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Exploring the Role of the Substrate in the Fading and Reversion behaviour of Prussian Blue Dye

Research Project submitted to the Department of Art in conformity with the requirements for the Degree of Master of Art Conservation

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Abstract

The pigment Prussian blue (ferric ferrocyanide) is known to exhibit phototropic behaviour due to its isotropic structure. Under prolonged exposure to light or in the absence of oxygen, Prussian blue will fade to Prussian white as its ferric iron is reduced to the lower oxidation state of ferrous iron. The compound will revert back to Prussian blue when re-exposed to dark, ambient conditions. While the fading and reversion mechanism of Prussian blue dye is largely understood, these behaviors are complicated by environmental conditions, by the presence of production additives, as well as by the substrate upon which it is suffused. For example, it remains undetermined if a proteinaceous substrate might influence the reduction-oxidation reaction of Prussian blue differently than would a cellulosic substrate. This is particularly important in the case of Prussian blue dye, which was of common use during the nineteenth century. Determining the degree and rate of colour change of Prussian blue dye on different substrates will yield significant information that could help refine more specific display guidelines for artifacts, as well as quantify the risks involved in the anoxic treatment of Prussian blue textiles. This study analysed the role of the substrate in the fading and reversion of Prussian blue dye by measuring the degree and rate of Prussian blue fading and reversion across samples of different substrates (cotton and silk). Samples were exposed to light using a microfade tester under ambient conditions as well as under anoxia. A second set of samples was exposed over a twenty-day period under regular and anoxic conditions in an LED light chamber at 100 klux. The colour change of these samples was measured using a portable spectrophotometer. Under ambient conditions, silk samples exposed to both LED light and to microfade testing were found to exhibit significantly greater overall colour change than cotton samples. The substrate was found to be a less significant variable in the fading of Prussian blue dye under anoxic conditions. Finally, the experiment demonstrated a discrepancy in results between silk samples exposed to microfading and silk samples exposed in the LED light chamber, suggesting that the intensity of the light source has an effect on the reduction behaviour of Prussian blue on silk (rather than overall dosage alone). This is a significant factor to consider if constructing future experiments that seek to mimic museum exhibition lighting conditions.

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1. Introduction

1.1 History of Prussian blue

Prussian blue (ferric ferrocyanide) is often referred to as the world's first synthetic pigment. While it has never been found in nature, Prussian blue is an inorganic pigment, traditionally prepared from iron and oxblood (Ware 1999, p. 21). Discovered in Berlin in about 1704, the chemistry of the pigment was not well understood until well into the next century. Prussian blue is precipitated when a solution containing ferric salts (Fe²⁺ ions) is mixed with a solution containing ferrocyanide ions. The resulting substance's bright blue colour is a unique property, and is due to the presence of iron in both of its oxidation states. The unusual behavior of Prussian blue quickly attracted the attention of chemists and remained a subject of study well into the nineteenth century (Ware 1999; Delamare 2013; Lowengard 2012).

The pigment was rapidly taken up as a substitute for increasingly expensive ultramarine and the unstable alternative of indigo. Prussian blue became popular in artist paints, but was also used in lower quality products such as wall paints and wallpaper. In the 1840s it was adapted for the photographic process of cyanotype printing (the process behind blueprints) (Ware 1999). It was first used as a dye at the beginning of the nineteenth century in France, when a shortage of indigo prompted Napoleon to issue a prize of 25, 000 francs to the person who determined a successful process to dye wool and silk with Prussian blue for the uniforms of the Grande Armée (Delamare 2013; Lowengard 2006, p. 2-3). The winning process, known after its inventor as the Raymond process, involved the mordanting of the textile with iron prior to dipping it in an acid solution of potash yellow prussiate (K4[Fe^{II}(CN)₆].3H₂O). This process proved problematic for dying wool until it was amended with a chlorine (bleach) bath in 1822, and later with the addition of tartaric acid in the ferric bath (Delamare 2013). The industrial practice of Prussian blue dyeing continued to develop over several decades as a greater understanding of the chemical process was achieved. The use of Prussian blue dye decreased significantly after the advent of synthetic dyes in the 1860s (Delamare 2013).

1.2. Chemical make-up and fading mechanism

Prussian blue (ferric ferrocyanide or iron(III) hexacyanoferrate(II)) is a mixed valence compound, meaning it contains iron in both of its oxidation states. The compound has a regular cubic crystal structure, with iron ions of alternating valence states occupying the corners and cyanide groups connecting them (Ware 1999, p. 138; Rowe 2004, p. 260). The open structure of the Prussian blue molecule allows the compound to act as a 'chemical sponge', 'trapping' heavy metals such as thallium and cesium within its lattice (Ware 1999). The compound's bright blue colour is a result of the "intervalence charge transfer between low-spin Fe²⁺ and high-spin Fe³⁺ centers, leading to adsorption around 640nm" (Gervais 2013, p. 1601). It is this energy adsorption that causes the blue colour. Prussian blue's charge transfer can be described by an equilibrium equation:

$$\{Fe^{3+} - C \equiv N - Fe^{2+}\} \rightleftharpoons \{Fe^{2+} - C \equiv N - Fe^{3+}\}$$

Eq. 1 (Gervais 2013, p. 1601)

