The Book and Paper Group Annual

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Online Version: www.conservation-us.org/bpgannual

The *Book and Paper Group Annual* is published once each year in print format by the Book and Paper Group (BPG), a specialty group of the American Institute for Conservation of Historic and Artistic Works (AIC).

The *Annual* is distributed as a benefit of membership to those who were members of the BPG in the date year of the issue. Additional copies or back issues are available from AIC. All correspondence concerning subscriptions, memberships, back issues, and address changes should be addressed to:

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The *Book and Paper Group Annual* is a non-juried publication. Papers presented at the specialty group session of the annual meeting are selected by committee based on abstracts. After presentation authors have the opportunity to revise their papers before submitting them for publication in the *Annual*; there is no further selection review of these papers. Independent submissions are published at the discretion of the BPG Publications Committee. Authors are responsible for the content and accuracy of their submissions and for the methods and/or materials they present. Publication in the *Annual* does not constitute official statements or endorsement by BPG or by AIC.

ISSN 0887–8978
Printed in the United States of America by Edwards Brothers, Inc.

The paper used in this publication meets the minimum requirements of ANSI/NISO Z39.48–1992,
American National Standard for Permanence of Paper for Publications and Documents in Libraries and Archives.

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Sizing in Nineteenth-Century Book Papers

ABSTRACT

Recent research into nineteenth-century machine papermaking processes reveals that gelatin surface sizing for book papers was phased out during the early years of the machine period while alum-rosin internal size gained use. In this preliminary investigation, the authors tested for gelatin (protein) using biuret and ninhydrin reagents. A total of sixty American-imprint books—five from each decade spanning 1790 to 1910—were tested. Protein is present in a large majority of the tested book papers from the 1790s through the 1830s, but from 1840 to 1910 only a small minority of papers tested positive for protein. These results seem to support the premise that gelatin sizing decreased with the increased use of alum-rosin size. An iodine-potassium iodide test for starch was also carried out; starch was found in a few book papers starting in the 1830s, rising to a presence in a majority from 1880 on. This test confirms the addition of starch to alum-rosin sizing as found in contemporary papermaking recipes. The testing procedures were modified from standard ones. Samples in the ninhydrin test were not only heated, but also allowed to develop color (if positive) over several hours. For the starch test a standard commercial iodine-potassium iodide solution was diluted to different strengths to facilitate clear interpretation of the resulting color stain.

PAPER SIZING IN THE NINETEENTH CENTURY

The nineteenth century was a period of tremendous change in all aspects of American life, due to a progression away from hand-crafted articles toward mechanization. Increasingly, manufactured goods were made cheaply and efficiently by machines. Books both fueled this industrial revolution and were a product of it. Their three interdependent technologies—papermaking, printing, and bookbinding—saw incredible innovations as the century progressed.

Presented at the Book and Paper Group session, AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

Early in the century the advent of both the fourdrinier and cylinder machines demonstrated that papermaking could be mechanized. Closely associated with these engineering inventions, a major chemical advance—alum-rosin sizing—made papermaking more efficient by giving the sheet enough internal strength at the wet end of the machine to allow the paper web to withstand the tension created as it traveled and shrank over the heated cylinders through the dry end. Although the discovery of alum-rosin sizing was first announced in 1807 by Moritz Illig, it was not used commercially in America until the 1830s, coinciding with the rise of the papermaking machines.

The traditional manner of sizing paper was to hand-dip loft-dried sheets in a tub of warm gelatin. Today this kind of sizing is called tub, surface, or external sizing. In the nineteenth century, it was called "animal sizing." The degree of gelatin sizing applied to paper depended on the anticipated use. Weak, soft, or slack sizing was appropriate for papers to be letterpress-printed with an oil-based ink. Slack sizing allowed book and news paper to be readily dampened before printing, which resulted in better impression, better ink deposition, and the consumption of less ink.

Conversely, strong or hard sizing was reserved for papers that had to withstand the application of water-based mediums and erasures. Additionally, intaglio plate papers required hard sizing because, once the paper was dampened, it had to be strong enough to stretch under great pressure.

Unfortunately tub sizing was a time-consuming and relatively wasteful practice. Gelatin solutions soured and putrefied in a few days, especially in the summer. In an effort to extend the working life of the gelatin, alum was added daily, and larger proportions of alum led to an increasingly acidic and softer sized sheet. Over several days this practice resulted in batches of paper that today exhibit differences in discoloration, acidity, and weakness. This progression, not noticeable at the time of printing, often can be seen across signatures in the same book.

Alum-rosin sizing occurred in the Hollander beater before sheet formation, and thus it is referred to as internal or engine

sizing—engine is another word for beater. In the nineteenth century it was called "vegetable sizing."

First, rosin soap was made by boiling powdered resin with caustic soda and water in a process known as saponification. An 1866 recipe for vegetable sizing called for the following (Proteaux 1866, 72):

100 parts by weight of dry pulp
4 parts of starch
8 parts of rosin soap
8 parts of alum

Perhaps surprisingly, starch was often added to the pulp when alum-rosin sizing. Contemporary sources reported that starch—usually potato or farina—made the paper stronger and less spongy, gave it a harder surface, which made glazing or calendering more effective, and rendered the sheet more moisture resistant, although Browning disputes this last claim (Browning 1977, 89). The starch was mixed in a small amount of warm water and added to the rosin soap solution.

Typical steps in alum-rosin sizing were:

- add processed pulp to the beater
- wash out the residues of any alkaline cooking liquor, bleach, or anti-chlor
- test the pulp to ensure the pH is between 7 and 9
- add alum to neutralize any alkalinity from processing residues or hard water
- add still more alum, which serves as a mordant, along with the rosin soap and starch
- beat the pulp for 3 to 5 hours

During beating the rosin and starch are precipitated and then melted onto the fibers as the paper travels over the drying cylinders.

Unfortunately, excessive amounts of alum result in acidic papers, whether it is added to the pulp in engine sizing or to the gelatin during tub sizing. Much of the gray-brown discoloration and overall weakness, but not necessarily embrittlement, of rag paper made during the nineteenth century can be attributed to the use of too much alum during sizing. The extreme embrittlement seen in some papers dating from the late 1880s into the twentieth century is the result of alum-rosin sizing added to groundwood pulp to make very cheap grades of news paper. This embrittlement is due to the large proportion of lignin in groundwood pulp, which, when combined with excess alum, contributes to severe cellulose degradation.

