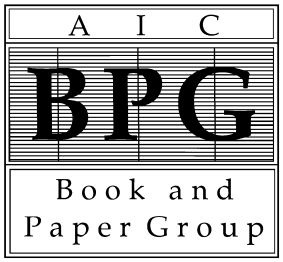

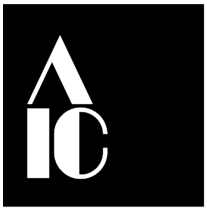

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MADISON BROCKMAN AND EMILY FAREK

Investigation into the Reduction of Foxing Stains in Paper

INTRODUCTION

The treatment of two water-sensitive chine collé lithographs by the famed French painter Pierre Puvis de Chavannes prompted an investigation into the reduction of foxing stains in paper using agarose gel to restrict moisture. Foxing is pervasive in works on paper and is difficult to reduce or remove, especially when full aqueous treatment is not a feasible option. Previous Winterthur/University of Delaware Program in Art Conservation student work has explored the use of combinations of chelators and enzymes; this is the first study to incorporate a novel reducing agent that targets the metal component, reducing Fe^{3+} to Fe^{2+}. This reduction renders iron into a more soluble form, enabling the use of common and accessible chelators for its removal. Preliminary testing indicated that sodium hypophosphite and the enzyme lyticase were highly effective in reducing foxing discoloration, and thus became the treatment protocol for the two prints. The prints were bathed differently—one on the suction table and one on TEK-Wipe, as a variant of blotter washing—to test the efficacy of the solutions in a variety of delivery methods. Treatment of the prints was safe and successful, significantly reducing the widespread foxing discoloration on both prints while preventing the delamination of the chine layers. This new protocol will provide wider applications for works on paper that cannot withstand aqueous treatment via full immersion bathing by using rigid polysaccharide gels.

TREATMENT OF CHINE COLLÈ LITHOGRAPHS

Each of the authors received one of a pair of chine collé lithographs, entitled *Le Ballon* and *Le Pigeon*, from the Winterthur/University of Delaware Program in Art Conservation study collection (fig. 1). The prints are reproduced paintings from the Franco-Prussian War, and much of their imagery was obscured by pervasive foxing. The original paintings were created in 1870 and 1871 by de Chavannes. They were immediately reproduced for distribution as lithographic prints by printmaker Emile Vernier, who also lived and worked in Paris during this time (Lacambre 2006).

In the chine collé technique, the primary support is commonly a thin Asian paper, *chine* in French. It is pasted on the verso and placed on a thicker secondary support; simultaneously, the two layers are fused together, and the image is printed as they go through the press. Chine collés can be difficult to identify and may be treated improperly, causing bubbling or complete delamination of the chine layer.

Looking at these objects with different illumination sources provides a wealth of information about their condition. Foxing can be organo-metallic in nature, with fungal components and metallic components that cause localized discoloration in the paper support. These discolorations appear as spots, yellow to dark brown in color, and diffuse to concentrated in shape. But what appear to be faint, rust-colored spots in normal light are typically brighter and more numerous when viewed in long-wave UV and in transmitted light.

Developing a treatment protocol that addressed the dual nature of foxing in a water-sensitive object was an interesting challenge. Preliminary testing of novel reagents began during an elective seminar on aqueous cleaning methods, building off of student research undertaken in previous years (Van Dyke 2004; Sullivan and Taira 2014; Sullivan, Brogdon-Grantham, and Taira 2014).

PRELIMINARY TESTING

To address the metallic foxing component in the primary support, two different reducing agents were tested: ascorbic acid and sodium hypophosphite. Reducing Fe^{3+} ions to Fe^{2+} ions would eliminate the need for dilute hydrofluoric acid or strong Fe^{3+} chelators like hydroxybenzyl ethylenediamine diacetic acid, allowing for the use of diethylenetriamine pentacetic acid (DTPA) or ethylenediamine tetracetic acid (EDTA). Two different enzymes were also tested to address the fungal component of foxing by targeting the chitin in the cell walls of the fungal growth. These included lyticase and a commercial blend of lysing enzymes.

