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The Use of Micro-Fading Spectrometry to Evaluate the Light Fastness of Materials in Oxygen-Free Environments

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ABSTRACT The use of inert gases for displaying sensitive objects and for the treatment of artifacts infected with bio-deteriorating agents is a current practice in many cultural institutions around the world. However, some artifacts may also experience color changes as a result of exposure to light in these reducing environments. Therefore, it becomes essential to evaluate the light stability of an object under anoxic conditions prior to its exhibition. This study dealt with the development of a procedure based on micro-fading spectroscopy to determine the light fastness of materials when exposed to an argon atmosphere. The efficacy of this method has been tested using turmeric/silk and Prussian blue/silk systems. Evaluation of color changes in real time have demonstrated that the approach provides an efficient way of detecting any potential variation occurring as a result of long-term anoxic display in a museum environment.

KEYWORDS anoxic treatment, microfading spectrometry, museum objects, visible reflectance spectroscopy

INTRODUCTION

Inert gases are typically used for displaying certain types of sensitive objects and for the treatment of collections infected with bio-deteriorating agents in archives, libraries, and museums.[1–3] Research conducted by Koestler and co-workers has indicated that low-oxygen environments created using argon (Ar) gas can be used for the suffocation of fungi and insects in cultural heritage objects.[4] As an inert gas, Ar does not directly react with objects, and its proper use holds no risk to human health, and therefore its use is favored for museum conservation purposes. In addition to the development of anoxic micro-environments for the treatment of museum objects suffering from biological attack, the investigations have focused on their use as a way of preserving objects that are highly sensitive to oxygen.[5] However, there has been some concern about the possibility of change in appearance of colorants such as Prussian blue in the absence of oxygen.[6,7] It is known that oxygen is an external factor that works in conjunction with light to produce damage on colored surfaces. If the color change of Prussian

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blue is the result of a reducing environment, there is reason to be concerned about the possibility that a similar change would influence other colorants that depend on oxidation during the dyeing process. Therefore, both light and oxygen must be considered since sensitive museum objects may experience significant color alterations due to their combined effects.[8] Based on the nature of the object, oxygen may act as a catalyst or inhibitor in the photochemical degradation reactions of an object.

This paper presents a new methodology suitable for performing micro-fading tests under an Ar atmosphere. Micro-fading spectrometry provides a viable way for testing the light-sensitivity of an object in a relatively short period of time when compared to traditional accelerated light-aging methods. Silk samples were enclosed inside a plastic bag during micro-fading testing. This approach provided an economical alternative to the use of a glove box in which a larger volume of gas is required. Building an enclosure around an object also gives more flexibility during testing since the dimensions of an oxygen-free chamber may not always allow placing large objects inside the unit or even setting up the reflection fixture of the micro-fading tester (MFT) properly. Whitmore et al. devised a testing setup in which the samples and the reflection fixture are enclosed using a plastic sheet to obtain an oxygen-free environment.[9] In their paper, those authors compare micro-fading results from tests conducted in air and after flushing the enclosure with nitrogen, revealing that the light stability of some materials may increase in the absence of oxygen. In contrast, other materials did not show a considerable change in fading behavior after comparison of tests conducted under oxic and anoxic conditions. Hence, the MFT can be used as a survey tool for verifying the light stability of materials that are expected to be exhibited under anoxic conditions. In the present study, the effect of Ar on the fading rate of Prussian blue/silk and turmeric/silk systems was investigated.

Prussian blue provided a way of evaluating the effectiveness of the proposed testing method since the chemical properties of this colorant, including its reversible redox processes, have been widely studied.[10,11] The fading of Prussian blue under anoxic conditions and the return of blue color after storage in the dark were first observed in the 1830s.[12,13] Rowe has also reported that the combination of anoxia and light exposure induces large color changes in Prussian blue/textile systems.[14] In this work, lesser fading of Prussian blue was observed for systems exposed to anoxic conditions in darkness. It is widely known that fading of Prussian blue under anoxic conditions is a complex phenomenon involving phototropic behavior of the dye if re-exposed to oxygen.[15]

