Investigating the Validity of Microfading Spectroscopy to Predict Photochemically Induced Color Change at Lower Light Levels

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Abstract

Two investigations of the validity of microfading spectroscopy to predict the fading behavior of a diversity of colorants at lower light levels is discussed. The specific research question being: what is the probability that a particular sample being tested with micro-fading will alter significantly differently from the same lux-hours light exposure at ambient light intensities? In one experiment two ISO Blue Wool Standards and 15 dyed papers were tested. Accelerated light aging at four illuminance levels stepping from 250 lux to one tenth of the microfading irradiance of 12.5 Mlux was conducted over different time periods using either standard fadometer lightfastness testing apparatus or a microfadometer. Samples received similar lux-hours exposure. In a second experiment a 2.2 Mlux illuminance from a microfadometer was compared to that of a QUV Weatherometer light aging chamber (with UV filtration). Ten different dyes were each faded for 10 minutes using the microfadometer and then for 21 hours using a QUV Weatherometer (with UV filtration). Samples again received the same lux-hours exposure. Results from both experiments illustrate a positive correlation between the compared light sensitivity testing methods, leading to the conclusion that fugitive colorants can be reliably highlighted by the microfading technique. In both experiments a lower value of induced color difference was observed when using microfading compared to standard lightfastness testing apparatus (light box aging) indicating that the quantative prediction of color change from real illumination in lower illuminance conditions is not secure. A short discussion of the origins of error in the technique follows.

Introduction

A microfadometer functions as a metrological instrument which measures photoinduced color change and is used to test the lightfastness of objects [1,2,3]. This happens through direct high intensity illumination of the material on a sub-millimeter scale where spectral measurements are carried out simultaneously. Within a small illuminated area, fading continues only to a certain level that is not discernible by the viewer, making it possible to directly test an object's light sensitivity. As such it has been used by major cultural heritage institutions worldwide to access the light sensitivity of their collections.

When considering why microfading measurements are typically made, most are not conducted to shed light on the degradation pathway, however numerous published works exists for this end along with many other applications of the technique for research purposes [4].

When analyzing results from the technique there are typically similar qualitative or quantitative outcomes which if achieved can be considered a successful use of the method:

Outcome A: Questions if the object under test is light sensitive.
Outcome B: Differentiates the degree of light sensitivity relative to that of ISO Blue Wool Standards [5].
Outcome C: Predicts accurately quantitative color change in low illuminance conditions.

To achieve outcome C, the time required to reach a degree of color change must be inversely proportional to the rate of incident photons on the material. This is the principle of reciprocity or the Bunsen-Roscoe law [6]. Reciprocity failure occurs when a coefficient of proportionality changes with variation in intensity. This is dealt with empirically by Schwarzschild's law [7].
There are numerous reasons that can cause reciprocity failure. For example there are certain photochemical reactions that do not follow the reciprocity principle, and are proportional to the square root of the intensity [8]. It has also been found that Alizarin Crimson and some other colorants are affected differently when exposed to a light/dark cycle rather than to continuous exposure [9]. When reviewing the literature approximately two-thirds of materials obey the reciprocity principle when photodegradation is concerned [10] although the range of intensity over which the testing has occurred is at the limit of that used in microfading.

Other concerns associated with microfading techniques may include: potential biphotonic events, diffusion-limited photo-oxidation reaction rates, dehydration and heating of the sample, the choice of different color difference units used resulting in confusing outcomes and comparisons, the variability of using and measuring ISO Blue Wool Standards with such a small measurement area, variation in spectral power distribution (SPD) between lamps used in object display and the lamp used in accelerated light aging, human and instrumental error in making spectral measurements, the small sampling area, the required number of samples to obtain quality data, the length of fading time, and the potential interaction of individual chemical components of the object tested.

All accelerated tests by their nature involve extrapolation which ideally requires justification from detailed physical or chemical knowledge of the effect of the accelerated variable on the degradation mechanism. This is rarely available or cannot be simply or practically determined [11]. In the face of this a significant strength of microfading is the possibility to bypass the need to understand the degradation mechanism.