These various combinations were tested with expendable foxed prints in full immersion baths in three steps. In

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Fig. 1. *Le Ballon* ("The Balloon," (a)) and *Le Pigeon* ("The Pigeon," (b)), by Pierre Puvis de Chavannes. Before treatment, recto, normal illumination.

the first step, the sample was placed into a deionized water bath containing the reducing agent and chelator. Second, the sample was rinsed in a bath of plain deionized water to remove excess reducing agent that would be harmful to the enzyme used in the next step. Third, the object was placed in a deionized water bath containing the enzyme. This preliminary testing showed that by visual analysis, sodium hypophosphite with DTPA proved to be more successful in stain reduction than ascorbic acid with EDTA. Lyticase was more successful than the lysing enzyme blend. Sodium hypophosphite has a higher reduction potential than ascorbic acid, and the lyticase enzyme is cheaper, purer, and not as sensitive to heat as the lysing enzyme blend. They also visibly appeared to reduce the foxing stains the most out of the reagents tested. This combination formed the foundation of the treatment protocol for the Balloon and Pigeon prints.

The treatment protocol developed begins with a pre-rinse step to remove easily water-soluble degradation products, followed by the steps tailored to reducing foxing discoloration. These include the reducing agent and chelator solution, followed by an intermediate rinse step. The enzyme solution comes next, with a final rinse to remove residues. This general treatment plan can be used for full immersion treatments or controlled applications of moisture.

METHODOLOGY

Agarose and other Gel Treatments

Gel treatments are currently in vogue in conservation, with good reason, although they may not be necessary in all applications. These two prints were excellent candidates for a gel treatment due to their inherent water sensitivity and need for aqueous cleaning solutions.

Many forms of gels are commonly used in art conservation, with polysaccharide gels such as agarose, gellan, and methyl cellulose most often used in paper conservation. Each gel has specific rheological properties that can act as a reservoir for solutions, restricting the flow of moisture into the paper support, while also acting as a poultice to draw water-soluble components out of the support. Some papers, such as the Balloon and Pigeon prints, readily absorb water, so controlling the amount of moisture is paramount. Rigid polysaccharide gel sheets also provide the benefit of physical restriction of the two chine collé paper layers during treatment, as the

weight of the gel sheet may help prevent separation caused by the differential expansion of the two layers when subjected to moisture.

Agarose was necessary for this particular treatment because it is a neutral gel—it carries no electrostatic charge. Gellan is a polyanionic molecule and could interact unfavorably with any ionic and enzymatic solutions added to it. Agarose does not have this issue and can carry solutions with aqueous chemistry like reducing/chelating and enzyme solutions. Agarose provides the additional benefit of strong capillary action, which is determined by its concentration.

Testing Different Delivery Methods

Given that the two objects were so similar in composition and condition, it was a unique opportunity to test the same treatment protocol using different delivery methods. The Pigeon print was bathed on a suction table, and the Balloon print was bathed on TEK-Wipe. TEK-Wipe is a highly absorbent, nonwoven fabric that is a blend of polyester and cellulose, and is a more sustainable choice than single-use blotter, as it can be washed and reused. The same aqueous solutions in agarose gel sheets were used in both treatments. In the suction table method, all rinse solutions were sprayed on the object while it was under suction, and each gel sheet was applied to the recto of the object for a total of 20 minutes. In the TEK-Wipe method, the TEK-Wipe was saturated with the rinse solutions, and each gel sheet was applied to the recto of the object for a total of 30 minutes. These treatment protocols were tested on expendable foxed chine collé prints to ensure they were safe and effective and to determine the gel dwell times for each method.