The 'fading' of Prussian blue with light exposure should really be considered as a discoloration process, as the mechanism is not photo-oxidation, but rather the photo-reduction of Prussian blue to Prussian white:

$$KFe^{3+}[Fe^{2+}(CN)_6] + K^+ + e^- \rightleftharpoons K_2Fe^{2+}[Fe^{2+}(CN)_6]$$

Eq. 2 (Gervais 2013, p. 1601)

When Fe³⁺ ions are reduced to Fe²⁺ ions, new cubic crystalline compounds are formed bearing only Fe²⁺ ions at the corners (ferrous ferrocyanide, or Prussian white) (Ware 1999, p. 138). In this compound, the charge transfer responsible for the blue colour demonstrated in the equilibrium equation above no longer occurs (Gervais 2013, p. 1601) When returned to darkness (in the presence of oxygen), re-oxidation can occur, allowing Prussian white to revert back to Prussian blue.

1.3. Factors that promote fading: the role of the substrate

Prussian blue 'fades' as it is reduced to Prussian white. Reduction is promoted when Prussian blue is exposed to light or deprived of oxygen, but also requires the presence of materials that can act as reducing agents (electron donors) by themselves being oxidized. The Prussian white product requires the presence of cations to stabilize its charge balance. The source of these cations is variable, and has not been clearly defined. Prussian blue in paint films is quite stable and is not subject to fading, likely because the pigment is so tightly bound within the medium and not exposed to sources of electron donors. Prussian blue in the form of a dye or as a cyanotype, however, could be exposed to a number of factors that might influence fading. For cyanotypes, it has been determined that the presence of sensitizers likely promotes the reduction of Prussian blue (Ware 1999, p. 116-118). Other factors could include the high lignin content of certain paper substrates, or the presence of alkaline-buffered archival materials in storage.

In the case of Prussian blue dye, the possible sources of electron donors increase significantly. It is significant to note that Prussian blue dye is in closer contact with its substrate than Prussian blue as a cyanotype. Mike Ware refutes the possible impact of the substrate in cyanotype fading, partially due to the limited surface interaction between the Prussian blue particles and the substrate (Ware 1999, p. 125). Whereas on cyanotypes Prussian blue sits on the surface of the paper, as a dye, Prussian blue is mechanically bound to the fibres of the textile. This close proximity with the fibres could possibly increase the chemical interaction of Prussian blue with the substrate.

The fading of Prussian blue dye is subject to many variables. Throughout the nineteenth century, dye processes became increasingly complicated and utilized a number of additives to achieve the desired colour as well as maintain the desired texture and quality of the textile fabric. For example, tin was sometimes added at the mordanting stage to redden the final colour (Rowe 2004, p. 261). Impurities found during dyeing processing, or within the textile substrate itself (such as weighted silk) could also potentially influence fading. Finally, the nature of the substrate - its chemical makeup, its texture, its ability to take up and distribute the dye, and its degradation properties - could potentially have a significant effect on the reduction mechanism of Prussian blue.

1.4. Significance in museum collections

Nineteenth-century textile artifacts that are dyed with Prussian blue pose unique problems within museum collections. Firstly, as artifacts enter collection stores they are screened, and in some cases immediately treated, for pest infestation. In many institutions, newly acquired artifacts, or artifacts returning from loans, are temporarily stored in anoxic environments (carbon dioxide or nitrogen 'bubbles') so as to ensure any potential pests associated with the artifacts are exterminated. Anoxic conditions are known to accelerate the fading of Prussian blue (both when exposed to light and when in darkness) (Rowe 2004; Korenberg 2008), and it has not been conclusively determined if full reversion back to Prussian blue will occur afterwards. It is therefore essential to increase our knowledge of Prussian blue fading and reversion behavior under anoxia in order to determine if certain Prussian blue artifacts should be exempt from anoxic treatment.

Another significant factor in the fading of Prussian blue artifacts is their exposure to light. While Prussian blue is known to revert back after fading once returned to dark, ambient storage, it has not been conclusively determined whether a complete reversion ever occurs. Nor is it known if Prussian blue on certain substrate materials is less likely to revert. It is therefore important to control light exposure of Prussian blue artifacts on exhibition by display rotation or other means, so as to avoid significant fading of the dye. Further knowledge of the fading behaviour of Prussian blue on various substrates will help inform exhibition restrictions.

2. Experimental

2.1. Aims

The primary aim of the experiment was to determine whether Prussian Blue dye fades at a higher or lower rate on cotton substrates than silk substrates. In comparing the effects of a cellulosic and a proteinaceous substrate, the experiment was designed to reveal how great of a role the substrate plays in the fading of Prussian blue. Determining this would provide a greater understanding of the Prussian blue fading mechanism. The samples were created in such a way as to isolate the substrate from all other variables that might influence fading. For example, fabrics were carefully selected and sourced from Testfabrics, Inc. to assure the substrate of the samples did not contain any impurities.

A secondary aim was to determine whether the age of the substrate had an effect on Prussian blue fading. The experiment design allowed a comparison between PB fading on artificially aged cotton and artificially aged silk. However, comparisons could not be drawn between PB fading on artificially aged and un-aged cotton and between artificially aged and un-aged silk, as the aged fabrics (being more crystalline in nature) took up a lower concentration of the dye.