With all the complexity it could be argued that a simple comparisons of microfading results with more accepted methods of accelerated light aging is helpful. This was the major motivation for this research.

Materials and Methods

Experiment 1

Fifteen dyes and ISO Blue Wool Standards #1 and #2 from the GCI reference collection were faded at different illuminance levels; 250 lux, 1.25 klux, 12.5 klux, 1.25 Mlux and 12.5 Mlux and exposure times were adjusted to provide the samples with the same exposure of approximately 2.1 Mlux-hours (however the exposure at 250 lux was terminated after 280 days at approximately 4/5th of required exposure). Light levels were controlled by the use of neutral density filters. This exposure regime is summarized in Table 1.

Table 1: A summary of light levels and exposure times for the samples.

Instrumentation	Light level	Fade duration
Light aging chamber	250 lux	280 days
Light aging chamber	1.25 klux	70 days
Light aging chamber	12.5 klux	7 days
Microfadometer	1.25 Mlux	100 minutes
Microfadometer	12.5 Mlux	10 minutes

The 17 samples selected from the GCI reference collection were both modern and traditional, and were chosen for the diversity of chemistry, color and typical applied purpose. All colorants were dissolved in deionised water and dyed onto Whatman filter paper. See Table 2 for a summary.

Table 2: 17 samples from the GCI reference collection with the name of the supplier.

Sample	Supplier
ISO Blue Wool #1	University Products, MA
ISO Blue Wool #2	University Products, MA
Rose Madder	Conservation Materials, NV
Extract of Fustic	Kremer Pigments, Germany
Extract of Logwood	Kremer Pigments, Germany
Cibacron Yellow	Kremer Pigments, Germany
Alizarin Yellow GG	Kremer Pigments, Germany
Fast Green	Kremer Pigments, Germany
Rhodamine B	Kremer Pigments, Germany
Lac Dye (Kremer)	Kremer Pigments, Germany
Tartrazine E102	Kremer Pigments, Germany
Coomassie® Blue	R Matheson Coleman and Bell
Crystal Violet A.C.S	Sigma-Aldrich
Carmine	Verfmolen, The Netherlands
Ultramarine	Winsor and Newton, London
Lac Dye (Zecchi)	Zecchi, Florence, Italy
Cochineal Red Dye	Zecchi, Florence, Italy

SOLUX MR16 true daylight lamps (D50 simulation) were used in the light aging chamber. The spectral power distribution (SPD) approximately matched that of the microfadometer. The relative SPD of light source incident on the samples measured using a calibrated Spectrascan 670 spectrometer from Photoresearch (reflected off a Spectralon white target) is shown in Figure 1 in comparison. SPDs were matched as equal luminance levels can have significantly different results due to radiometric differences of the light sources.

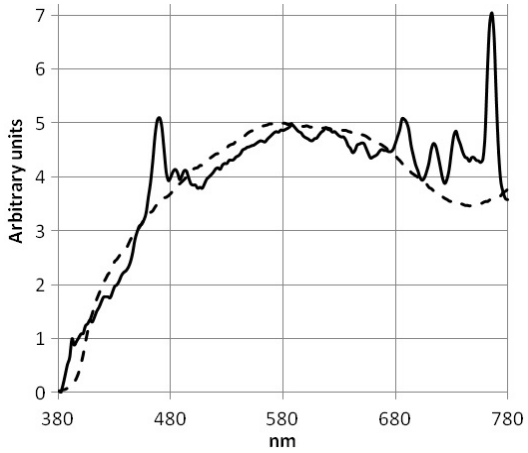


Figure 1. *The relative SPDs of SOLUX MR16 true daylight lamp (shown with a dashed line) and the Xenon source of the microfading spectrometer (shown as a solid line).*

Either a Spectrodensitometer from Xrite or the microfading apparatus was used to measure the color difference between the faded and not faded sections of the samples exposed. The monitored spectra were converted into CIE 1976 L* a* b* color space and calculated for the 2° standard observer under CIE standard illuminant D65 to provide a calculation of color difference of the fading area. The unit of color difference is represented by ΔE_{oo} [12,13].