EXPERIMENT

Materials Preparation

The bathing portion of each treatment required the same aqueous solutions:

1. A pre-rinse citrate solution for the first and intermediate rinse steps
2. One phosphate buffer and reducing/chelating solution to be turned into a 3% agarose gel
3. One phosphate buffer and enzyme solution to be turned into a 3% agarose gel
4. A calcinated, alkaline solution for the final rinse step

These materials must be prepared immediately prior to treatment due to their limited shelf life, as otherwise they will oxidize with exposure to the air. First, create a buffered solution of sodium phosphate and citric acid, to be used for the two gels. Add DTPA and sodium hypophosphite to half of the buffered solution for the reducing and chelating gel, and add the lyticase enzyme to the other half of the buffered solution for the enzymatic gel. Add 3% weight by volume of agarose to each of the solutions. Dry agarose powder is insoluble in water at room temperature and must be heated to solubilize it. Cook each gel solution, then pour out into a Mylar tray to form a gel sheet large enough the cover the object. A plastic squeegee is a useful tool to help evenly spread the gel and ensure a consistent thickness of approximately 0.25 in. The agarose gel sets as it cools, forming a rigid sheet. Although large sheets can be difficult to handle if they are too thin or too thick, rolling the gel up like a rug makes them easier to handle.

Citrate rinse solution recipe: Use deionized water. Add enough sodium citrate salt (or citric acid and sodium hydroxide) to reach a conductivity of the solution that is within one order of magnitude as the conductivity of the object being bathed. Readings can be taken from the surface of the object with an agarose plug and a conductivity meter. Adjust the solution to pH 6 with citric acid.

Gel solutions recipes: Gel solutions recipes are shown in table 1.

Treatment of the Pigeon Print via Suction Table

Treatment of the Pigeon print began with surface cleaning (fig. 2). After humidifying the object overall in a Gore-Tex package, the print was pre-rinsed with a buffered solution of sodium citrate and citric acid at a pH of 6, with a conductivity close to that of the print. Conductivity and pH readings were taken from the surface of both prints with agarose plugs and digital meters. The rinse solution was sprayed with a Dia sprayer while the object was under suction, helping to pull water-soluble degradation products down into the blotter beneath it. After changing the blotter and applying a *gampi* barrier layer on top of the object, the first gel sheet was applied, which contained

Phosphate Buffer Solution	Reducing/Chelating Gel	Enzyme Gel
Per 300 mL of deionized water:	*Per 150 mL of phosphate buffer:*	*Per 150 mL of phosphate buffer:*
1.5 g sodium phosphate	Add 1.5 g of DTPA	Add 1.5 g of lyticase enzyme
Adjust pH to 7.5 with citric acid	Add 2 g of sodium hypophosphite	Add 4.5 g of agarose powder
Divide solution in half	Adjust pH to 7.5 with sodium hydroxide	
	Add 4.5 g of agarose powder	

Table 1. Gel solutions recipes

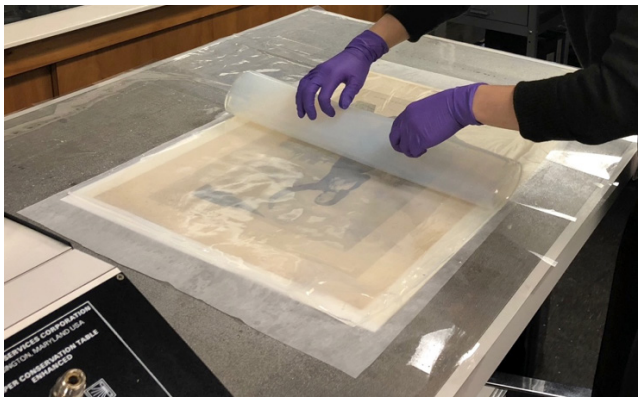

Fig. 2. "The Pigeon" being treated with reducing/chelating solution in agarose gel on the suction table.