2.2. Sample Preparation

The experiment was carried out on Testfabrics, Inc. Desized, Unbleached Cotton Print Cloth (400U) and 8mm Silk Habutae (609). Artificially aged Testfabrics, Inc. Desized, Unbleached Cotton and Silk Crepe de Chine were donated by Season Tse, senior conservation scientist at the Canadian Conservation Institute. These fabrics were artificially aged by Tse in 1996 at 80°C and 65% RH for a period of two weeks. The pH measurements were taken on all fabrics prior to dyeing. The un-aged cotton and silk were found to both have an approximate pH of 6, and the aged cotton and the aged silk both have a pH in the range of 5-6.

The fabric was dyed according to a nineteenth-century tub-dyeing recipe that was adapted by a previous study (Rowe 2004, p. 262; Gervais 2014, p. 2) for small-scale laboratory use. In this method, Prussian blue was precipitated onto the textile substrate by soaking the textile in a bath containing Fe^{3+} ions (ferric nitrate), and subsequently in a bath containing $Fe(CN)_6^{2-}$ ions (potassium ferrocyanide). This process was followed by

an acidic rinse in an aluminum sulphate bath to neutralize any possible residual alkali in the textile (Rowe 2004, p. 261). The recipe used to dye the fabric is as follows:

To three tubs containing one litre of cold tap water each, add the following:

Tub 1: 7.65 g iron (III) nitrate nonahydrate

Tub 2: 7.56 g potassium ferrocyanide [potassium hexacyanoferrate(II)] 1.9 ml hydrochloric acid (0.4 M)

Tub 3: 0.95 g alum (aluminium sulphate)

Note that the above recipe differs from the Rowe 2004 recipe with the use of hydrochloric acid rather than sulphuric acid.

The fabric was cut into swatches roughly 12cm x 20cm to fit the size of the shallow solution baths. The swatches were immersed into the solutions lying flat, supported by sheets of non-woven polyester. The fabric was soaked for 30 minutes in Tub 1, with intermittent stirring of the solution. The fabric was then removed from the bath, and excess solution was extracted from the fabric by even pressing with blotting paper. The blotting paper method was chosen over wringing the cloth with the intent of maintaining an even distribution of ions throughout the fabric substrate. The swatch was then soaked for 15 minutes in Tub 2 with intermittent stirring of the solution. Again, upon removal from the bath, excess solution was extracted with blotting paper. The soaking procedure of Tubs 1 and 2 was then repeated. Finally, the fabric was soaked in Tub 3 for 3 minutes. The fabric was then removed and rinsed with repeated cycles of distilled water. The fabric was left to dry in the dark at room temperature.

The dyed fabric was then cut into 4cm x 4cm swatches. These swatches were chosen at random for the various experimental techniques. The purpose of this was to enhance the arbitrariness of the sample location measurements during microfade testing and measurements with the spectrophotometer. This ensured that if the dye was not taken up evenly by the textile substrate, representative data sets could still be achieved through a random distribution of measurements locations.

2.3. LED Fading

Samples were faded in an LED light chamber for 20 days at roughly100 kilolux. (see Figure 1 for spectral power distribution). Three 4cm x 4cm swatches of each substrate type (un-aged cotton, un-aged silk, aged cotton, aged silk) were selected to be faded in the LED light chamber in air. Three 4cm x 4cm swatches of each substrate type were selected to be faded in the LED chamber in anoxia. Additionally, three 4cm x 4cm swatches of un-dyed cotton and un-dyed silk were faded for 20 days in the LED light chamber as control samples. All samples were mounted by sewing them to pieces of neutral grey matboard. The pH of the matboard was measured and was found to be approximately pH 6, indicating that the board was not alkaline buffered.

The anoxic environment created for the sample sets being faded in anoxia within the LED lightbox was constructed using Escal[®] barrier film. The samples were heatsealed within a 15cm x 25cm bubble of the film, along with 4 packages of RP-K[®] oxygen scavenger. RP-K[®] was chosen over Ageless[®] due to its ability to control the relative humidity conditions within the chamber.



Figure 1. Spectral power distribution of LED strip used in lightbox. Indicates the absence of UV, but a sharp peak at 450nm.

2.4. Colour Reversion

After the 20-day fading period in the LED lightbox, the samples were transferred to an ambient, dark chamber (samples faded in anoxia were removed from their anoxic bubble) for a 20-day reversion period. Readings were taken every 2 days during this reversion period in an attempt to establish the rate at which reversion was occurring.

2.5. Colourimetry

Colour measurements were taken before and after the 20-day fading period, and once every two days during the 20-day reversion period. Chromatic readings were conducted using a CM-700d Spectrophometer. Data was logged by SpectraMagic software, which calculated colour change (ΔE_{00}) and chromaticity coordinates L* (lightness), a* (red-green), and b* (blue-yellow) using the 2000 CIE L*a*b* system formula. Using a 3mm aperture, fifteen measurements were taken at random locations across the three swatches of each substrate type (five readings per swatch) in order to account for variance.

2.6. Microfade Testing

Microfade testing was conducted at the Canadian Conservation Institute using a Newport-Oriel microfade tester with a 75 W xenon arc lamp (see Figure 2 for spectral power distribution for the first day of testing). The beam of light focused an exposure spot measuring approximately 0.3 mm in diameter on the sample. Infrared and ultraviolet filters removed UV radiation and heat from the light source. The spectral power distribution and luminous flux of the light spot was measured prior to exposing the sample using an ILT950 spectral radiometer with an INS150 integrating sphere and SpectralLight III software (International Light Technologies). The averaged flux of the light spot is 0.363 lm with intensity equivalent to 5.13 Megalux. Reflectance spectra of the test areas were collected every 30 seconds for 10 minutes using a CDI photo-diode array spectrometer (Control Development) with SPEC32 software.