Replicate sampling of the surface when microfading at 12.5 Mlux (5 replicates) was conducted due to small inhomogeneity of the sample surface (and therefore the degree of fading). Repeat measurements when using standard lightfastness testing apparatus were also made, but were shown to be unnecessary as results were repeatable to $\pm 0.2\ \Delta E_{oo}$.

Experiment 2

In a second experiment a comparison was made between the 2.2 Mlux illuminance from a microfadometer to that of a QUV Weatherometer light aging chamber (Xenon bulb with UV filtration). Samples received a total exposure of 0.36 Mlux-hours. Ten different dyes supplied by Dr. PH Martin (two drops of dye with 5 ml distilled water on Whatman filter paper resulted in a visually uniform distribution of color) were each faded once for 10 minutes using the microfadometer and then for 21 hours using a QUV Weatherometer. The samples received the same lux-hours exposure.

Samples were microfaded once. Color measurements were made with the QUV Weatherometer before and after exposure as in Experiment 1 and were repeatable within $\pm 0.25\ \Delta E_{oo}$.

Results

Experiment 1

Color differences after exposure are summarized in Table 3, as are the average results and the standard deviation [14] of the five results made using the Microfadometer at 12.5 Mlux.

Table 3: Results after exposure and the average and standard deviation of 5 results using the Microfadometer at 12.5Mlux.

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Sample	Light Aging Chamber / ΔE_{00}			Microfadometer / ΔE_{00}		
	250 lux	1.25 klux	12.5 klux	1.25 Mlux	12.5 Mlux (Av.)	St. Dev.
ISO Blue Wool #1	13.8	18.5	14.4	5.6	3	0.8
ISO Blue Wool #2	4.2	8.4	6.1	5.2	2	1.4
Rose Madder	6	8	7.2	2.3	2.5	0.1
Extract of Fustic	6.1	9.7	0.4	0.9	0.8	0.3
Extract of Logwood	7.3	11.3	7.4	8	9.7	1.2
Cibacron Yellow	1.2	0.5	0.3	1	1.2	0.1
Alizarin Yellow GG	0.4	2.6	0.8	0.3	1.2	0.9
Fast Green	8.4	9.5	10.3	2.6	5.4	0.8
Rhodamine B	23.9	26.9	23.3	16.3	13	2
Lac Dye (Kremer)	2.5	3.7	1.6	2.4	1	0.5
Tartrazine E102	1.3	2.3	0.6	0.6	0.6	0.6
Coomassie® Blue	25.1	27.2	16.2	10.5	8	1
Crystal Violet A.C.S	24.5	26.2	22.4	12	11.7	0.5
Carmine	7.4	9.8	7.4	3.6	2.6	0.3
Ultramarine	16.1	19.4	16.7	11.8	8.4	0.9
Lac Dye (Zecchi)	1.4	4.3	3.2	1.4	0.6	0.4
Cochineal Red Dye	10.2	12.6	6.8	6	2.7	0.6

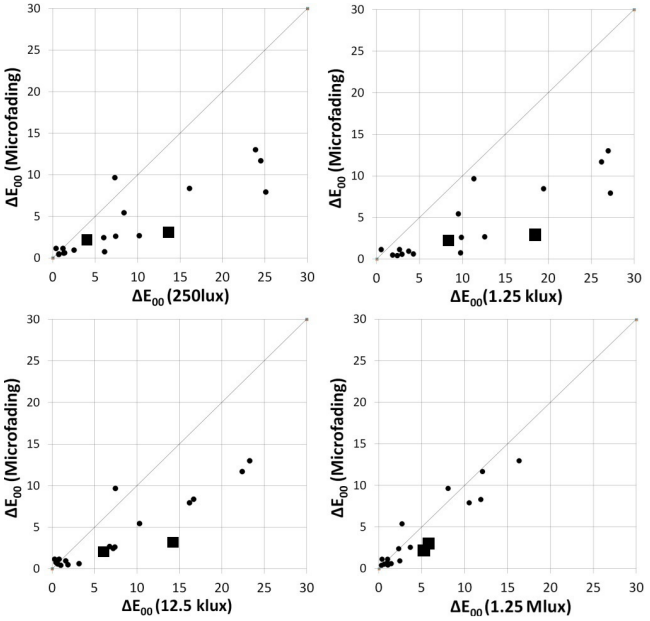


Figure 2. *Color differences of the sample set immediately after microfading are shown on the y axis of all subplots, compared to the x axis of all subplots which show results for 250 lux aging for 280 days (upper left), and 1.25 klux light box aging for 70 days (upper right), 12.5 klux light box aging for 7 days (lower left) and 1.25 Mlux microfading over 100 minutes (lower right). Blue Wool #1 and #2 are represented by the larger squares.*