Cathleen A. Baker has been researching the technologies and materials of American nineteenth-century papermaking in preparation for a book to be published in 2010. From her research she concluded that alum-rosin sizing in machine-made book papers resulted in the virtual elimination of external, gelatin sizing as the century progressed. As always, there was a practical reason for this shift. By midcentury book and newspaper printing presses had evolved from the simple iron hand press to steam-powered, high-speed bed-and-platen and cylinder presses. Even though machine-made paper was available in reels, the roll was still cut into sheets to facilitate sheet-feeding these presses with dampened paper. By the end of the Civil War, however, it was increasingly common to find automatic web- or reel-fed presses in action. It was no longer necessary to manipulate dampened sheets into the grippers of the press, and so the need for gelatin-sized news and book papers decreased.

EXPERIMENT

To confirm that there was a trend away from surface-sized papers for letterpress-printed books in the nineteenth century, the authors devised the following experiment in the summer of 2008. The hypothesis was that the incidence of gelatin would be highest at the start of nineteenth century and decrease with time, while starch would show an inverse trend, increasing across the century as it was introduced into papermaking as an ingredient in alum-rosin sizing.

METHODOLOGY: COLLECTING AND PREPARING PAPER SAMPLES

Paper samples taken from five books from each decade between 1790 and 1910, for a total of sixty books, were tested for protein, i.e., for gelatin, with the biuret and ninhydrin reagents. The iodine-potassium iodide test was used to detect starch. Each book selected for testing was at least one hundred pages long, published or printed in the United States of America, still in the original binding, and without major repairs or conservation treatment. Additionally, selection preferred books that seemed to have had minimal handling because any residue left on paper after contact with skin may test positive for protein, and books with minimal disruption of the paper surface because most of any gelatin sizing will be found in the paper's outermost fiber layers. Finally, books were chosen without regard to paper condition, so as not to inadvertently select for or against gelatin, in case there is a correlation between gelatin sizing and paper condition after natural aging (Barrett 1992).

In selecting sample areas for testing, the first and last signatures were avoided because these sections are more frequently handled. Additionally, pages with inscriptions, such as underlining or marginalia, were not used because their surfaces have clearly been handled extensively by readers. Finally, samples were not taken from the gutter in case animal glue, which would test positive for protein, was used during binding.

All three analytical tests used in this project are destructive because they usually stain the paper surface. Of the sixty books sampled, destructive testing was possible on about half—in these cases testing was performed directly on a page taken from the book. When destructive testing was not possible, fiber samples scraped from the page surface were tested instead. To obtain fibers for testing, a clean scalpel held perpendicular to the paper surface was used to scrape fibers from both sides of the sheet onto a microscope slide. The area scraped was typically one to two square centimeters. As the test results are based on colored stains, each slide was examined under magnification before testing to find any colored fibers that would interfere with the test results. Colored fibers were occasionally found and removed from the slide.

METHODOLOGY: ANALYTICAL TESTS

In the biuret test for protein, two reagent solutions were used in sequence (Browning 1977, 103). First, a single drop of Reagent A (1.0 g copper sulfate dissolved in 50 ml deionized water; solution is a turquoise blue color) was deposited directly on the sample, without touching the paper or slide surface with the dropper. Next, after several minutes, a single drop of Reagent B (2.5 g sodium hydroxide dissolved in 50 ml deionized water; solution is colorless) was deposited on the same spot. To wick away excess reagent at each stage, a blotter corner was touched to the domed drop of reagent, with careful attention not to let the blotter touch the paper. This step was not necessary if the reagent absorbed quickly into the paper.

Interpretation of the biuret test is based on the color of the resulting stain. A violet color indicates the presence of proteins; a sky-blue color indicates a negative test result. Having known samples of positive and negative results to reference was essential for interpreting the results. The colored stains of both positive and negative tests changed again within a couple of days after the test was performed; in particular, positive tests showing a violet color consistently changed to a blue-green color, resembling negative tests. It was thus helpful to retest reference samples for each testing session in order to have a fresh sample to reference.

Because the biuret test is not sensitive to small concentrations of proteins or to certain amino acids, the extremely sensitive ninhydrin test for protein was used for some samples. Whereas interpreting the color results of the biuret test was difficult, positive and negative results of the ninhydrin test were much more easily distinguished.

The ninhydrin test for protein uses a single reagent composed of two solutions prepared separately (Browning 1977, 103). First, 0.14 g sodium hydroxide and 0.43 g citric acid are dissolved in 49 ml deionized water; separately, 0.5 g ninhydrin powder is dissolved in 49 ml acetone. Once both solutions are dissolved, they are mixed together. The resulting reagent is a transparent pale yellow color.

To perform the ninhydrin test, one drop of the reagent was placed on the paper and allowed to rest several minutes before heating. If necessary, excess reagent was drawn off with a corner of blotting paper. Fiber samples on microscope slides were not blotted out of a concern for removing fibers from the slide surface. Samples were heated for several minutes and examined for results.

Like the biuret test, the results of the ninhydrin test are indicated by color: red through violet to blue indicates the presence of some kind of protein, while a negative test is colorless. When the test was performed directly on the paper surface, colors ranging from dull red to magenta to violet were observed, and the intensity of the color varied greatly. Reference papers known to be heavily sized gave more intense, stronger colors, and the results appeared more quickly than reference papers sized with less gelatin, which yielded colors that were fainter and took longer to appear.

When the test was performed on fibers rather than on a page, colors from the ninhydrin test were most often visible only under magnification. A range of results were observed on the slides under magnification, including fibers stained entirely or partially red, and small globules stained red or violet, situated next to or tangled in fibers. (It is unclear what exactly these globules were; it is probable that they were accretions of gelatin that had dissolved in the testing reagent.) For this project, one of two results constituted a positive ninhydrin test: either multiple fibers or fiber portions stained red or violet, or the presence of multiple stained globules and some partially stained fibers.

The samples—both the pages and the microscope slides with fibers—were heated on a large tacking iron plugged into a rheostat and turned upside down (fig. 1). This set-up allowed the tester to place a sample on the tacking iron and have hands free to continue other tasks. The sample had to be heated for several minutes before a color appeared. If heated for too long, the slide surface showed areas of orange-brown film, although the red or violet color of a positive test was still visible under magnification.

The ninhydrin test proved also to give results without heating, although the reaction time is slower. During a review of the samples, it was observed that one test that had previously appeared negative showed a positive result several days later. A second round of testing was performed on that sample, as well as on some other previously tested samples, using new pages taken from the same books. These samples were not heated at all. The reagent was applied and allowed to air dry, and then each page was placed in a plastic bag. The next day, some test papers were plainly positive and others negative, for the most part corresponding to the results of the heated tests. After two hours the unheated test shows only a little color, while the heated test shows the strong purple

color of a positive result (fig. 2). After nearly two days however, the heated and unheated tests are equivalent (fig. 3).