Figure 2. Spectral power distribution of Xe-Arc lamp in Newport-Oriel Microfade Tester.

Three 4cm x 4cm swatches of each substrate type were selected for microfade testing. When reversion was being monitored, the light was blocked and data collection was stopped after the initial fading. Light and data collection resumed after a 10-20 minute reversion period. The Spec32 software calculated colour change using the CIEDE1976 colour difference formula (ΔE_{76}) and chromaticity coordinates L* (lightness), a* (red-green), and b* (blue-yellow). Further processing of the Spec32 data was done using the Getty Spectralviewer software, where both ΔE_{76} and ΔE_{94} (CIEDE1994) colour difference formulas can be plotted against time. The CIEDE2000 data is not used as the formula is not yet available with the current version of the Getty Spectralviewer software. Two measurements were taken on each of the three swatches of each substrate type.

2.7. Statistical Analysis

Un-paired, 2-tail t-tests were performed for L*, a* and b* values of each substrate type before and after the 20-day fading period to confirm colour change. Change was said to have occurred if the p value was found to be smaller than 0.05.

Standard deviations of L*, a*, and b* values for each substrate type were calculated in Excel. However, overall colour change (ΔE_{00}) for each substrate type before and after fading was calculated based on the averages of 15 sample readings of L*, a* and b* values. For this reason, to determine possible variance of the overall colour change, ΔE_{00} was calculated using the maximum possible variance and minimum possible variance of each data set (L*, a*, b* values) in addition to the averages of each data set. These values were plotted in a bar graph as error bars. The probability of rejecting the null hypothesis was determined by the degree to which the error bars of different substrate types overlapped.

2.8. Historic Samples

Two historic examples of Prussian blue dyed textiles were also used in this experiment. The first example, an 1860s printed silk vest with Prussian blue elements at the Canadian Museum of History (Figure 3), was analysed by microfade testing in the summer of 2014. The behaviour of this vest (specifically, the unknown behaviour of Prussian blue on a proteinaceous material) is what gave rise to the research question for this project. The second example, a Prussian blue-dyed cotton fabric sample swatch from an original volume of Knecht and Rawson's 1893 *Manual for Dyeing* (Figure 4), underwent microfade testing in 2015 at the Canadian Conservation Institute. The behaviour patterns of the historic examples of Prussian blue-dyed cotton and silk were compared to those of the samples created for this experiment.



Figure 3. Prussian blue printed on mid-19th century silk vest. Collection of the Canadian Museum of History, 982.9.3.



Figure 4. Page from Knecht and Rawson's 1893 *Manual for Dyeing* with sample swatch of Prussian blue-dyed cotton.

3. Results

3.1. Fading in LED light chamber

After a twenty-day fading period in the LED light chamber, all samples except cotton in air samples showed visible signs of colour change. Visible colour change (also described as the threshold exposure point) is defined as a ΔE_{00} of 1.0. Statistical analysis of the data confirmed that all samples experienced significant colour change (p \leq 0.05) during fading. (However, cotton samples faded in air did not undergo statistically significant change in lightness (ΔL^*) values, only in red-green (Δa^*) and blue-yellow (Δb^*) values.) Results of overall colour change during fading are summarized in Table 1.

Sample Type	ΔE00	ΔL*	Δa*	Δb*
Cotton in air	0.72	0.06	0.52	0.84
Silk in air	3.17	-1.74	2.19	2.61
Aged cotton in air	1.13	0.41	0.36	1.93
Aged silk in air	2.86	-1.83	2.79	1.69
Cotton in anoxia	1.35	0.35	0.44	2.25
Silk in anoxia	2.37	-1.07	1.81	2.16
Aged cotton in anoxia	2.59	2.06	-0.39	3.01
Aged silk in anoxia	2.61	-1.52	2.7	1.7
Cotton Control	3.56	2.28	-0.37	-3.72
Silk Control	0.29	0.33	-0.11	-0.11

 Table 1. Mean colour change after 20 days of fading

The cotton control samples underwent typical light bleaching (increase in lightness L* and a loss of yellow, decrease in b*), whereas the silk control samples underwent very little overall colour change (ΔE_{00}).

The results indicate that silk samples underwent the most overall colour change (ΔE_{00}) when exposed in the LED light chamber in both air and in anoxia. This is demonstrated by the graph in figures 5 and 6, showing overall colour change. The error bars in these graphs indicate the minimum and maximum possible variance in the overall colour change. For the samples faded in air (Figure 5) the error bars of each different

substrate type do not overlap, therefore it can be concluded that the overall colour change on the different substrate types is not the same. However, for the samples faded in anoxia (Figure 6), the error bars do overlap, and therefore it cannot be concluded that there is significant difference in overall colour change across the different substrate types when faded in anoxia. Additionally, for samples faded in air, no significant difference in overall colour change was seen between aged and un-aged cotton samples or between aged and un-aged silk samples. Finally, it is significant to note that the colour change of samples faded in anoxia varied less between substrate types than samples faded in air.