The differing illuminance used in the two methods of accelerated aging did not fundamentally change the ranking between various sensitive materials (see Figure 2). In observing this correlation the microfadometer does not induce a new regime of fading that differs fundamentally from that in light aging chambers at lower illuminance levels. A new regime would result in a much reduced or no correlation.

Notably, Extract of Fustic failed to fall into a positive correlation and only faded to a noticeable degree at illuminances of 12.5 klux and below.

Importantly a correlation of equal color difference exists between all results for the standard lightfastness testing apparatus at all illuminance levels employed and another such correlation exists between the microfaded dyes at the two illuminance levels applied. See Figure 2 (lower right subplot) and Figure 3 below. The approximate one to one relationship between the different illuminance levels is lost when the accelerated aging technique is altered from standard lightfastness testing apparatus to microfading, indicating a systematic failure of the microfading technique to achieve Objective C in this instance.

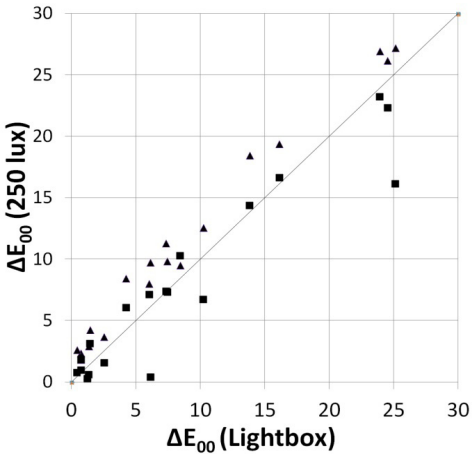


Figure 3. *Color differences for the sample set faded using the standard fadometer lightfastness testing apparatus at 1.25 klux for 70 days (diamonds) and 12.5 klux light box aging for 7 days (squares) compared to that achieved with 250 lux light box aging (y axis).*

Experiment 2

For the second experiment, see Table 4 below for a comparison of results

Table 4: Color difference after exposure of the sample set.

Dye (Product Code)	ΔE_{00} (Light box)	ΔE_{00} (Microfading)
Scarlet (5a)	2	0.4
Juniper Green (12a)	4.7	6.8
Moss Green (24b)	8.6	3.4
Persimmon (3a)	5.8	4.5
Wild Rose (19b)	15.2	10.5
Amber Yellow (16b)	2	0.8
Daffodil Yellow (15b)	4.2	1.3
Turquoise Blue (8a)	3	1.2
Grass Green (11a)	6	4.7
Moss Rose (7a)	18.8	12.5

In Figure 2 the results of microfading are shown on the y axis, and standard lightfastness testing apparatus aging on the x axis. A correlation exists between the two fading regimes (as shown by the solid line produced using the least-squares method), but the same lux-hours total exposure did not result in a similar color difference (results would follow the dashed line if this were the case).