The extreme sensitivity of the ninhydrin test can make it difficult to work with. A single fingerprint on a reference paper known to be gelatin-free can show a very slight purple color in this test (Barrett and Mosier 1992). The more contact a sheet has had with skin, the more distinct the positive result will be. For this reason, it is necessary to define the intensity of color constituting a positive test. Also, special care is required to handle the samples, slides, and other materials with forceps or gloved fingers so as not to contaminate the results.

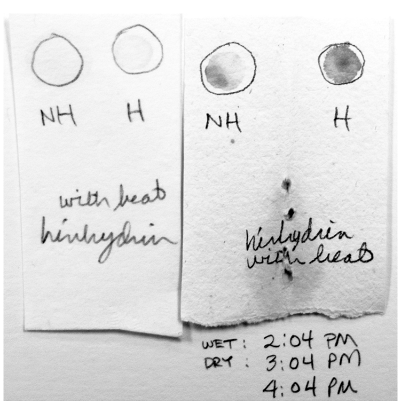
Fig. 1. The ninhydrin test setup using a tacking iron connected to a rheostat for heating the sample

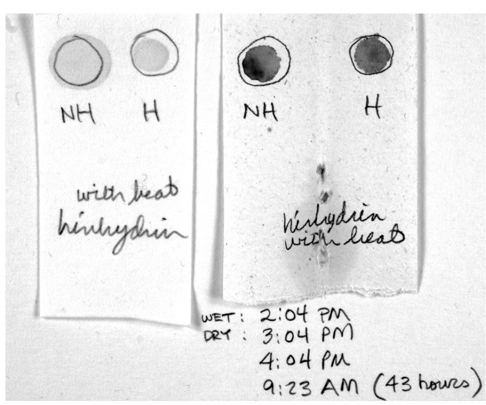
To test for starch, a prepared iodine-potassium iodide solution purchased from Fisher Scientific was used (Browning 1977, 91). The solution straight from the bottle is a dark yellow-brown color and is so concentrated that positive and negative tests are indistinguishable. When the solution was diluted down to 0.5% with distilled water, however, it became a straw color, and a drop applied to a known paper gave a distinctly blue positive result or a colorless negative result.

The results of the starch test were the most straightforward of the three tests to interpret. A blue color indicates the presence of starch. In practice a positive test shows a wide range of colors from blue to green to purplish-red, indicating starches from different plant sources as well as from different processing (Browning 1977, 88–100). In most cases, the stain of a positive test dried to a bluish color; occasionally the test showed a blue color initially and then dried colorless. These samples were retested with a more concentrated iodine-potassium iodide solution to confirm a positive or negative result.

RESULTS

Samples from all sixty books were tested for protein, either with the biuret or the ninhydrin tests; some samples were tested with both. Positive tests for protein (represented in dark hatching in fig. 4) are most prominent in the early part of the century from 1790 to 1839, while negative tests dominate the latter part of the century from 1870 to 1910. The latest samples that tested positive for protein were from two books published in 1866. Forty-five of the sixty samples, representing all the decades, were tested for starch; fewer tests were performed on papers from earlier decades. Starch was detected in a sample as early as 1837, and most of the positive tests for starch (represented in light hatching in fig. 4) were in books published after 1870.





LEFT TO RIGHT

Fig. 2. The ninhydrin test on unheated (NH) and heated (H) samples after two hours. At the left is blotting paper testing slightly positive for protein due to handling; at the right is a gelatin-sized paper showing a strong positive test for protein. The heated tests show a stronger color change than the unheated tests.

Fig. 3. The same samples as in figure 2, showing the ninhydrin test on unheated (NH) and heated (H) samples after nearly two days. Color differences between the heated and unheated tests are negligible.

DISCUSSION

While these results suggest general trends in nineteenth-century American book paper sizing, the sample set is too small and too few tests were completed for the results to be statistically significant. To be more confident in the trends, it is necessary to increase the sample set, as well as to increase the repetitions of each test on each sample. It could also be useful to perform both the biuret and ninhydrin tests on all samples in order to compare the results. Another inconsistency in the experiment as performed is the fact that about half the sample papers were tested directly on intact pages, while the other half were tested as fiber samples. Because of the significant differences in procedure and interpretation between the two groups, it is possible that the results would vary if all books had been tested in exactly the same manner.

The location of the test area on the page is also a concern. The fore edge, top, and bottom margins are the areas of the page most likely handled by readers. In an attempt to avoid possible contamination by proteins transferred from skin in the past, samples were selected from the inner margin, hugging as closely to the text area as possible. Testing in the fold area was avoided, where glue from the binding might interfere with results. For paper that could be tested as intact pages, future experiments would do better to scatter multiple repeat tests across it, both in the inner margin and in the text area.

Lastly, the results of all three of these tests are based on the interpretation of colors appearing on the test papers. While interpreting colors is a simple enough task on bright white paper, it becomes increasingly difficult as the discoloration of the paper intensifies. A large proportion of the sample papers were discolored to some degree and interpretation of the results was sometimes difficult. A test that does not involve color interpretation would be useful in testing papers from this time period (Ormsby et al. 2005).

CONCLUSION

A major outcome of this sequence of tests has been the refinement of the testing procedures and the identification of areas for future research. One interesting implication of these tests is that starch, as a typical ingredient in alum-rosin internal size, can be detected with the starch test on the paper's surface. The starch test is by far the easiest of the three to perform, and the concentration of the iodine-potassium iodide test solution can be varied to give an appropriately obvious result.

The results of this research have revealed three important characteristics of nineteenth-century American book papers that impact directly on the conservation assessment of their condition and subsequent treatments, especially those that include washing and resizing book leaves.