The IUPAC name for Prussian blue is iron(III) hexacyanoferrate(II). Prussian blue is identified as Pigment Blue 27 in the Colour Index (CI), and its constitution number is 77510. The composition of the pigment can vary between insoluble Prussian blue—$\mathrm{Fe^{III}}_4[\mathrm{Fe^{II}(CN)_6}]_3 \cdot x\mathrm{H_2O}$, where $x = 14$–16—and soluble Prussian blue—$\mathrm{M^{I}Fe^{III}[Fe^{II}(CN)_6]} \cdot y\mathrm{H_2O}$, where $y = 1$–5. M is a monovalent cation such as $\mathrm{K^+}$ or $\mathrm{NH_4^+}$. Figure 1 shows the crystal structure of Prussian blue, which consists of a face-centered cubic lattice of Fe(II) ions interlocked with another face-centered cubic lattice of Fe(III) resulting in a cubic lattice with iron ions occupying the corners.[16] The $\mathrm{CN^-}$ ions are located at the edges of the cubes between each Fe(II) ion and the neighboring Fe(III). As a consequence, Prussian blue has two types of Fe ions: high-spin and low-spin Fe ions. High-spin Fe(III) ions are coordinated to N atoms of $\mathrm{CN^-}$ groups, while low-spin Fe(II) ions are coordinated to C atoms of $\mathrm{CN^-}$ groups. The centers of the cubic cells can include ions or molecules. In soluble Prussian blue, half of these cavities are usually occupied by potassium ions. In contrast, insoluble Prussian blue has an irregular lattice in order to balance the charges. A quarter of the Fe(II) centers is absent since the ratio of Fe(III) to Fe(II) is 4:3. Therefore,

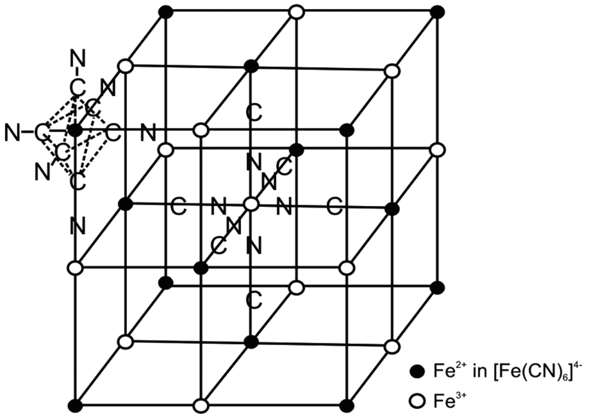

FIGURE 1 Simple cubic lattice of Prussian blue.

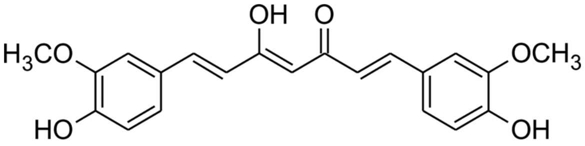

FIGURE 2 Molecular structure of curcumin in enolic form.

the vacant sites are occupied by water molecules instead. As a result, the Fe(III) centers are surrounded on average by 4.5 nitrogen atom centers and 1.5 oxygen atom centers, the latter from water.[17,18] The characteristic blue hue of Prussian blue is due to light-induced resonance between the two oxidation states of iron.[19] In the presence of oxygen, the Fe(II) ions formed in the above process are transformed into the original Fe(III) ions.

The light fastness of a turmeric/silk system was also evaluated under oxic and anoxic conditions. Turmeric dye is extracted from the *Curcuma longa L.* rhizomes. The deep orange–yellow powder obtained is commonly employed as a dye, food colorant, and spice. One of the compounds present in turmeric is curcumin, which is the main curcuminoid responsible for imparting the color. The IUPAC name of curcumin is (1E,6E)-1,7-bis(4-hydroxy-3-methoxyphenyl)-1,6-heptadiene-3,5-dione. The CI name is Natural Yellow 3, with constitution number 75300. The chemical structure of curcumin is shown in Fig. 2. The curcumin molecule consists of two aryl buten-2-one (feruloyl) chromophores joined by a methylene group. Curcumin can exist in two tautomeric forms, keto and enol, but the latter one is more stable if present in solid form or in a solution.[20] Previous studies indicate that the cis-enolic form gives extra stabilization to the molecule and allows conjugation between the β-electron systems of the two feruloyl chromophores.[21] Nowadays, commercial curcumin consists of a mixture of three naturally occurring curcuminoids: curcumin, demethoxy-, and bisdemethoxycurcumin, with curcumin as the main constituent. Curcumin is known for having very high sensitivity to oxygen and light.[22]