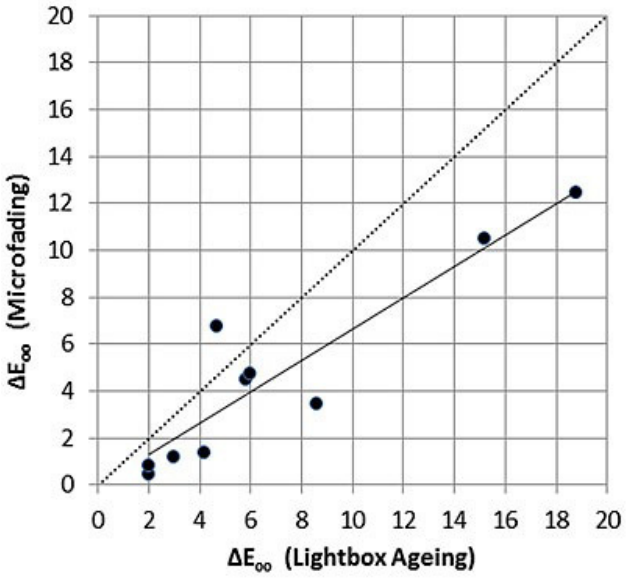


Figure 2. *Color differences of the sample set immediately after microfading are shown on the y axis, compared to* a QUV Weatherometer light aging chamber (Xenon bulb with UV filtration) on *the x axis*.

Conclusions

Results from both experiments yield similar conclusions. With respect to Outcome A (asking if the object under test is light sensitive), a correlation exists between results at lower illuminance levels and those found with microfading. Fugitive colorants are highlighted in each case. The exception was in Experiment 1 where Extract of Fustic was not highlighted as fugitive.

When using the same results to achieve Outcome B (asking to differentiate the degree of light sensitivity relative to that of ISO Blue Wool Standards), ISO Bluewool #1 did not always fall into a positive correlation. The substrate on which ISO Bluewools are dyed is wool and antagonistic reactions take place depending on the irradiating wavelengths. The resultant color change of the irradiated wool fabric is a function of relative strengths of the concurrent UV induced photoyellowing and the visible photobleaching processes during irradiation and will not be linear to total exposure [14], and are highly sensitive to incident SPD variation [16,17]. A textile structure combined with a very small sampling area can lead to problems with obtaining a repeatable geometry in measurement and as a result a variation in measured color [18]. Furthermore the choice of color difference unit (for example ΔE_{oo} or ΔE^*_{ab}) will sometimes considerably alter the conclusion when differentiating the degree of light sensitivity relative to that of ISO Blue Wool Standards.

The ability of the microfadometer system to rank the dyes in terms of lightfastness was shown to perform well. For example in Experiment 2 only Moss Green (24b) and Juniper Green (12a) were out of order.

Microfading underestimated the degree of color change for the same exposure under lower illuminance levels, so further work is required before accurate prediction of quantitative color change in low illuminance conditions is possible. The proportional offset observed could be explained as a systematic error from incorrect calculation of the irradiance and is thought most probable. The exposure of the sample prior to making the initial color measurement at the beginning of the microfading test may also contribute. Empirically determining the offset of each microfading instrument compared to real change in low light levels could be a method to predict quantitative color change in low illuminance conditions.

The source of measurement error in microfading is a combination of typical sources of uncertainty in any color measurement. The degree is dependent on the instrument, color and sample. Large, abrupt changes in reflectance factor have the greatest uncertainty in the calculated values [19] and materials can display thermochromism, inhomogeneity, angular and/or polarization dependence. Color measurements made using a microfadometer are not always accurate or precise by industrial standards but this is not an issue to highlight fugitive colorants. It is possible the two color measurement methods resulted in some disparity, although the large degree of difference observed makes this an unlikely explanation of the disparity between the two accelerated aging methods.

A commonly cited concern of microfading (due to the high light levels employed) is that of two photon absorption (2PA) involving the step-wise or simultaneous interaction of two photons that would not take place at lower illuminance. For a nonmonochromatic Xenon light source (often employed to initiate 2PA), two photon energies must be half the corresponding single photon absorption (1PA) and the sum of those energies would also need to be within the "photochemical window" that exists from approximately 200 nm to 800 nm for an electronic transition of an outer electron to occur. Infrared radiation in microfading is typically filtered and can be considered limited, however a number of reactions corresponding to an equivalent UV initiated 1PA could take place from simultaneous 2PA in the visible range. Secondary photolysis when photons irradiating a photolyte are absorbed by a photoreactive excited state (known as excited state absorption (or ESA)) is also a concern. Extending the assumptions of Schaeffer [20] (made for flash photography) the likelihood in microfading rises to 1 in 10 to 1 in 81000 (for an excitation time of 10^{-5} to 10^{-7} seconds respectively). This result indicates for a typical

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10 minute fade approximately 100 2PA events would take place [21]. It is however necessary to factor the limiting role of the different absorption and activation spectrum of the sample and the chemically different species created by the electron in the excited state. Only photon energies corresponding appropriately to the band gap are of concern and a significant reduction in likelihood results.