Figure 5. Overall colour change (ΔE_{00}) of samples faded in LED light chamber under ambient conditions (in air) for 20 days.



Figure 6. Overall colour change (ΔE_{00}) of samples faded in LED light chamber under anoxic conditions for 20 days.

Figures 7 and 8 demonstrate the significance of characterizing the samples not only by overall colour change, but also by the change in individual colour co-ordinates (ΔL^* , Δa^* and Δb^*).



Figure 7. Change in colour co-ordinates of samples faded in LED light chamber under ambient conditions (in air) for 20 days.



Figure 8. Change in colour co-ordinates of samples faded in LED light chamber under anoxic conditions for 20 days.

Silk and aged silk samples exposed in the LED light chamber in air and anoxia all exhibited negative mean ΔL^* values, indicating that the silk samples underwent darkening rather than fading in addition to their loss of blue (Δb^*). Silk samples, particularly aged silk samples, also underwent a loss of green (increase in a*) resulting in

a significant shift towards the red that was not seen in the cotton and aged cotton samples. In both air and anoxia, all samples except for aged silk experienced their most significant colour shift under the (Δb^*) co-ordinate, indicating that the most significant colour change for those samples was a loss of blue.

3.2. Reversion of samples faded in LED light chamber

Reversion measurements of samples that were faded in the LED light chamber presented less conclusive results. Measurements were taken every two days over a 20-day reversion period with the aim to observe the rate at which re-oxidation occurred. As before, 15 measurements were taken at random locations across the three swatches of each substrate type. Colour co-ordinates of the samples as they reverted were compared to the colour-coordinates of the samples before they were faded. Therefore the lower the (ΔE_{00}) , the closer the reverted samples are in colour to what they were before fading. Table 2 lists the results of the reversion period measurements. Reversion measurements from Day 8 are not listed due to outlying results. Likely, the wrong aperture size was used when measurements were taken on Day 8.

Table 2. Reversion measurements over a period of 20 days in data	rk, ambient conditions. Expressed in
overall colour change (ΔE_{00}) from samples prior to fading.	

Samuela Tama	Last day	Reversion								
Sample Type	of Fading	Day 2	Day 4	Day 6	Day 10	Day 12	Day 14	Day 16	Day 18	Day 20
Cotton in air	0.72	0.46	0.46	0.57	0.62	0.54	0.52	0.65	0.60	0.64
Silk in air	3.17	2.06	2.11	1.99	1.83	1.63	1.72	1.80	1.88	1.72
Aged cotton in air	1.13	0.79	0.87	1.05	1.21	1.09	1.07	1.20	1.23	1.19
Aged silk in air	2.86	2.11	1.93	2.19	1.86	1.95	1.97	1.96	2.00	2.14
Cotton in anoxia	1.35	1.52	1.79	1.96	2.03	1.99	1.89	2.03	2.03	2.06
Silk in anoxia	2.37	2.36	2.49	2.78	2.84	2.84	2.75	2.90	2.89	2.87
Aged cotton in anoxia	2.59	1.14	1.15	1.21	1.31	1.34	1.36	1.34	1.35	1.44
Aged silk in anoxia	2.61	2.40	2.36	2.76	2.56	2.52	2.73	2.71	2.71	2.92

Most significantly, no samples underwent complete reversion during the 20-day period in dark, ambient conditions. Only cotton samples faded in air finished at a point of non-visible colour difference ($\Delta E_{00} < 1.0$) from before they were faded. (However, cotton samples faded in air never reached a point of visible colour difference during fading.) Samples faded in air underwent their most significant reversion during the first two days of the reversion period. Cotton, silk, and aged silk samples faded in anoxia did not revert, and instead continued to exhibit very gradual colour change (along the same trends as when exposed to light and anoxia), despite being stored in dark, ambient conditions. None of the samples exhibited a consistent reversion trend over the 20-day reversion period.

Figures 9 through 16 display the reversion of each sample type over the 20-day period when broken down into change in individual colour co-ordinates (ΔL^* , Δa^* and Δb^*). However, as measurements were first taken after the first two days of the reversion period, these graphs do not record the early reversion that occurred during the first two days. In comparing ΔE values between the last day of fading and the second day of reversion, it appears that significant reversion occurred during this time.



Figure 9. Cotton faded in air reversion.



Figure 11. Aged cotton faded in air reversion.







Figure 15. Aged cotton faded in anoxia reversion.



Figure 10. Silk faded in air reversion.



Figure 12. Aged silk faded in air reversion.



Figure 14. Silk faded in anoxia reversion.



Figure 16. Aged silk faded in anoxia reversion.

3.3. Microfading

Microfading results for overall colour change (ΔE_{94}) roughly paralleled some of the trends found with the samples faded in the LED light chamber. In both air and anoxia, silk samples exhibited significantly greater overall colour change than cotton (fig. 19). However, unlike what was seen in LED light chamber, silk samples always exhibited positive ΔL^* value, indicating lightening or fading. Also unlike samples faded in the LED chamber, aged silk did not exhibit significantly greater overall colour change than aged cotton (Figure 17).



Figure 17. Mean microfading rates for samples faded in air at 32% RH.