MATERIALS AND METHODS

Silk samples dyed with Prussian blue and turmeric were prepared and kindly provided by Anthony Maiorana and Cathy Zaret. The dyeing procedures were developed by Mary Ballard from the Smithsonian Museum Conservation Institute. Turmeric powder (≥98%), iron(II) sulfate heptahydrate (≥98%), and potassium ferricyanide (>99%) were purchased from Acros Organics (New Jersey, U.S.A.). Silk fabric was obtained from a commercial source in Washington, DC. Turmeric was applied as a direct dye. The dye bath was prepared by adding 100 g of turmeric powder to 6000 ml of distilled water. Dyeing of the silk was performed at 80°C for 1 h. After dyeing, the samples were rinsed with distilled water and then air-dried. Prussian blue/silk systems were prepared following the traditional dyeing methods described elsewhere.[23,24] The dyeing procedure required the use of two different types of baths. The first bath contained 83 parts of ferrous sulfate, 5 parts of concentrated sulfuric acid, and 13 parts of concentrated nitric acid, by weight. The second bath contained 10% by weight of fabric in potassium ferricyanide and 10% by weight in concentrated hydrochloric acid. This solution was then diluted with distilled water to a 100:1 ratio. The silk was worked for 1 hr in the first bath, which was maintained at room temperature. Afterward, the silks were rinsed with distilled water and then immersed into the potassium ferricyanide bath at 50°C, with constant stirring for approximately 1 hr. Finally, the silks were rinsed again with distilled water and were left to dry for 48 hr. Dyed silks were cut into 10 cm × 15 cm fragments prior to their exposure to light from the MFT.

Dyed silk samples were mounted to a piece of black cardboard similar to the one used for matting photographs. Another piece of cardboard was placed over the sample leaving a small circular path for incident and reflected light. Afterwards, the mounted sample was placed inside a low–gas-permeability transparent plastic bag, which was further closed using a portable heat and pressure sealer. The plastic bag had two small openings, one on the side for gas inflow and another one on the top to prevent interferences on the paths of illuminating and transmitted light. This second opening on the top also served as an outflow for the excess of Ar gas. The bag was filled with Ar gas that had first passed through water until a positive pressure was achieved and then manually evacuated three times. Water is typically employed to avoid hygrometric shock to museum objects during fumigation.[25] Therefore, the gas streams were humidified before being passed into the bag in order to simulate testing of an actual museum artifact. Afterward, a small flow of Ar was maintained, making sure that the bag had positive pressure by observing the pillow-like shape of the system. The top of the bag contacted the bottom part of the reflection fixture, providing a partially closed system. The appropriate working distance of 1 cm was established by using a jig, and after all necessary adjustments were made, every adjusting knob on the reflection fixture was secured in place using set screws. The sample was placed under the illumination beam, making sure that the opening on top of the bag provided enough clearance for the reflected light. This was done by measuring spectra in continuous mode and making sure that the spectrum shape and intensity were adequate. A diagram of the proposed setup is shown in Fig. 3.