At faster accelerated rates it is also possible that photo-oxidation reactions may be limited by oxygen diffusion. The photo-initiated oxidative degradation pathways of pigments and dyes naturally rely upon oxygen to occur [22]. A reduction in access to oxygen can be dependent on particle density, particle size, concentration and permeability of binders and/or coatings, or other stratigraphic contributions. Oxidative degradation research at low oxygen levels has been investigated leading to a diverse set of results [23,24,25,26,27]. This leads to the conclusion that some colorants will have a reduced degree of induced color change with any reduction in oxygen. Others require oxygen levels equal to or significantly less than parts per million to see a reduction in the degree of oxidative degradation. Very few pigments and dyes may fade more with a reduction in oxygen.

Accelerated aging tests can raise or alter the temperature of the samples and alter the rate of chemical reaction. High temperatures in accelerated aging can also lower samples equilibrium moisture content [28]. Different reactions are thought possible compared to those in natural aging for the same material [29,30]**.** Although heating can create excited states it is ineffective for the excitation of the organic molecules and only accelerates the degradation pathways. The Boltzmann distribution law indicates that heating typically reported for microfading of 3°C to 60°C would make a negligible difference to electronic excitation. Even at 500ºC only 0.03% of molecules (with typical excitation energy of 400 kJ/mol-1) would experience electronic excitation.

When considering the previous discussion (which was not comprehensive) it can be argued that microfading results at present can vary in accuracy (but much less so for the simpler outcomes mentioned). Nonetheless the technique remains a success in highlighting fugitive colorants in collections as it has with most of the samples in this work.

It is clear further research into the techniques limitations is necessary to move with confidence to more complex outcomes critical to the conservation and the display of the most light sensitive cultural heritage.

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Author Biographies

Andrew Lerwill, Research Scientist, joined IPI in 2013 after completing a postdoctoral fellowship at The Getty Conservation Institute in Los Angeles, which he entered after working in research as a scientist at The Tate Gallery in the UK. He has a BSc in Physics from the University Of Hertfordshire, an MSc in Applied and Modern Optics from Reading University and his PhD dissertation "Micro-fading Spectrometry: an Investigation into the Display of Traditional Watercolour Pigments in Anoxia" was published in 2011 at The Nottingham Trent University. His research interests have surrounded the use of diverse technologies to measure, predict and control damage to cultural heritage, which is a subject on which he publishes and consults.

Christel Pesme received a BA in Biochemistry from the University of Paris 6-Pierre et Marie Curie, an MA in Art History and an MA in paper conservation, both from the University of Paris 1-Pantheon Sorbonne (France). For many years, she has been working as an assistant scientist at the Getty Conservation Institute, Los Angeles, carrying out light sensitivity for the Getty Research Institute, and the J. Paul Getty Museum. She is now working in Basel (Switzerland) as a private paper conservator. She also provides Microfadotesting services and has developed training for conservators interested in using MFT for lighting decisions.

Vincent L. Beltran is an Assistant Scientist at The Getty Conservation Institute and has been an active participant in the assessment of environmental management systems in hot and humid climates, the design and monitoring of anoxic storage and display systems, and examinations into the effect of anoxia on the color change of various materials.

James Druzik is a Senior Scientist at The Getty Conservation Institute, where he has worked since 1985. His research interests have been highly varied over the years and have included image processing (Caltech/JPL), the origin and fate of oxidant air pollutants and particulates in museum environments and their control technologies in conjunction with Glen Cass (Caltech). His group now routinely carries out assessment of light sensitivity for the Getty Research Institute, and the J. Paul Getty Museum and carries out research on solid-state lighting. He has previously worked at the Los Angeles County Museum of Art and the Norton Simon Museum in Pasadena, California. Druzik holds a B.S. in Chemistry.

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