First, by the 1850s, due to new kinds of printing presses being used to print books, the external sizing of paper with gelatin gave way in favor of the already well-established practice of internal sizing with alum-rosin. Thus machine-made news and book papers dating from about 1850 or 1860 on were probably never gelatin sized. Therefore, the gelatin resizing of washed book leaves from this period, which has been a typical treatment to replace the presumed washed-out

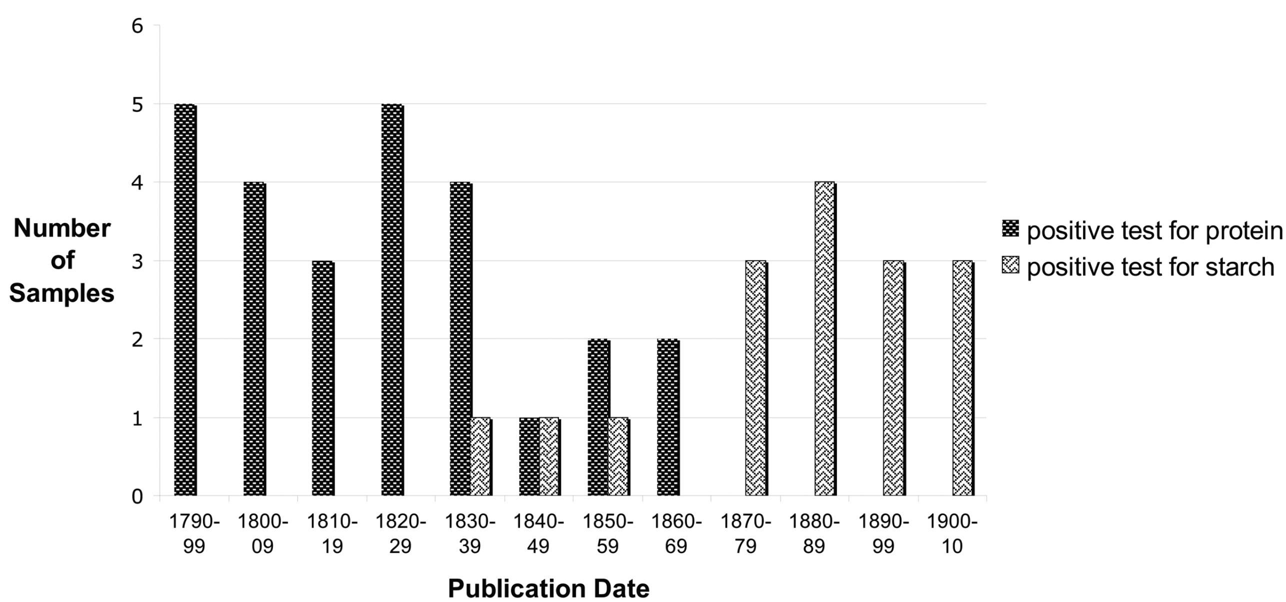


Fig. 4. Graph showing positive tests for protein and starch

gelatin, is an unnecessary treatment step. This is not to say that sizing washed book papers that remain weak is not a valid treatment, and either gelatin or an appropriate cellulose ether can be used to enhance strength and increase rattle.

Second, the presence of starch, an ingredient included in numerous alum-rosin sizing recipes, can lead to incorrect findings during certain typical analytical tests. For example, an unknown adhesive used for an overall lining or local repair undergoes an analytical test and a positive result for starch occurs. This test may actually detect the presence of starch in the sizing, not necessarily a starch-based adhesive. As a result, if a starch-specific enzyme treatment is used, it may not dissolve the adhesive but might remove a significant component of the internal sizing.

Finally, the presence of starch in alum-rosin sized papers may have led to certain kinds of discoloration, weakness, and even embrittlement that heretofore have not been considered by paper and book conservators.

ACKNOWLEDGMENTS

We gratefully acknowledge generous support for this project from a University of Michigan Librarians' Forum Research and Creative Projects grant and a Carolyn Horton Award from Foundation of the American Institute for Conservation of Historic and Artistic Works.

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Treatment of Persian Lacquer Bindings

ABSTRACT

Persian lacquer work is a method of decorative painting on a prepared surface and involves the use of shellac. From the 14th century onward a variety of decorative objects were lacquered in the Persian speaking world, most commonly pen boxes, mirror cases, and bookbindings.

The production of a Persian lacquered bookbinding is a laborious process. Layers of ground are applied by brush on the pasteboard substrate, followed by a coating of shellac. After the design is laid out, the painting is blocked out in layers of opaque paint consisting of pigments bound with animal glue. Intricate details are painted on these blocks of color. Upon completion of the painting, the entire surface is coated with shellac. Any gilding or metallic decoration is laid into this tacky shellac. The piece is finished when a smooth homogenous surface is attained after repeated applications of shellac interspersed with sanding.

The nature and construction of these materials creates many challenges for the conservator. Because the underlying structure is often misunderstood, many traditional paintings, objects, and book conservation techniques could be detrimental to the delicate painted surface. This presentation will share preliminary observations and analytical findings concerning the structure and materials used to produce Persian lacquer objects, chiefly bookbindings, and share a number of treatment approaches for the various damages associated with Persian lacquer bookbindings.

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Presented at the Book and Paper Group session, AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

An Examination of Anoxic Color Fading for Selected Gouaches, Watercolors and Textiles

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ABSTRACT

As a part of its Museum Lighting initiative, the Getty Conservation Institute explored the effects of anoxia on color fading for a wide range of colorants. Expanding upon the limited sample sets of previous research, this study attempts to further quantify the benefit and disadvantage of display in the absence of oxygen.

Following an experiment examining light-induced color fading of pigments and natural history specimens, the subsequent set focused on samples of selected gouaches, watercolors, and textiles. In addition, samples of fluorescent highlighters, ISO blue wool cards, and other fugitive pigments were included.

Samples were housed in two hermetically-sealed cases, one with an air atmosphere and the other containing less than 50 ppm of oxygen. Irradiated under a bank of MR-16 halogen lamps, temperature and relative humidity were tightly controlled by internal radiator plates connected to constant water temperature baths. The air and anoxia sets (121 samples each) were exposed for ~17.5 million lux-hours.

ΔE values (2000 calculation) were determined for each sample by pre- and post-exposure spectrophotometric analysis. The range of ΔE values observed for the air sample set ranged from 0.2 to 55, while that of the anoxia set exhibited a narrower range from 0.2 to 30. Comparing the ratio of $\Delta E_{Air}:\Delta E_{Anoxia}$ for each sample, the vast majority displayed values above 1 (indicating less fading in the absence of oxygen than in air) and most of these samples showed ratios above 2 (fading in air was 2x greater than anoxic fading). However, a number of samples had ratios below 1, indicating enhanced fading in anoxia—these samples were mostly fugitive pigments, though included were gouache samples fluorescent yellow (ratio of 0.67, ~1.5x more fading in anoxia) and orange lake light (0.91) and textile samples reseda luteola on wool (0.94) and laccifer lacca on silk (0.94).