The MFT used in this study was developed by Whitmore and co-workers from the Art Conservation Research Center at Carnegie Mellon University in Pittsburgh, PA.[9] Individual components of this instrument are manufactured by Newport Oriel Corporation (California, U.S.A.) and are sold as Oriel 80190 Fading Test System. The device consists of a visible reflectance spectrophotometer coupled to an accelerated light-fading micro-tester. Spectrocolorimetric data are recorded periodically, and their change is evaluated over time. The instrument uses a 0/45 geometry for illumination of the sample and collection of reflected light (Fig. 3). The diameter of the illuminated spot is approximately 0.4 mm. All micro-fading tests were performed using an illumination intensity of 6 Mlx. The integration time was 6 ms, and 10 spectra were averaged. The Commission Internationale de l'Eclairage (CIE) illuminant and observer combination used was D_{65} and 2°, respectively. Reflectance spectra were reduced to a set of parameters according to existing conventions adopted by the CIE for describing color.[26] Color differences were calculated using the CIE $L^*a^*b^*$ equation, which permits the researcher to evaluate complex changes, which usually involve shifts in hue and lightness. These coordinates refer to the lightness–darkness (L^*), redness–greenness (a^*), and yellowness–blueness (b^*) of the sample.

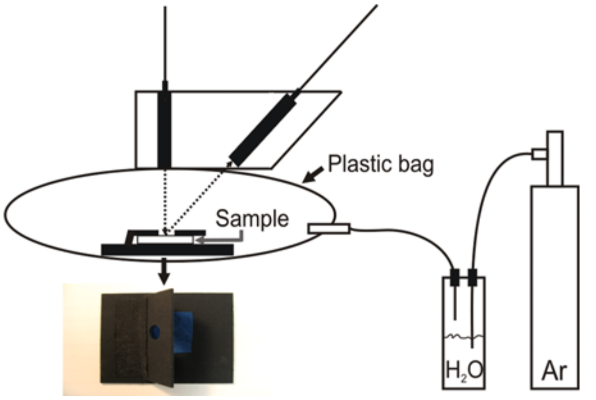

FIGURE 3 Schematic diagram showing experimental set-up used for micro-fading tests performed in an Ar environment. The photograph shows a Prussian blue/silk sample placed between two black cardboard supports.

RESULTS AND DISCUSSION

Figure 4a shows the visible reflectance spectrum of a Prussian blue/silk system, which is characterized by a band from 420 nm to about 600 nm with a

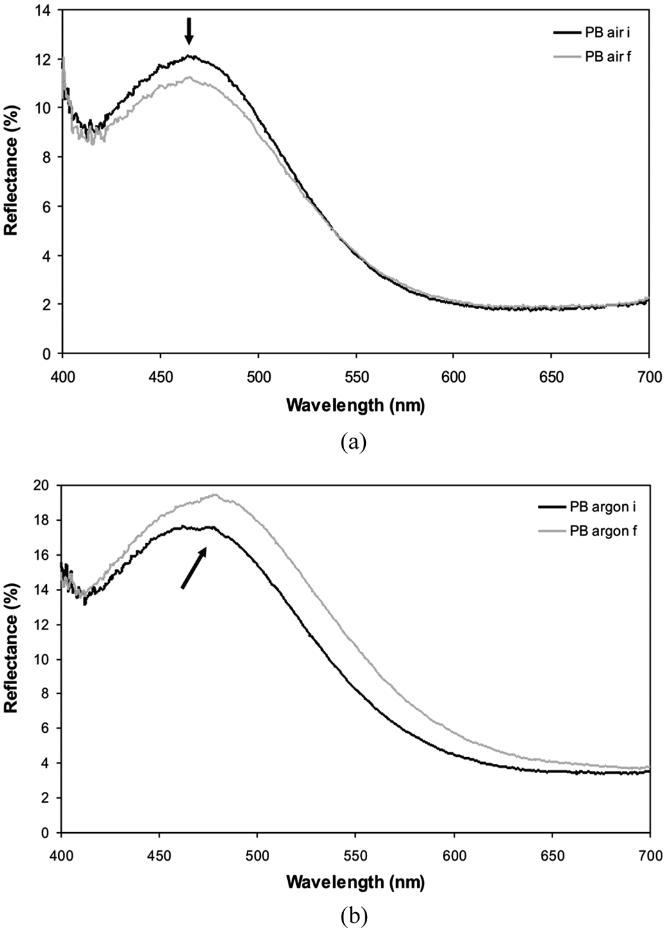

FIGURE 4 Initial and final reflectance spectra recorded for a Prussian blue/silk system in the presence of oxygen using an intensity of 6 Mlx (a), initial and final reflectance spectra recorded for a Prussian blue/silk system using an intensity of 6 Mlx under an Ar atmosphere (b).