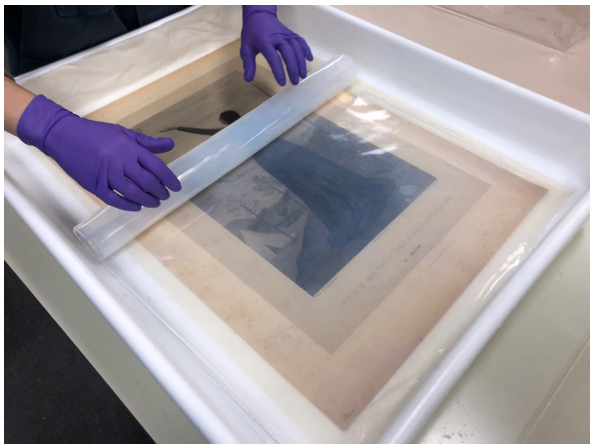

Fig. 3. "The Balloon" being treated with reducing/chelating solution in agarose gel in a TEK-Wipe bathing chamber.

the reducing and chelating agents. The gel sheet had a total dwell time of 20 minutes, after which the gel and *gampi* were removed and a new blotter was placed on the suction table. The object was rinsed again with the same buffered citrate solution as in the pre-rinse step. The blotter was changed again after rinsing in preparation for the next gel application.

The enzyme gel was applied to the object (with *gampi* barrier layer), and the same treatment steps were followed as for the reducing and chelating gel. Again, the gel had a total of 20 minutes dwell time, after which the gel was removed and the blotter changed. The object was rinsed with a final calcinated water solution of filtered water adjusted to pH 8 with calcium hydroxide. After aqueous treatment was complete, the object was placed in a drying stack of polyester interleaving, blotter, and felts.

Treatment of the Balloon Print via Tek-Wipe

The TEK-Wipe method proceeded similarly to the suction table method (fig. 3). The bathing chamber was prepared by saturating the TEK-Wipe with the pre-rinse citrate solution. A squeegee proved useful again to ensure even saturation and planarity of the TEK-Wipe. After surface cleaning and humidification in a Gore-Tex package, the print was placed onto the saturated TEK-Wipe and sprayed lightly overall with the same citrate rinse solution to ensure even wetting.

To prepare for the first gel application, a new layer of TEK-Wipe was put down and saturated in the rinse solution. The object was covered with a *gampi* barrier layer and the reducing and chelating gel. Air bubbles were pressed out to ensure overall contact, and the gel was left on for a total of 30 minutes of dwell time. The print was rinsed after removing the first gel by changing the TEK-Wipe again, spraying the print overall with the rinse solution using a Dia sprayer, and then letting the print bathe for 20 minutes. A new *gampi* layer was laid down and the enzyme gel was applied in the same way as for the reducing/chelating gel. Treatment was finished with a final rinse solution of calcinated water, adjusted to pH 8 with calcium hydroxide. After the final rinse, the print was placed in a drying stack like the Pigeon print.

RESULTS

Visual Observations

Overall, these treatments proved successful in reducing overall and local discoloration and did not result in delamination of the chine layer from the secondary support. Examination in UV light indicates a more drastic reduction of foxing spots in the Balloon print, although there is a visible reduction of the Pigeon print's foxing as well (fig. 4). The treatment was more successful in reducing the discoloration caused by the pale, diffuse form of foxing, but severe foxing spots show a dramatic improvement after treatment as well.

After treatment, the Pigeon print is visibly brighter overall and appears slightly less yellow (fig. 5). More diffuse areas of foxing appear to be reduced more significantly than more concentrated areas of foxing, especially in the upper right corner. The overall brightening and stain reduction are easily visible on the verso, where there is no media. Examination in UV light also shows overall brightening and the slight reduction of foxing spots, although it is evident that foxing is still widespread, if not visible in normal illumination. There is also a rectangle of brighter fluorescence visible on the verso, addressed in the Discussion section.