Presented at the Book and Paper Group session, AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

REBECCA CAPUA

Material Japonisme in American Art, 1876–1925

ABSTRACT

Much has been written about *Japonisme* in America and Europe in terms of aesthetics, but comparatively little has been written about the use of actual Japanese materials by Western artists. The author examines works on paper by artists such as Arthur Wesley Dow, Arthur B. Davies, Bertha Lum, and John La Farge who all used Japanese materials such as papers, brushes, and media. Several different avenues of research are brought together, including analytical research on the materials themselves (as examined or sampled from the works on paper or historical samples), an examination of artists' techniques and their attitudes toward the use of these materials, and a discussion of the history and availability of Japanese art supplies to American artists in the late nineteenth and early twentieth centuries.

JAPONISME AND THE "JAPAN CRAZE": ARTS AND CRAFTS AND COMMERCE

For all that has been written about the influence of Japanese art on Western artists, material concerns have largely been overlooked in discussions of the era of *Japonisme* in America, and *Japonisme* tends to be observed as a singly aesthetic or stylistic concept. In examining the materials that artists actually used, and the way that these materials were obtained, marketed, and handled we will arrive a better picture what Japanese art meant to Americans and why it was so influential around the turn of the twentieth century.

The phenomenon of American *Japonisme* was as much a movement of mass-consumption of everyday objects as it was one of artistic influence within the realm of fine art. At the same time, the rise of *Japonisme* in American art largely complemented the growth of the Arts and Crafts movement of the 1890s and first decade of the 1900s, with its emphasis on the honesty of simple, natural materials and quality of craftsmanship in all types of objects. One might suppose that these two fetishized roles of Japanese goods, that of the status item for mass-consumption and that of a symbol of purity and high quality, were at odds with each other, and particularly towards the waning stages of "the Japan Craze," as the phenomenon was sometimes called, they were indeed.

Although Japan was opened to foreign trade in 1854, it was not until the Philadelphia Centennial Exhibition of 1876 that the arts of Japan truly captured the attention of the American public, long after Europeans had begun to embrace and emulate Japanese culture. The Exhibition showcased a Japanese-style "dwelling" and a bazaar, which presented and sold the types of Japanese wares that would soon become necessities in middle-class homes as displays of cultural refinement. Americans' desire for the kinds of goods presented at the Centennial, many made specifically with an American market in mind, would soon be fanned into a full-fledged "craze" through the growing influence of women's magazines filled with advice on homemaking for the aspirational middle-class wife. Although this study's main focus is on the cultural and overseas-trade centers of New York and Boston, mail-order catalogs made the acquisition of the requisite Japanese fans and umbrellas available across the country.

At the same time, Japanese works on paper were capturing the attention of the highest cultural influencers and most well-known artists. In the years following the Centennial, collectors such as Edward Sylvester Morse, William Sturgis Bigelow, and Ernest Fenollosa began traveling to Japan and amassing great collections of pottery, paintings, and woodblock prints. Woodblock prints were popular in America in part because they were quite accessible through print dealers as well as reproductions in publications like Sigfried Bing's *Artistic Japan*.[1] Furthermore, the concept of an art for the masses that was beautifully and thoughtfully designed resonated with the ideals of the Arts and Crafts movement. As we will see, it was the woodblock print that engendered the association in the American mind between Japan and high color, which had implications for the types of art supplies sold at this time.

Presented at the Book and Paper Group session, AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

PURVEYORS OF JAPANESE ARTISTS' SUPPLIES

While Japanese art supplies may have been available through American retailers, at least sporadically, from 1854 on, it was not until the late nineteenth century that interest in Japanese art warranted a steady supply in America. Papers, brushes, and inks were likely to be found in general Japanese-goods stores, as well as through general artist supply catalogs. Often, catalog entries for alleged Japanese products are very vague, such as an entry for "Japan ink" from the 1878 F. W. Devoe & Co. mail-order catalog. The composition or origin of said ink is unknown, but clearly its association with Japan had some perceived benefit to the supplier whether or not the item was authentic. The majority of the examples of Japanese art supplies that can be found in trade catalogs are from the first two decades of the twentieth century, such as a "Japanese watercolor brush" from the 1914 F.W. Devoe & Co. catalog. It seems likely that American artists who used Japanese products before around 1885 purchased them personally in Japan or in Europe.

Japanese general-goods stores, also called Japanese novelty stores or fancy-goods stores, often stocked an assortment of kakemonos, woodblock prints, and other art objects in addition to household goods. Many of these stores were founded by native Japanese and Chinese entrepreneurs, especially after immigration from Japan to the U.S. was legalized by the Japanese government in 1885. First Japan Manufacturing and Trading Company proprietor Hiromichi Shugio was a distinguished Japanese aristocrat, a prominent historian of Japanese art, print collector, and organizer of New York's influential first exhibition of Japanese ukiyo-e at the Grolier Club in 1889. His influence among prominent New Yorkers served to increase appreciation for Japanese art, which in turn, of course, benefited his business.

Another Japanese entrepreneur, E. T. Shima, advertised a variety of Japanese products sold in his New York store, including Japanese prints, pottery, and art supplies including brushes, ink sticks, ink stones, watercolors, woodblock tools, drawing and design books, and drawing paper (fig. 1). Like many contemporary art supply vendors, Shima geared his marketing largely towards schools, advertising in magazines like *School Arts*, and stressing the suitability of his products for use in the classroom.

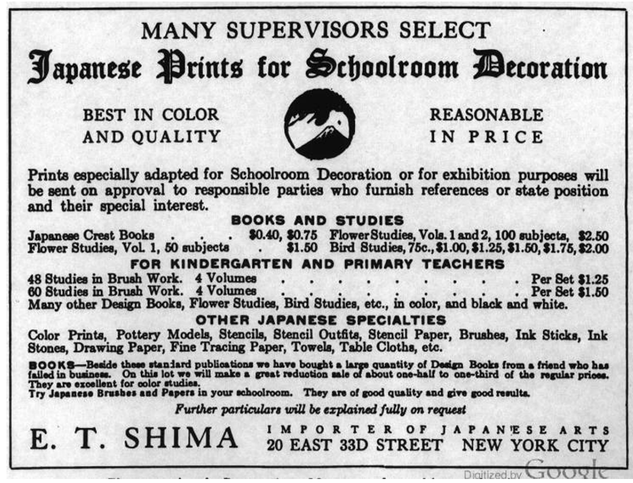


Fig. 1. Advertisement for E.T. Shima, from *School Arts Magazine*, November, 1914, p. xii

In addition to Japanese novelty stores, there were companies who imported Japanese art and craft supplies exclusively, and often these stores had a wider selection of materials. One such concern was the Japan Paper Company, which was established in New York in 1901 and eventually became the now-defunct retailer Andrews/Nelson/Whitehead. I examined a large collection of their sample books dated around 1907, which are in the library collection of the Brooklyn Museum. The accompanying literature claims that the papers would be advantageous for use in advertising, musing that "It is important that the high class commodity be advertised in a high class, distinctive manner." At this time Japanese paper was also used for printing deluxe editions of publications.