maximum at 464 nm. It can be observed that some of the blue colorant is lost after 18 min of exposure to an illumination intensity of 6 Mlx. This was confirmed by a 1% reduction in reflectance observed after comparing initial and final spectra after accelerated aging. No detectable changes were observed in the 530–700-nm region as a result of accelerated aging. The same Prussian blue/silk system was evaluated under an Ar atmosphere using an intensity of 6 Mlx. Initial and final reflectance spectra are compared in Fig. 4b. After the accelerated aging protocol, this system showed an augment in lightness evidenced by increasing reflectance by approximately 2% over the entire visible spectrum. After 18 min of exposure, this system also experienced a shift in reflectance maximum from 466 to 479 nm.

Micro-fading curves for the Prussian blue/silk system evaluated in air and Ar atmospheres are shown in Fig. 5. After comparing spectra shown in Fig. 4, it was found that in the absence of oxygen the Prussian blue/silk system showed an entirely different spectroscopic change than the one observed when the test was performed in an oxygen-rich environment. A much larger color change was observed when the test was conducted under Ar. After 18 min, a ΔE^* of 6.4 was registered by the sample tested using Ar, while its counterpart only showed a 1.4 ΔE^* value. A color change smaller than 2 ΔE^* units would not be noticeable even for an experimented observer. Furthermore, the sample tested in open air exhibited a Type IV fading curve[27] characterized by a rapid initial color change, followed by a slower change after 2 min, showing evidence of some reverse fading.

The total color changes recorded during these two tests were further decomposed into their CIE $L^*a^*b^*$ components. It was observed that as a result of accelerated aging, both systems experienced increases in yellowness and lightness. However, this effect was larger for the system tested under Ar. For example, ΔL^* values experienced by samples tested in air and Ar atmospheres were +0.7 and +3.6, respectively. The Δb^* values recorded for samples tested in air and Ar were +1.3 and +3.6, respectively. Opposite behaviors were observed on the redness scale, and they were dependent on the type of environment used. For instance, the system tested in the presence of oxygen experienced a 1.4 increase in redness, while its counterpart registered a 3.8 reduction. These results are consistent with published data on the fading of Prussian blue/textile systems.[14]

Figure 6 shows the results of a test protocol that consisted of a longer test carried out to evaluate the effect of variable atmospheres on the color changes registered by Prussian blue. Initially this Prussian blue/silk system exhibited a rapid color change, reaching a ΔE^* of 9.8 after 90 min. At this point, the flow of Ar was interrupted, and immediately a recovery of the original color was observed. A significant reduction in ΔE^* by approximately 7 units was recorded after 100 min. This was followed by a linear increase in ΔE^* from 2.9 to 3.2 between 100 and 150 min. At this moment, the

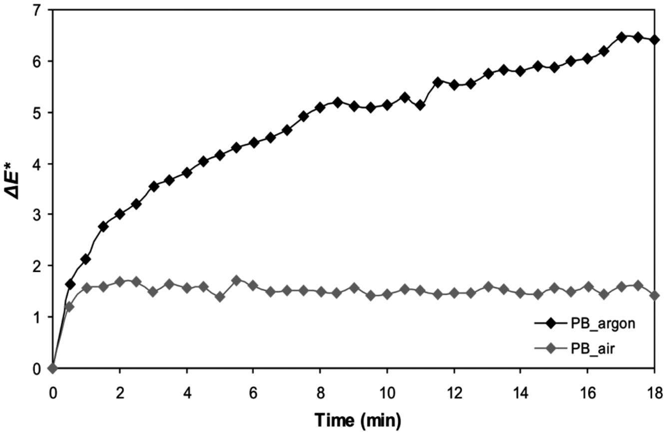

FIGURE 5 Micro-fading curves obtained for a Prussian blue/silk system using an illumination intensity of 6 Mlx showing different fading rates in air and Ar atmospheres.