Initial microfade testing of samples in anoxia seemed to indicate that under anoxia there was less difference in overall colour change between different substrate types (Figure 8). However, further testing demonstrated a very similar difference in colour change between cotton and silk samples microfaded in air, and cotton and silk samples micro faded in anoxia (Figure 19). Microfade testing indicated that reversion occurred at very similar rates between substrate types for samples faded in air (Figure 20), but for samples faded in anoxia (which remained in anoxia during the roughly 10-minute reversion period) did not revert and continued to fade (Figure 18). Reversion is represented in these graphs by the slopes of the lines between the last data point before the light was blocked and the first data point when the light resumed.

All samples microfaded in air underwent visible overall colour change after one and a half minutes. Visible colour change is considered to occur at a ΔE_{94} at or above 1.5. This number is approximate, as the colour discrimination threshold of the human eye varies by chromaticity, saturation, and hue (Konika Minolta 2007, p. 50). All samples exhibited a rate of fading similar to or higher than that of a Blue Wool 1 standard. However, the experimental samples faded much less rapidly than the two historic examples of Prussian blue dyed cotton and silk (Figure 21). The Prussian blue on the 1860 silk vest, faded to a visibly white spot in less than two minutes, but the colour reverted to original in two days.



Figure 18. Microfading results of samples faded in anoxia at 41% RH. Reversion period is indicated by the black line between exposure readings for each sample type.



Figure 19. Microfading results of un-aged cotton and un-aged silk in air and in anoxia.



Figure 20. Microfading in air at 32% RH with a 10-20 minute reversion period.



Figure 21. Microfading and reversion of historic example of Prussian blue dyed cotton (from Knecht, E. and C. Rawson. 1893) and microfading of historic example of Prussian blue-dyed silk (Canadian Museum of History silk vest circa 1860s, printed with Prussian blue).

4. Discussion

4.1. Samples

There is an inherent problem in making comparisons between Prussian blue reduction on different substrate types, and that is the issue of dye concentration. Prussian blue "fading" is a reduction reaction, and therefore does not follow the reaction laws of more typical, photo-oxidative fading. Specifically, Prussian blue does not "fade" more readily at higher concentrations of dye (as would be generally expected with light degradation kinetics where the reaction is dependent on the quantity of dye to be consumed). Rather, evidence has shown that Prussian blue fades at lower concentrations (Kirby and Saunders 2004; Gervais 2013, p. 1606; Bromelle, 1964). The molecular structure of the substrate, as well as its texture and weave, will influence the degree to which a fabric will take up a dye. This was evident in the current experiment with the dyeing of the aged cotton and aged silk samples. Due to the increased crystallinity of the molecular structure that occurs upon ageing, the aged fabrics took up the dye much less readily than the un-aged fabrics. This resulted in a discrepancy in the dye concentration of the aged and un-aged samples (Figure 22). However, the results of the experiment did not show a significant difference between the overall colour change (ΔE_{00}) of aged and un-aged cotton or between aged and un-aged silk (Figure 5). There were, however, significant differences in the change of individual colour coordinates (Figure 7). Aged silk, for example, underwent a more significant increase of red (Δa^*) and less significant loss of blue (Δb^*) than un-aged silk.

In the context of this experiment, concentration difference was only an issue between aged and un-aged samples. In general, however, the issue of dye concentration makes it difficult to compare Prussian blue on different substrate types which all take up the dye differently. Cotton and silk fabrics chosen for this experiment are very similar in texture and weave, which may have helped contribute to the relatively equal concentration of Prussian blue dye between un-aged cotton and un-aged silk samples. Both of these substrate types exhibited a spectral reflectance peak between 25 and 30% (Figure 22).



Figure 22. Spectral reflectance curves of different substrate types indicating relative concentrations of Prussian blue dye.

4.2. Role of the substrate

This project demonstrated a very clear difference in the behaviour of Prussian blue dye on cotton and on silk substrates. This is significant, as it indicates that the substrate participates in the reduction reaction of Prussian blue "fading", either as a reducing agent (a substance being oxidized) or as a reservoir of Prussian white stabilizing cations. If the substrate were being oxidized in order to facilitate the reduction of the Prussian blue dye, it would be interesting to explore the degree to which the substrate is being degraded in this process. However, if the role of the substrate is chemical, it is still unknown what component of the substrate is having the effect. The difference in behaviour between the cotton and silk samples could be attributed to the difference in their cellulosic and proteinaceous compositions, or rather it could have more to do with the overall stability of the material and the reactivity of its degradation products. Another possibility is that the role of substrate is primarily mechanical, and its molecular structure and/or fibre structure either facilitates or inhibits the reduction of the dye. The structure of the substrate may influence how the dye is dispersed within the fabric, or could inhibit or facilitate access to oxygen, reducing agents, or Prussian white stabilizing cations.

Regardless, the results of this experiment indicate that the substrate likely plays a more complicated role in the reaction than merely facilitating the production of Prussian white. While silk samples exhibited significantly greater overall colour change (ΔE_{00}) than cotton samples, the characterization of that change indicates that the silk samples underwent darkening during their exposure in the LED light chamber rather than lightening. The silk samples exhibited the expected loss of blue (as seen under the Δb^* colour co-ordinate), indicating that the Prussian blue dye was still reacting to form a new product. However, the darkening, or decrease in L*, suggests that it is unlikely that this product was Prussian white. If Prussian white is being produced via photo-reduction, the overall darkening seen on the silk samples would need to be occurring within the silk substrate itself. However, the un-dyed silk control samples proved to be very stable under exposure in the LED light box and did not exhibit any darkening.