The leader in the business of importing Japanese artists' supplies was undoubtedly Bunkio Matsuki, whose store in Boston and mail-order catalog offered a wide selection of items such as brushes of various types, papers, ink sticks, patterns and stencils, design books, and silks. Matsuki's early employer, Almy's Department Store in Salem, Massachusetts, touted his personal supervision of the Japanese department as a guarantee of the products' quality and authenticity, stating in 1891, "The national pride is strong in him and there is no sturdier enemy of the Chinese-imitated Japanese wares which flood the market." In 1897, Matsuki's reputation allowed him to open his own store, ensuring customers in his catalog that:

> every article offered for sale in my store is carefully selected by myself in Japan, and I am the only dealer in art objects from the Far East in Boston who makes an annual trip to Japan at great expense to select my own goods, not having confidence in anyone to select goods for me.

In his 1904 catalog (fig. 2) Matsuki offered explanations for how to properly use the materials, and also made suggestions for which items should be used in schools. He also published and sold instructional and design books such as a study book of Japanese brush motifs, *First lessons in free-hand ink drawing (as used in Japanese schools).* This book of lithographs on Japanese paper appears to have been made in Japan circa 1900–1910, with Japanese descriptions of the subject on each page. Presumably, Matsuki simply adapted a pre-existing book used in Japanese schools with his own title page for Americans.

Matsuki also published a magazine about Japanese art called *Lotus*, which was contributed to by members of

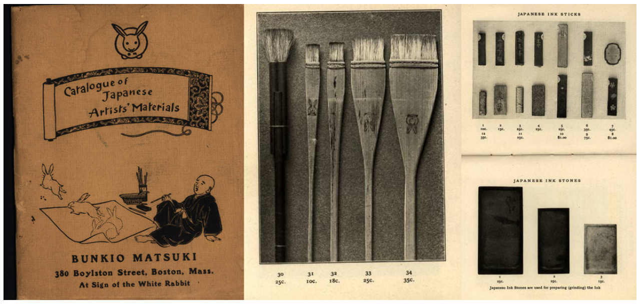


Fig. 2. Cover of catalog, Bunkio Matsuki, c. 1904, and catalog pages depicting hake brushes (stamped with Matsuki's logo), ink sticks, and ink stones

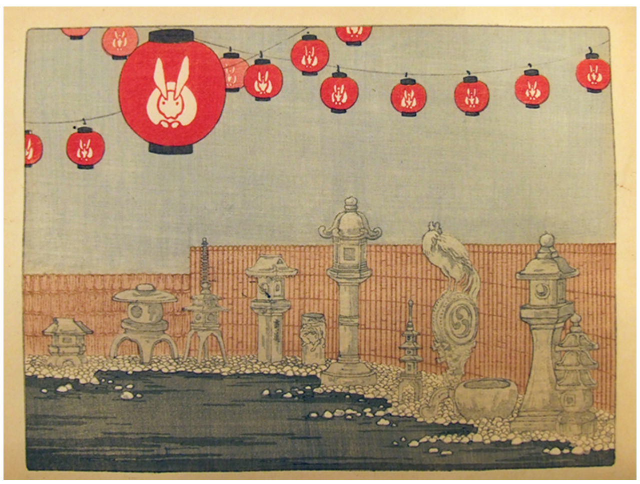


Fig. 3. Advertisement for Bunkio Matsuki, Japanese Fine Arts, c. 1904. Color woodblock print

Fig. 4. Bertha Lum, *Bamboo Road*, 1913. Color woodblock on Japanese paper. The Metropolitan Museum of Art, Harris Brisbane Dick Fund, 1924 (24.58.15). © The Metropolitan Museum of Art

the Boston circle of *Japonisme*-enthusiasts, such as Ernest Fenollosa, curator of Oriental Art at the Boston MFA, and Matsuki himself. Some of the articles, such as a 1903 piece about Japanese stone garden sculptures, were directly related to imported items sold in his store. In this woodblock-printed advertisement, small stone monuments are displayed exactly as in the photographs illustrating Matsuki's article (fig. 3), and one can also see an example of a modern business technique that Matsuki mastered: the logo, in his case that of the white rabbit. For Matsuki and many other Japanese entrepreneurs, commerce, society, and fine art existed within the same sphere.

ARTISTS' EDUCATION IN JAPAN AND AT HOME

By the turn of the century, a trip to Japan was becoming *de rigueur* for a class of Americans, particularly those with an interest in Japanese culture. John La Farge made his own trip to Japan in 1886, documented in the travel journal *An Artist's Letters from Japan*; he may have bought some supplies then, but he had also been using Japanese papers and brushes at home before that point, and it has not been documented whether he obtained them in the United States or in Europe.

Woodblock artist Bertha Lum traveled to Japan on her honeymoon in 1903, and after unsuccessfully searching for someone to teach her about the technique, on the last day of her trip she finally found a store that sold woodblock carving tools. Lum recounted to *Vogue* magazine in 1914 that on that occasion she bought thirteen tools for twenty dollars, only to find out later that she had overpaid for inferior tools and on her next trip bought fifty of the best tools for five dollars. Clearly there was a market for unsuspecting Westerners who were willing to pay exorbitant prices for "genuine" Japanese artists' supplies. During her next trip to Japan in 1907, Lum studied for fourteen weeks, first in the studio of a block cutter and then in a printer's workshop, where she gained enough mastery of the process to continue printmaking at home in Minneapolis (fig. 4). For Lum, her first naïve foray into the reclusive *mileu* of Japanese printmaking was taken as a kind of initiation, an obstacle which, when overcome, contributed to the impression of her authenticity as a practitioner of this traditional craft.