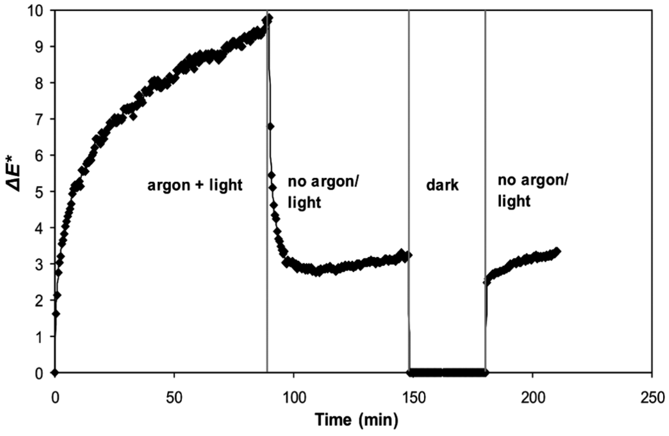

FIGURE 6 Micro-fading test conducted on a Prussian blue/silk system showing an increasing effect on the color change due to the presence of Ar environment followed by interaction with oxygen which reverses the original color shift.

illumination was interrupted by closing the shutter located in the focusing assembly of the MFT. Everything was left in place for 30 min, and afterward the illumination was restored by re-opening the shutter. As a result, a similar gradual increase in ΔE^* to the one observed between 100 and 150 min was recorded again from 180 min until the end of the test. These two color changes had the same magnitude. The data suggests that in the absence of light and in the presence of oxygen this Prussian blue/silk system recovered some of its original color since a decrease by 0.7 ΔE^* was produced during the dark period. It can be observed that a high reducing environment produces a rapid color change on the Prussian blue/silk system. Hence, the fast recovery of color observed from 90 to 108 min is not a strange occurrence due to the increased light sensitivity of the sample under anoxic conditions. However, the data shows that at the experimental conditions used, the reduction of Prussian blue to Prussian white is not fully reversible since the sample retained approximately 30% of its original color. A similar effect but to a lower extent was observed for the sample tested under oxic conditions. A longer dark period, greater than 30 min, could have resulted in a larger recovery of color during the last part of the test since reoxidation of Prussian white to Prussian blue is a slow process.[18]

Total color changes were decomposed into their individual color parameters. Figure 7 shows the change in CIE $L^*a^*b^*$ parameters with time observed for a Prussian blue/silk system under anoxic and oxic environments. The rapid initial color change observed in Fig. 6 was due to some loss of blue colorant, and a shift in hue from blue to green. This was confirmed by the spectroscopic data presented in Fig. 4. During the first 90 min of the test, increases in lightness and yellowness accompanied by a decrease in redness were detected. The direction of the color changes was inverted during the second phase of the test when the flow of Ar was stopped. After 30 min of darkness when the shutter was re-opened, the total color change observed was equivalent to the one observed from 90 to 150 min. However, the direction of change of individual CIE a^*b^* parameters was different. It can be observed that the redness parameter decreased slightly, while the yellowness parameter increased. The direction of the color change was similar to the one observed during the first part of the test performed using Ar. However, these changes had a lower magnitude in the oxygen-rich environment. The lightness parameter continued showing a decreasing behavior similar to the one observed between 90 and 150 min. These results are consistent with the partial fading of Prussian blue by reduction to Prussian white.[15]

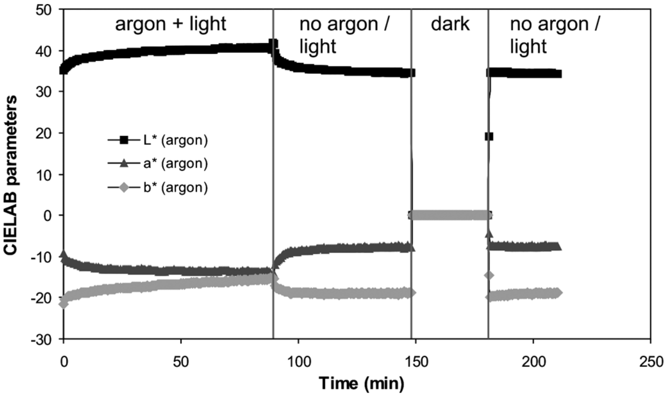

FIGURE 7 Change in CIELAB parameters with time for a micro-fading test conducted on a Prussian blue/silk system in presence and absence of Ar gas.