The Balloon print also brightened overall and appears slightly less yellow after treatment (fig. 6). This object had a paler, more diffuse form of foxing than the Pigeon print and thus exhibits a more drastic stain reduction, easily visible on the verso. Under UV light, the foxing appears to be reduced but is still present in some areas. On the verso, it is evident that the foxing spots that remain are less sharply defined.

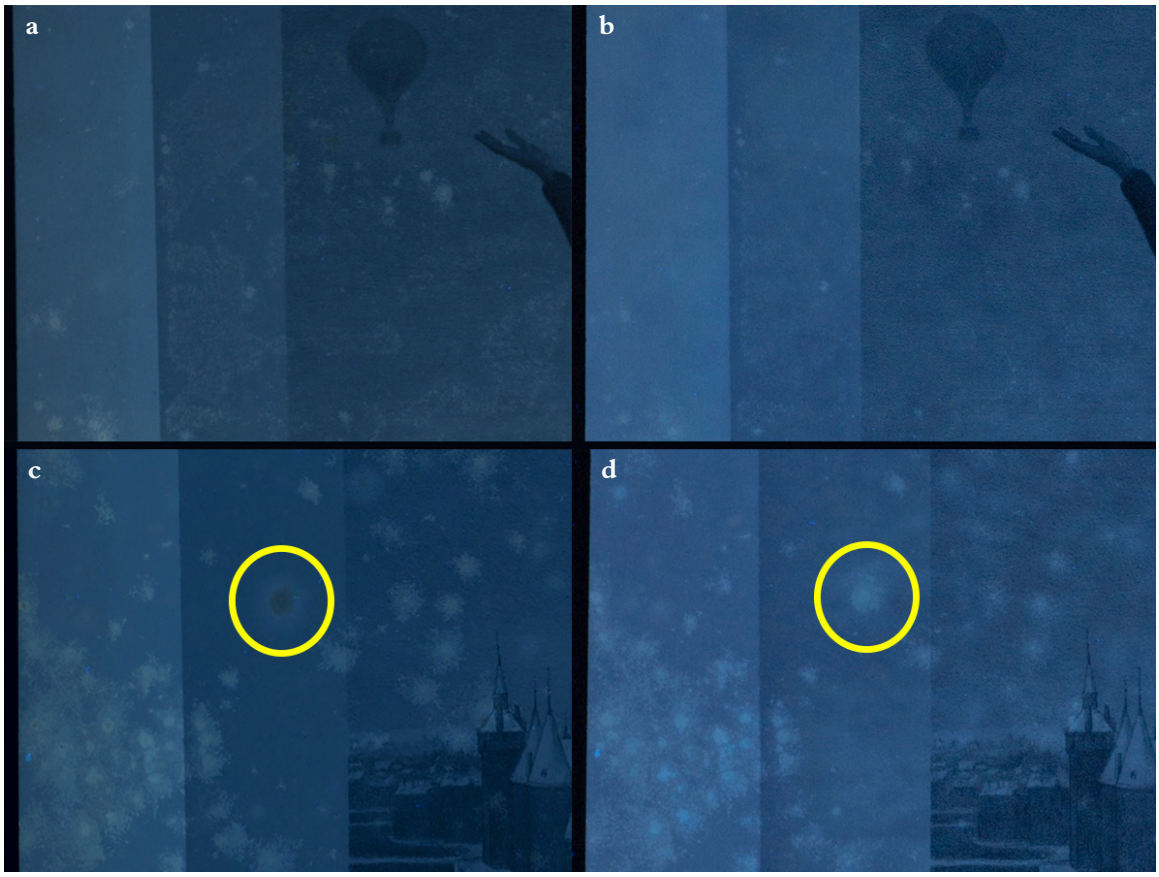

Fig. 4. Details before treatment of "The Balloon" (a) and "The Pigeon" (c), compared with after treatment (b and d, respectively). Reduction of severe foxing spots especially is visible in longwave UV illumination in "The Pigeon" (circled areas).