While Lum had the opportunity to gain training in Japan, artists such as Arthur Wesley Dow (fig. 5), who learned to print using Japanese materials and techniques in the United States, were likely indebted to the 1892 published report from the U.S. National Museum detailing the traditional methods of woodblock printing as described by T. Tokuno, a Japanese government official. Dow did study printmaking in Japan in 1905, but this was after he had already been using the technique for over a decade. For artists who were not able to study Japanese art in Japan, reports such as this in the service of cul-

Fig. 5. Arthur Wesley Dow, *Above the Old Bridge, Ipswitch*, c. 1893–1895, part of the series *Along the Ipswitch River*. Color woodblock print on Japanese paper. The Metropolitan Museum of Art, Gift of Mrs W. L. Putnam, 1942 (42.54.2). © The Metropolitan Museum of Art

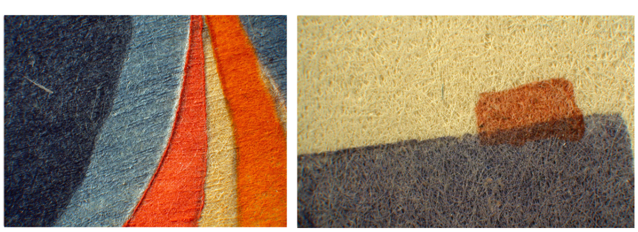


Fig. 6. Photomicrograph of Bertha Lum, *Bamboo Road* (left). Photomicrograph of Arthur Wesley Dow, *Above the Old Bridge, Ipswitch* (right)

tural and anthropological education helped encourage the use of traditional materials.

VARIATIONS ON JAPANESE TECHNIQUE

Despite the affinities that the Arts and Crafts viewpoint had with Japanese art, the two were not always compatible. Bertha Lum chose the traditional Japanese division of labor between designer and printer over the Arts and Crafts anti-industrial ideal of the hand of the artist being present at every stage of production. In her memoirs, Lum's daughter recounted how she and her mother supervised their four Japanese printers all day long in the studio.

Dow, however, wholeheartedly embraced the Arts and Crafts ethos, and mitigated the laborious process of carving and printing himself by using small blocks of pine wood (switching later to a harder maple) measuring only around seven by four inches. Lum's employment of presumably experienced printers resulted in a striking difference in appearance between her prints and Dow's (fig. 6). While both printers employed the technique of hand printing, where the Japanese *baren* is rubbed over the paper applied to the inked block, Lum's printers used much greater pressure (and possibly a more dampened paper) and imparted the texture of the wood onto the printed ink. This high level of technique produces a beautiful textural contrast between the printed areas and the fluffy reserve, resulting in an almost sculptural effect. In Dow's *Ipswitch Series* prints, there is almost no difference in the surface of printed areas verses reserve areas, and the focus is more on layered planes of transparent color.

THE AVAILABILITY AND VARIETY OF JAPANESE ARTISTS' MATERIALS: PAPER

The material that was probably most widely used with the most variety of types available was paper. There was an immense amount of variation in Japanese papers from this time with respect to fiber composition, color, and texture, both due to the use of modern Western methods of production, and one supposes, a demand for colorful and decorative papers from the American market.

Before continuing, the reader should be aware of the extreme confusion that can be encountered regarding nomenclature of papers from Asia from this period. While papers from Japan are relatively consistently named (as opposed to the term "Japan paper" which is occasionally used for a simulacrum of Japanese papers), other names are sometimes used interchangeably, even by the native cultures themselves. Take for example this explanation from the Japanese delegation's *Official Guide to the Japanese Section at the Philadelphia Centennial* on page 84: "The paper called *Chikushi*, notwithstanding that the translation of this word is bamboo paper, is not made of bamboo; the name only has been borrowed from China."

When dealing with Asian papers, especially from this time of early industry, one must consider whether the name of the paper is descriptive of the region that it is from, its appearance, composition or handling properties, or none of the above.

Decades earlier than many artists, John la Farge regularly used white Japanese paper for finished drawings and sketches dating from the 1860s up until his death in 1910. He used thin translucent Japanese tissue as tracing paper in his many window designs in which he constantly re-used photographs of models. La Farge's drawings with charcoal on thicker, soft and wooly Japanese paper took advantage of the ability of the long-fibered soft surface to hold the charcoal to create deep shadows with rich blacks, such as in his sketch of a horse's head (fig. 7). Examination under magnification shows the charcoal particles deep in the interstices of the paper surface, and also the characteristic furring of the surface from La Farge's rubbing the charcoal over it.

Fig. 7. Detail of John La Farge, *Horse's head and two riders*, undated (above). Charcoal on Japanese paper. © Columbia University, Avery Drawings and Archives Department. Photomicrograph of *Horse's head and two riders* (left)

Around the turn of the century, the variety of Japanese papers available in the United States increased to match a greater demand for colorful, unique papers. A representative collection of sample papers from the Japan Paper Company in the Brooklyn Museum's library collection, dated to around 1907, exhibit an array of colors and textures that are quite delightful. Many of the papers have large inclusions of bits of leaf or straw to create random patterning. Some sheets are dusted with mica flakes, creating a shimmery sparkly surface; Bertha Lum used a similar paper to emphasize the mystical quality of a print, titled *Procession* from 1916. Included in the sample books are also papers called "Chinese colored papers" which are dyed incredibly bright colors, and are thinner, finer, and smoother than the Japanese papers.

Japanese art and consumer products were often associated with bright colors, especially those objects made from paper such as fans, paper lanterns, umbrellas, and prints. In 1903, arts education advocate and *Japonisme*-enthusiast Henry Turner Bailey wrote that the greatest impact of *Japonisme* on American education was that of color, recalling that before the introduction of Japanese concepts, "Not only were the walls untinted, and pictures unknown, flowers never thought of, and ornaments of every kind non-existent, but every means of expression was colorless."(Bailey 1908, 35) At the same time that colorful Japanese papers became popular, a number of companies such as Milton Bradley began producing brightly colored Western papers for educational use in schools.

Arthur B. Davies was an artist who made good use of the new availability of colored Japanese papers. Davies is best known for his enigmatic painted landscapes with figures, but he also made many works on paper, rarely exhibited in his lifetime; the Metropolitan Museum has a collection of over seventy pastel drawings on mostly colored paper, the subject matter mainly landscapes or figure studies. The drawings are undated, but most are thought to be from the 1890s through the first decade of the twentieth century. Unlike some contemporary artists working in pastel (especially those working a decade or two earlier), Davies did not treat his drawings as paintings, working on canvas or paper mounted to stretched canvas, but instead preferred to work on a small scale, using the bright pure colors to create intimate, ethereal, and harmonious landscape and figure studies.