The mixed valence character of Prussian blue gives rise to the formation of various iron/cyanide complexes. In the presence of oxygen, Prussian blue may be oxidized to Prussian yellow, which contains exclusively Fe(III) centers, $\mathrm{Fe^{III}[Fe^{III}(CN)_6]}$. Under anoxic conditions, Prussian blue is reduced to Prussian white, which contains only Fe(II) ions. This white compound has several alternative names such as *Berlin white, Everitt's salt*, and *Williamson's salt*.[18] The name *Berlin white* is often used when referring to ferrous ferrocyanide, $\mathrm{Fe^{II}[Fe^{II}(CN)_6]^{2-}}$. However, to avoid confusion with lead white, which is sometimes also termed *Berlin white*, the term *Prussian white* is preferred. The photoreduction to Prussian white depends on the presence of some impurity that can be likewise oxidized to supply the electrons as described by the equation below:

$$\mathrm{Fe^{III}[Fe^{II}(CN)_6]^- + e^- \xrightarrow{h\nu} Fe^{II}[Fe^{II}(CN)_6]^{2-}}$$

In the presence of air, Prussian white is slowly reoxidized to Prussian blue. Micro-fading data suggests that some of the Prussian white compound was transformed back into Prussian blue once the flow of Ar was stopped and the sample regained contact with air.

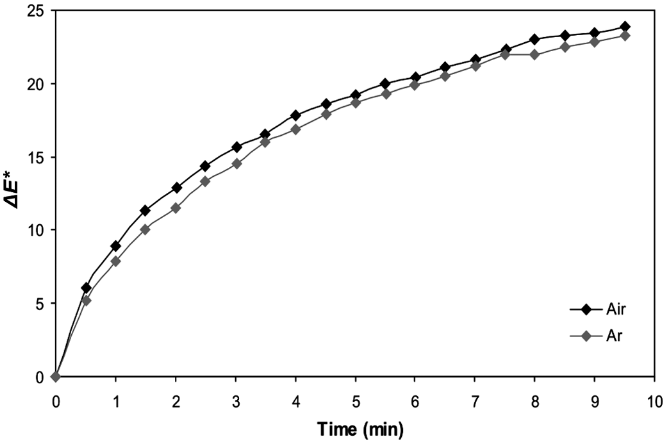

FIGURE 8 Micro-fading curves obtained for a turmeric/silk system using an intensity of 6 Mlx. Similar fading rates are observed after comparing tests conducted in air and Ar environments.

Micro-fading results revealed that turmeric has less sensitivity to Ar relative to Prussian blue. Micro-fading curves obtained for a turmeric/silk system using air and Ar atmospheres are compared in Fig. 8. A minor increase in light fastness was observed in the data recorded using Ar relative to the test conducted in air. However, this difference was very small indicating that the light stability of the turmeric/silk system is not changed in the new oxygen-free environment. Figure 9 shows the visible reflectance spectrum of a turmeric/silk system, which is characterized by a broad band from 500 to 700 nm. After 9 min of exposure, the major spectral change consists of an increase in reflectance in the purple–blue region of the spectrum. Some reduction in reflectance is also observed in the 535–620-nm wavelength range along with a slight increase recorded in the 620–700-nm region. Light-induced spectroscopic changes observed in air and Ar environments were comparable. After inspecting the change in CIELAB parameters, it was found that the overall fading behaviors were equivalent, consisting in large decreases in yellowness parameter along with minor increases in redness values. The magnitude and direction of these changes in a^* and b^* values were very similar. However, the ΔL^* values recorded for turmeric/silk systems tested in air and Ar were $+0.1$ and -2.9, respectively. It can be observed that the use of Ar seems to produce a darkening effect on the turmeric/silk system although the total color changes were equivalent for the two atmospheres evaluated.