The behaviour of Prussian blue dye on the silk samples presents a number of questions that will remain unanswered until further research is done on the subject. However, it can be concluded from the present research that it is critical to characterize the colour change resulting from the reduction of Prussian blue dye by breaking it down into its individual colour co-ordinates. An effective way of displaying colour change results is by plotting the changes undergone to the sample's spectral curve (see Appendix A for the spectral curves of each sample type over the course of the experiment).

The results of the experiment also seem to indicate that the substrate played less of a role in the fading of the samples exposed under anoxia. Difference in overall colour change was significantly smaller across substrate types in anoxia than across substrate types in air (Figures 5 and 6). Sample fading was not only generally greater in anoxia, but also more consistent. This indicates a slightly different mechanism in the Prussian blue-Prussian white redox reaction under anoxia (Figure 23). In anoxia, the Prussian white product is more stable as there is no oxygen to allow re-oxidation to Prussian blue. Similarly, the substrate is more stable in anoxia as it is less prone to oxidation. If the substrate does not oxidize, then it does not act as a reducing agent for the Prussian blue dye. The substrate's role in the fading of Prussian blue is therefore minimized in anoxia. Nevertheless, samples faded in anoxia will inevitably undergo greater colour change due to the inability of the Prussian white product to re-oxidize to Prussian blue. In anoxia, the redox reaction therefore is favoured to the right, as shown in the equation in figure 23.b.



Figure 23. Gervais 2014. Diagram demonstrating different reaction mechanisms of Prussian blue in air and Prussian blue in anoxia.

4.3. Fading results in context

Microfade testing indicated that noticeable change occurred across all sample types faded in air after only one and a half minutes. When converted to lux hours (knowing the intensity of the microfade spot to be approximately 5 megalux), this represents visible change after 128 000 lux hours. Therefore, all of the samples would exhibit visible change after 1.8 months if exhibited continuously at 100 lux (or 3.6 months if exhibited continuously at 50 lux).

Some problems were raised throughout the experiment regarding the ability of accelerated fading methods to effectively simulate exhibition lighting conditions. The silk samples exhibited darkening rather than lightening when exposed in the LED light chamber at 100 kilolux. In microfading, the silk samples had a similar rate of overall colour change but lightened, as would normally be expected with prolonged light exposure. However, the darkening in the LED chamber did not only occur on one sample of silk, but on un-aged silk in air, aged silk in air, un-aged silk in anoxia, and aged silk in anoxia. The repetition of these results is too significant to ignore.

It is therefore important to analyse what variables may have been involved in the fading in the LED light chamber that were not present during microfading or during experiments in previous research. The most significant difference is the light source. While it is useful to compare the spectral distribution curves of the light sources used in the different experiments, it has been noted by Ware that Prussian blue has adsorption

bands in both the red and blue regions of the spectrum, and "presumably either is effective in promoting fading" (Ware 1999, p. 122). For this reason, the actual distribution of the light source does not appear to be particularly significant (so long as it does not include UV radiation, which does have a significant effect on fading). However, the intensity of the light source is critical in the reduction of Prussian blue, as it does not obey the photochemical Law of Reciprocity (Ware 1999, p. 120). This means that if two identical samples were exposed for 100 kilolux hours, but each at different intensities, they may exhibit different degrees of fading. This could explain why the silk samples faded in the LED chamber at 100 kilolux exhibited different behaviour than the silk samples microfaded at 5 megalux. Furthermore, it indicates a need for research experiments examining the fading of Prussian blue artifacts to imitate museum exhibition lighting as much as possible if researchers wish to draw conclusions that can be applied to the practical realities of the museum.

Further inconsistencies were found between the results of the experiment's samples and those observed in real museum contexts. The research began with the microfading of a Prussian blue print on a nineteenth-century silk vest at the Canadian Museum of History (Tse 2014). At a later date, microfade readings were performed on a sampled of Prussian blue cotton from a late nineteenth-century dyer's manual (Knecht and Rawson 1893). The historic Prussian blue cotton sample exhibited extremely rapid fading (Figure 21), resulting in significantly greater (nearly double) colour change (ΔE_{94}) than the historic Prussian blue printed silk vest (Figure 21). In the context of these two historic samples, Prussian blue dyed cotton faded more readily than Prussian blue dyed silk, the reverse result of all fabricated samples faded in this experiment. This inconsistency suggests that for the historic samples, other variables (possibilities might include weighted silk, mercerized cotton, tin mordants, alkaline soaps, etc.) likely have a more significant influence on the reduction of the Prussian blue dye than does the substrate. The conclusion that must be drawn in this case is that for historic textiles dyed in Prussian blue, unless all of the variables can be characterized, the degree and rate of fading can be unpredictable.

Despite the rapid colour change of the Prussian blue textile samples, it is nevertheless unlikely that a Prussian blue textile would ever be exposed to light continuously within a museum context: that is to say, the calculated exposure limits discussed above do not take into account the reversion cycle that would occur during each night of an exhibition. In order to achieve a real idea of the behaviour of Prussian blue dye on museum artifacts, and properly assess the risk of permanent colour change, it would be necessary to monitor the fading and reversion cycles over real time. While fading can be accelerated, reversion cannot, and therefore longer, exhibition-based experiments would be the ideal way to determine if repeated cycles of fading impact the ability of Prussian blue dye to undergo complete colour reversion.