Quantifying the Results

The extent of foxing reduction and overall brightening is recognizable in visible examination, but quantifying these changes with colorimeter readings provides data to support the observed success of the treatment. A Minolta CR-221 colorimeter was used to take measurements of representative areas of each support, including foxing, and the minimum and maximum densities of the printed image (Table 2). The secondary supports of both prints appeared significantly brighter, with a $\Delta L^\star$ value greater than 2 for each. The human eye can detect a change in $L^\star$ value greater than 1, which explains why the overall brightening is so apparent. Furthermore, the $b^\star$ value decreased remarkably for both, indicating a reduction of the supports' yellow hue. Tracking the $\Delta L^\star$ and $\Delta b^\star$ values of foxing spots also indicates the efficacy of the treatments. The foxing spots measured on the Balloon print had a much greater degree of brightening and reduction in yellowing than the Pigeon print. This may be because of differences in the type of foxing found on the two prints, and their response to the treatment protocol and delivery method.

DISCUSSION

There is a great deal to discuss in regard to the success of these specific treatments, as well as their wider applications. A comparison of the two delivery methods yields some interesting factors to consider, which will guide the conservator in choosing between them.

The most apparent difference between these methods is the equipment necessary. The suction table is a common feature in paper laboratories; however, it does require an investment in a large, expensive piece of equipment. Laboratories without this specialized equipment can get good results with smaller, more easily available supplies such as absorbent material, like TEK-Wipe or blotter. The conservator must also consider the amount of time each treatment requires. The suction table has a quicker total treatment time, whereas the TEK-Wipe treatment is much longer. Each treatment has a different level of intensity, in which the suction table requires full attention and active participation

Fig. 5. "The Pigeon" before treatment (a) and after treatment (b), recto, normal illumination.

Fig. 6. "The Balloon" before treatment (a) and after treatment (b), recto, normal illumination.

	The Pigeon				The Balloon			
	ΔL*	Δa*	Δb*	ΔE	ΔL*	Δa*	Δb*	ΔE
Primary Support	+1.79	−0.30	−2.60	3.17	+1.92	−1.19	−2.10	3.08
Secondary Support	+2.03	−0.33	−3.23	3.08	+2.09	−0.58	−2.3	3.16
Primary Support Foxing	+3.06	−0.76	−4.09	5.16	+5.76	−1.62	−3.84	7.11
Secondary Support Foxing	+1.03	−0.16	−0.91	1.38	+4.94	−1.57	−4.63	6.95
Dmax	+0.75	+0.11	−0.88	1.16	−1.77	+0.23	−0.12	1.79
Dmin	+1.82	−0.23	−2.07	2.77	+1.41	−0.35	−1.52	2.10

Table 2. L*a*b* results.

throughout the entire treatment. Conversely, the TEK-Wipe treatment proceeds more slowly, so the conservator has more time to monitor the treatment or make changes.

Although the agarose gel sheets control moisture in both treatments, the gel used in the suction table method acts as a reservoir to slowly dispense the aqueous solutions while under suction. The gel used in the TEK-Wipe method functions as a poultice as it dries, actively drawing up water-soluble components into the agarose matrix. The amount of pressure exerted on the object also differs between the two techniques. The pull from the suction adds to the weight of the gel sheet in the suction table method, whereas the object in the TEK-Wipe method has only the weight of the gel on it. The needs of the object will dictate what treatment method to pursue. The suction table offers greater physical restraint and control of moisture but does exert more pressure on the object. Thus, the TEK-Wipe method may be more suitable for delicate objects or those that are not relatively planar.

