions, lignins). Similarly, thermal degradation of some natural fibrous materials including linen and wool has been seen resulting from irradiation with 1064-nm radiation after they have been artificially soiled,[47] which might imply that the irradiation promotes interaction between the fibres and the soiling to the detriment of the artefact.

Thus it is not unreasonable to suggest that the changes observed in the relative peak heights from this artefact as a result of extended photobleaching in this study may be indicative of changes to some component of the artefact that may lead to detrimental interaction between the artefact and the paint or surface soiling, which may, subsequently, result in longer-term degradation of the artefact.

As such, we are led to conclude that the benefits of photobleaching to remove background fluorescence and facilitate pigment identification may be far outweighed by the potential longer-term damage caused to these valuable, fragile artefacts.

It has been reported that laser irradiation during Raman spectroscopy of pigments can result in thermal degradation of the pigment red lead, dilead(II)lead(IV)oxide (Pb_3O_4), to litharge, tetragonal lead (II) oxide and massicot, orthorhombic lead (II) oxide (PbO).[1,45,48] However, the thermal decomposition of red lead into the yellow lead monoxide massicot

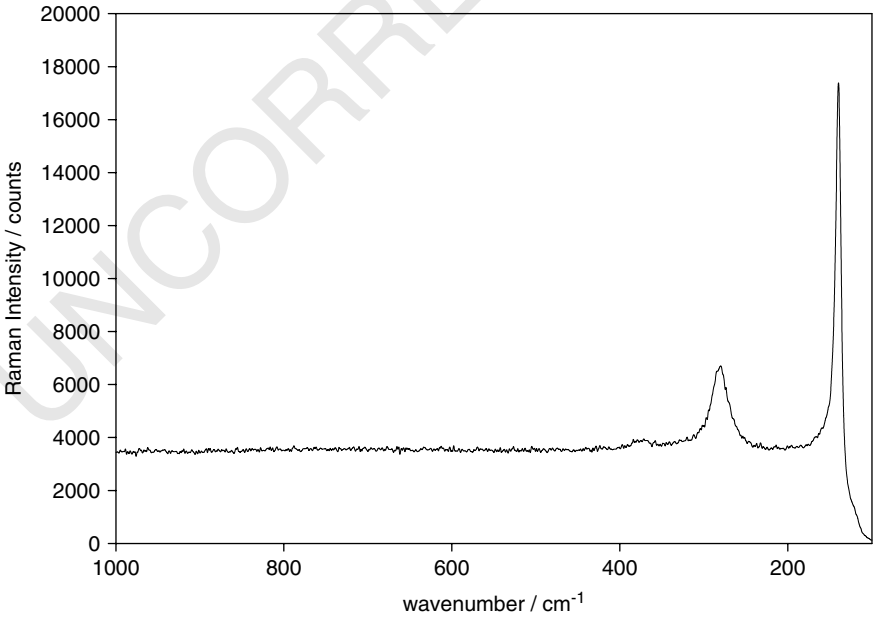

Figure 3. Raman spectrum of massicot obtained from red flower showing peaks at 143,289 and 385 cm^{-1}.

was demonstrated only at high temperatures while laser irradiation of red lead with a 1064-nm wavelength Nd : YAG laser with two pulses of duration of 30 ns and a fluence of approximately 300 mJ cm^{-2} did not result in the formation of the bright yellow lead monoxide pigment. Instead, there was a blackening of the surface that was attributed to the reduction of red lead to either $Pb0_2$ (quoted as lead suboxide) or the formation of metallic lead.[48] In addition, red lead has been shown to be stable under irradiation at laser wavelengths greater than 600 nm but susceptible to change when irradiated at wavelengths corresponding to its absorption maximum between 400–600 nm with immediate decomposition to massicot, demonstrated clearly when irradiated with laser wavelengths of 514 and 488 nm.[45]

Throughout this study, there were occasions where a spectrum such as that shown in Fig. 3 was obtained, which resembled that of the yellow PbO pigment massicot. Further careful visual observation of the red lead pigment sample indicated that there was a range of crystal sizes and, indeed, large yellow or brown crystals were seen mixed into a more homogeneous red matrix. By choosing to focus on these crystals the massicot spectrum could be obtained, whereas selecting other areas where such crystals were not visible resulted in a red lead spectrum. This would lead to one of two conclusions: either Chinese artists of this period were using lead monoxide pigments mixed in with other lead-based pigments to achieve particular paint effects, or the production of red lead by roasting lead white ($2PbCO_3.Pb(OH)_2$) resulted in the formation of crystals of massicot as has been shown elsewhere.[49] If the artists working on painted textiles in 1750s were indeed using a mix of pigments to achieve particular colour effects, this would indicate that they were using paint techniques closer to those of artists working in fine art rather than the techniques employed by illuminators of manuscripts where typically only a single pigment is reported for each colour when such artefacts are subjected to scientific analysis.

CONCLUSION

We have shown that photobleaching can reduce the overall fluorescent background level inherent in many pigments on painted textiles and increase the signal to noise ratio in the resulting spectrum. However, the changes of relative peak heights observed here throughout the photobleaching process indicate that extended irradiation may initiate physical or chemical changes in some component of the sample, though not necessarily in the pigment under scrutiny, which would be unacceptable in the context of fragile artefacts such as historic textiles. This is in agreement with the work on the damage caused by laser irradiation during laser cleaning of artefacts published elsewhere and indicates that the total irradiation of a sample should be minimised to prevent long-term damage. However, the occasional spectrum indicating the presence of a lead monoxide pigment such as litharge or massicot during Raman spectroscopic analysis was not found to be due to decomposition resulting from laser irradiation, as red lead is stable under the longer wavelength used. Instead, we would suggest that the original pigment included massicot particles among the red lead.

This may have been deliberate in order to create a certain shade or may have been a result of the common technique of roasting of lead white to create red lead.

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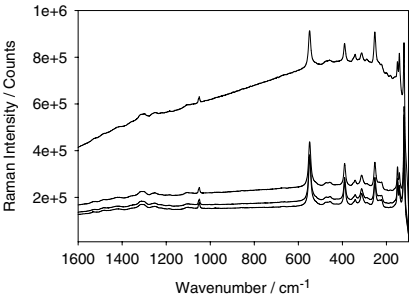
Extended photobleaching was shown to facilitate pigment identification by reducing the background fluorescence and increasing the signal to noise ratio of the spectrum. However, degradation of the artefact was shown to occur as a result of the photobleaching, though not to the extent of decomposing the pigment red lead into massicot, as has been reported elsewhere.



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