4.4. Reversion results in context

In this experiment it was found that even after a single 20-day exposure, Prussian blue dye did not achieve full colour reversion after a 20-day period in dark, ambient conditions on any of the substrate types. Samples that were faded in anoxia generally did not revert when returned to dark ambient conditions, but exhibited slightly further colour change.

The results emphasize that the reduction-oxidation reaction is not reciprocal: the rate of reduction of Prussian blue to Prussian white is not equal to the rate of oxidation of Prussian white to Prussian blue. Nor are these rates fixed: there are many variables and environmental conditions that could favour one of these reactions over another. Moreover, these reactions do not occur independently, but rather they occur simultaneously: the degree of fading or reversion is the degree to which one reaction surpasses over the other.

Cyanotypes have been extensively studied and have been characterized to achieve full reversion in a relatively short period of storage in dark, ambient conditions (Ware 1999, p. 130). This experiment demonstrated that textiles dyed with Prussian blue do not share this fortunate property. The dyed textile samples in this experiment did not achieve complete reversion, but furthermore none of the sample types exhibited any clear reversion patterns. It also appears that different elements of colour (colour co-ordinates ΔL^* , Δa^* and Δb^*) will revert at different rates and to different degrees.

It is not surprising that dyed Prussian blue textiles do not exhibit the same behaviours as cyanotype prints; the two forms differ not only in their methods of production, but also in the relationship between pigment and substrate. It is possible that the deep penetration and close interaction of Prussian blue dye with its textile substrate is a factor in the slower reversion process. In cyanotypes, "the surface contact between the Prussian blue particles and [the substrate] may be rather restricted" (Ware 1999, p. 125). For Prussian blue dyed textiles, the situation is quite the opposite: the dye is embedded in the textile and in close proximity to the substrate fibres. If the dye is embedded in the textile, it is likely that oxygen may not have as much access to the pigment particles as it does in cyanotypes, limiting the re-oxidation of Prussian white. The fine distribution and close proximity of the dye to the substrate fibres also creates a more suitable situation for the substrate to act as a reducing agent. It is therefore possible that the deep penetration and close interaction of Prussian blue dye with its textile substrate is a factor in its slower reversion behaviour.

5. Conclusion

The role of the substrate in the fading and reversion of Prussian blue dye was studied in this experiment by LED lightbox fading with spectrophotometry and microfade testing. Results from both methods showed that Prussian blue reduction does behave differently on cotton than it does on silk. However, when Prussian blue is reduced in anoxia, the substrate appears to have less of a role in the reduction mechanism. Based on these results, it remains unclear whether the substrate has an effect on the reversion (oxidation) of Prussian blue dye.

The rate of colour regain of Prussian blue dyed textiles is not reciprocal to the rate of fading. After 20 days of fading, none of the lab prepared samples achieved full reversion during the 20-day reversion period, and some (particularly samples faded in anoxia) achieved hardly any reversion at all. This is inconsistent with the Prussian blue dye on the 19th century silk vest (Canadian Museum of History), which visually showed reversion of colour within two days after microfade testing.

One of the most significant revelations throughout this experiment was the unexpected darkening behaviour of Prussian blue on silk when faded in the LED lightbox at 100 kilolux. Further research needs to be done in order to confirm this observation with Prussian blue dyed silk, to understand the nature of these results and to characterize the properties of the silk that are influencing the reaction.

Finally, this experiment has shown that there is a difference in the fading behavior of the silk samples when exposed in the LED light box versus the microfade tester. This is likely due to the differences in the spectral power distributions and/or the illuminance of the two light sources (100 klx for LED box vs. 5.1 Mlx for the microfade tester). These differences illustrate the limitations of accelerated light aging methods. It is uncertain which one of these methods more closely resembles ambient light fading of Prussian blue dyed textiles in air and in anoxia. The microfade results seemed to be more consistent with the current understanding of fading mechanism, i.e. that both dyed cotton and silk fade faster in anoxia, and reversion occurs only in ambient conditions and not in anoxia. Because Prussian blue converts to Prussian white by a reduction rather than an oxidation mechanism, it may respond differently to these accelerated light aging methods than materials that undergo more typical photo-oxidative fading reactions. While accelerated light aging techniques can accelerate the reduction of Prussian blue to Prussian white, it is not possible to accelerate reversion. It is not likely that Prussian blue dyed textile artifacts would ever be exposed to a continuous light source without undergoing periods of reversion in the dark. To properly characterize reversion properties of Prussian blue dyed textiles, fading and reversion cycles of a typical exhibition period need to be examined at ambient museum light levels.



Appendix A: Spectral curves of samples faded in LED light chamber















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Biography – Sophia Zweifel

Sophia Zweifel is currently completing her final year in the Queen's Master of Art Conservation Program, artifacts stream. Sophia completed her undergraduate degree in Art History and Chemistry at the University of British Columbia in Vancouver, and went on to complete an MA in Art History at University College London. She has carried out pre-program and curriculum internships at the Vancouver Art Gallery, the UCL Special Collections Library, and the Canadian Museum of History. She is very much enjoying her current internship at the Canadian Conservation Institute.