The degree of rinsing varies greatly between the two delivery methods. The suction table allows for more rinse solution to be sprayed overall in multiple passes, whereas the amount of solution necessary to saturate the TEK-Wipe is a limiting factor. Similarly, the uniformity of rinse solution application also varies. The suction table relies on a sprayed application

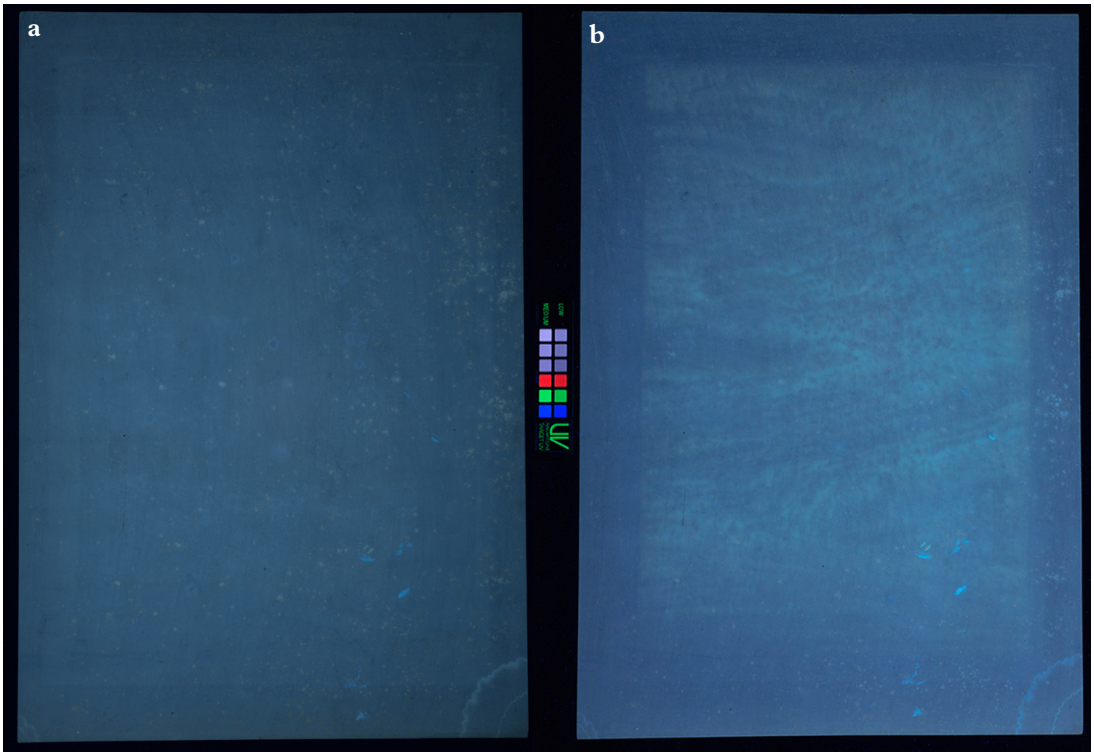

Fig. 7. Details of "The Pigeon" before treatment (a) and after treatment (b) in longwave UV illumination, showing the rectangle of autofluorescence that may be adhesive that migrated into the secondary support during treatment.

of the rinse solutions, which has the potential for unevenness due to human error. The TEK-Wipe method provides more even wetting because the absorbent layer is saturated overall with the rinse solution before the object is placed on it. This could be a reason the Pigeon print was brighter than the Balloon print after treatment: more solubilized products were moving out of the Pigeon print on the suction table. It could also appear brighter because it is cooler, due to the greater reduction of yellow hue reflected in the $\Delta b^{\star}$ value. An examination of their versos in UV light displays another result of rinsing (fig. 7). The Pigeon print, on the right, has a rectangle of brighter fluorescence, which may be the chine collé adhesive migrating through the secondary support matrix. Although the chine layer did not separate from the secondary support, some of the adhesive may have moved in the rinsing steps due to the pull of the suction. This degree of adhesive movement is not seen on the verso of the Balloon print.

Finally, one topic of particular interest is sustainability, both environmental and economic. The suction table requires electricity, whereas the TEK-Wipe method does not. TEK-Wipe can be washed and reused, and may be a more sustainable choice than blotter, which cannot be reused after it is saturated with degradation products. Although blotter was used in the suction table protocol, TEK-Wipe could be used instead. Similarly, this gel treatment protocol strove to use relatively inexpensive materials that did not have adverse environmental effects.