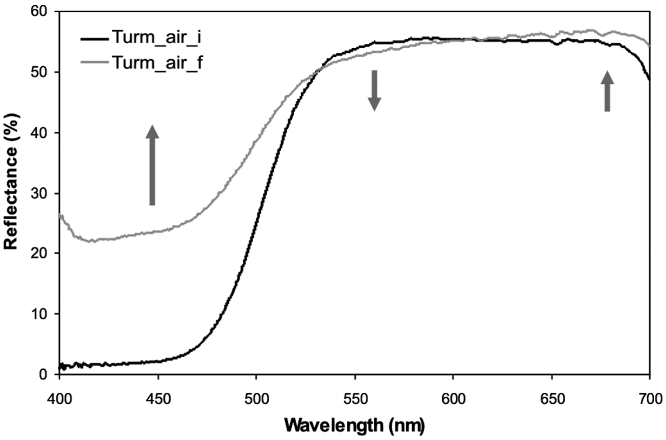

FIGURE 9 Initial and final visible reflectance spectrum recorded for a turmeric/silk system in the presence of oxygen. The arrows show the direction of the spectral changes observed after 9 min.

Although the photodegradation of curcumin has been reported both in solution and in solid form, the mechanism of photodegradation is still not clear. However, the collected data indicates that the presence or absence of the phenolic OH group does not play a significant role in the degradation of the dye. Previous studies have confirmed that the reaction mainly proceeds through the breaking of the β-diketone bond forming smaller phenolic compounds.[21,28] Some of the main products identified during photodegradation of curcumin include vanillin, vanillic acid, and ferulic acid (Fig. 10). Micro-fading trials may result in similar photodegradation mechanisms involving breaking of the β-diketone link. An evaluation of light-induced color changes for turmeric/silk systems indicates that the route of degradation is probably equivalent under

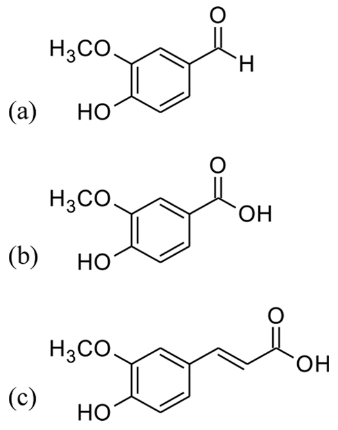

FIGURE 10 Chemical structures of some phenolic photoproducts of curcumin: vanillin (a), vanillic acid (b), and ferulic acid (c).

oxic and anoxic environments. However, further experimentation is still needed in order to confirm these statements.

CONCLUSIONS

A new experimental setup was developed for conducting micro-fading tests in anoxic environments. A turmeric/silk system showed no sensitivity changes in the absence of oxygen, while a Prussian blue/silk system exhibited high sensitivity to light when Ar was present. It was possible to follow the reversible fading of Prussian blue under variable atmospheres. It was found that Prussian blue exhibited high light sensitivity when tested under Ar, while the opposite effect was observed in an oxygen-rich environment. An evaluation of individual CIE $L^*a^*b^*$ parameters with time permitted us to establish the direction of the color change. The Prussian blue/silk system showed some loss of shade and a hue shift towards green when tested under Ar. These changes were inverted when the flow of Ar was stopped and oxygen came into contact with the sample. Micro-fading results indicate that Ar fumigation or exhibition under inert gas atmospheres could potentially discolor many cultural objects containing Prussian blue or other dyes sensitive to photo-reductive fading.

The proposed method is very promising for future micro-fading studies in anoxic environments. This kind of information about the materials is essential prior to designing an exhibit involving oxygen-free cases since the combined effect of light with inert gases may result in detrimental effects for the artifact. On the contrary, some materials may show higher light stability when displayed under anoxic conditions, while other could show no difference after comparing the two environments. For this reason, this type of survey is recommended prior to any anoxic conservation treatment or display policy involving inert gases. This methodology has demonstrated itself to be effective and therefore is presented as an initial step for future studies involving a larger number of samples.

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