Future Research

It is the authors' hope that this is only a first step toward an increase in research led by other conservators and students. This general treatment protocol for the stepwise reduction of foxing stains can be applied to a variety of delivery methods based on the needs of the object in question. These include full immersion baths or more controlled applications of moisture such as gels or blotter washing. Further testing can be done with enzymes that have a higher activity level than lyticase, which may further reduce discoloration from foxing. Similarly, repeated steps or multiple applications of the reducing/chelating and enzymatic solutions may provide better results. One could also undertake other paths of research and analysis such as residue studies and artificial aging experiments.

CONCLUSION

The treatment described is an innovative one, which provides a method for overall aqueous treatment of foxed chine collé prints, including the use of a new reducing agent, enzyme, and gel delivery method. The combination of sodium hypophosphite and DTPA reduces and chelates the metallic component of foxing, whereas the lyticase enzyme targets the chitin of fungal cell walls in the fungal component of foxing. A dilute citrate rinse solution, used together with the aforementioned reagents, works to reduce both overall and local discoloration in the paper supports. A gel delivery method enables the application of aqueous solutions to objects that are extremely sensitive to moisture. Furthermore, this stepwise treatment protocol can be adapted to other delivery methods, for either controlled or overall stain reduction. Having multiple delivery methods to choose from allows laboratories with varying resources to execute a treatment with the same basic chemistry and allows for customization for each object that needs treating. The conservator has many options that each have their own advantages; it all depends on the needs of the object and the resources available.

ACKNOWLEDGMENTS

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APPENDIX

Case Studies at Other Institutions

In the past year, variations of this treatment were tested on several other objects at two different institutions. These investigations continue to add to the authors' collective understanding of how the chemical reagents synergize with each other in different delivery methods.

During a 2018 summer internship at the Fine Arts Museums of San Francisco, Madison Brockman (co-author) assisted with a similar foxing reduction treatment that had good results. The print in question had pervasive foxing in the secondary support and was unexhibitable in that state. Brockman and Fine Arts Museums of San Francisco conservator Victoria Binder carried out the same reducing/chelating and enzymatic protocol, this time using ascorbic acid and EDTA. As an important note, sodium hypophosphite is rather difficult to obtain for those not located in large research institutions due to its DEA class 1 protected status. Ascorbic acid is an excellent alternative, as it is economical,

easily obtainable, and produces satisfactory results. Sufficient rinsing is needed to clear ascorbic acid residues that discolor when oxidized. The print was bathed on TEK-Wipe and then rinsed on the suction table in a hybridized delivery method. After a first round of agarose gel bathing, the print was brighter overall and the foxing discoloration was greatly reduced.

Brockman also completed a special treatment project in early 2019 with Michelle Sullivan, Associate Conservator of Drawings at the J. Paul Getty Museum. This treatment involved the gellan gum gel–based aqueous treatment of a large chine collé photogravure with a stretched canvas lining. Gels were an excellent tool in this case given the inherent water sensitivities of the object and the necessity of moisture in the majority of the main treatment steps. The treatment protocol called for enzymatic adhesive reduction, overall bathing, and local stain reduction, all using gellan gel sheets. Gellan was selected over agarose due to the amount of gel needed for the entire multistep treatment, which would have been cost prohibitive given the higher price of agarose. The compatibility of enzymatic solutions and the gellan sheets were investigated prior to treatment. Despite commonly held beliefs, it appeared that the enzymatic solutions were not adversely affected by the polyanionic polymer structure of the gellan gum. The combination worked to successfully soften the thick adhesive, which was partially solubilized and imbibed by the gel matrix and largely removed mechanically from the paper support. The end results were similar to those seen in other prints mentioned in this article: the supports were brighter, local discoloration was reduced, and the chine layer remained in place.

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