About one-fifth of the drawings are on Japanese paper; this however, is an estimate, as not all of the papers were able to be sampled, and some, like this soft navy paper, are not immediately identifiable as Japanese paper (fig. 8). Unlike watercolor, which relies on the transparency of the medium for its luminosity, pastels form an opaque light-scattering layer. They therefore are appropriate for use on colored papers, either in a more traditional method where the toned paper is used as the midtone for a drawing, or more experimentally, as

Fig. 8. Arthur B. Davies, *Mountains in Moonlight*, c. 1890–1910. Pastel on blue Japanese paper. The Metropolitan Museum of Art, Gift of A. W. Bahr, 1958 (58.21.30). © The Metropolitan Museum of Art

used by Davies, where the tone and texture adds to the overall harmony of the color relationship and composition. Textured papers at this time were also popular with artists working in pastel, both for the aesthetic effect and the increased ability to hold the applied pastel. The Japanese papers with shives and large fiber inclusions may have appealed to Davies as a more organic variety of textured papers.

Some of the colored papers that Davies used were found to be comprised solely of softwood fibers and these papers can be hard to distinguish visually from those containing Japanese fibers. Additionally, some papers that appear plainly to be Japanese (or East-Asian) actually were found to contain no Japanese papermaking fibers, only linen and wood.

The main conclusion that was reached, based on fiber analysis using polarized light microscopy and Graff's C-stain, of eighteen Japanese (or Japanese-like) papers from the 1890s through the 1910s, is that one should never assume what the fiber composition is; the results were quite diverse in terms of fiber origin and processing. Findings roughly paralleled those reported by Antoinette Dwan in her 1993 article, where she determined that three out of eight papers sold by the Japan Paper Company in 1910 contained around 15% woodpulp (Dwan 1993, 113). Six papers were found to contain either hardwood or softwood fibers. Four papers were found to have cotton fibers in addition to Japanese fibers. Nine papers had kozo fibers, and three had mitsumata fibers. The paper used for a print by Arthur Wesley Dow, which today appears somewhat yellowed and embrittled, contained all kozo fibers, but the fibers were extremely macerated, evidence of Western methods of production being used at this time by the Japanese. The paper in the poorest condition that was examined came from the aforementioned Japanese book of brush designs published and sold by Matsuki. The pages were yellowed and cracking at the gutter, as one would expect of a high lignin-containing paper;

Fig. 9. Detail, *showing paper surface*, John La Farge, *The Wheel of Fortune*, 1900. Watercolor and graphite on bamboo paper. The Metropolitan Museum of Art, Bequest of Susan Dwight Bliss, 1966 (67.55.170). © The Metropolitan Museum of Art

Fig. 10. "Peerless Japanese Transparent Watercolors" sample book and advertising literature, c. 1897

however, under polarized light magnification the fibers were identified as very macerated kozo, mixed with a comparatively large amount of chalky filler.

A number of samples of a particular paper used by John La Farge in his watercolors dating from 1887 to 1900 were also examined, and the majority of the fibers were determined to be bamboo (fig. 9). The use of bamboo was advocated by some western papermakers such as English millowner Thomas Routledge, partly due to fear of an impending pulp-wood famine, but the fiber never became widely used in the West. Like Japanese paper, the surface is smooth but very absorbent, which allowed La Farge to apply layer upon layer of color without pooling.

COLORED MEDIA: WATERCOLORS AND DYES

Another example of the influence of Japanese art on American concepts of color is this product, "Peerless Japanese Transparent Watercolors" made by the Japanese Water Color Company (fig. 10). This sample book dates to around 1897, but the company itself was founded in 1885,

and interestingly, still exists today, selling very much the same product, although with "Japanese" dropped from its name. The product is a booklet of paper strips coated with a film of transparent watercolor, off of which, according to the instructions in the booklet, one would snip a piece and place it in a shallow dish of water, thereby easily achieving a transparent wash for coloring photographs or other purposes.

What is more interesting than the actual item is the erroneous association of this product with Japan. The Japanese inscriptions found in the margins of the booklet pages are meaningless, derived at some point from an example of Japanese writing but certainly not by someone who spoke Japanese.[2] The English text explains the "Japanese" quality of the watercolor this way, perhaps referring to the subtle tonal gradations of woodblock prints: "The art of transparent tinting had its origin in Japan and the wonderful skill of the Japanese artists in this line of work has excited universal admiration." Thus, in this case, the idea of *Japonisme* is used merely as a marketing strategy, cautioning us to carefully consider the veracity of such claims regarding other materials.

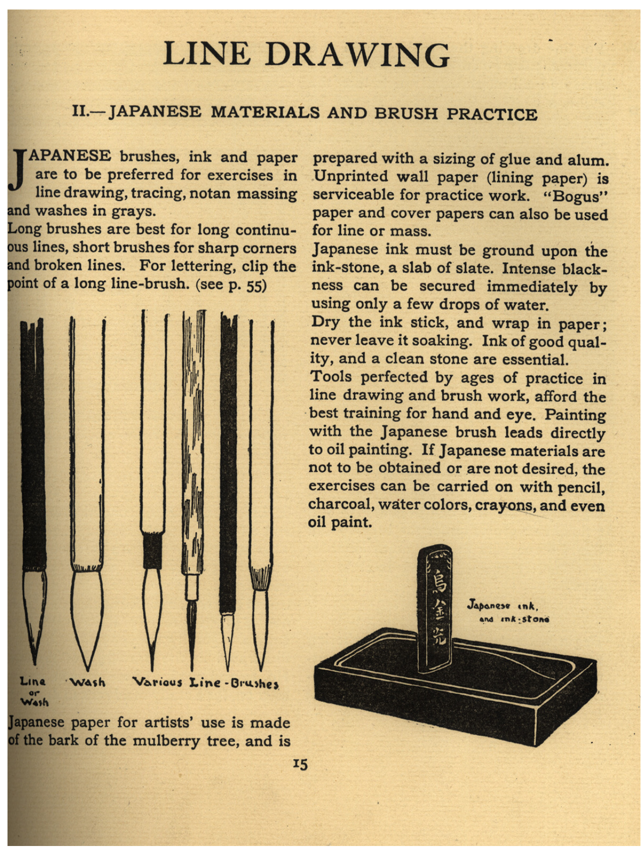
With regard to pigments and dyes, research was restricted mainly to determining colorants present in the blue papers examined. Three types of analysis were used in pursuit of this question: polarized light microscopy, XRF spectroscopy[3] and RAMAN spectroscopy[4]. Even though the range of blue hues was broad, the results were similar, all the papers being colored with Prussian blue rather than traditional indigo. The RAMAN analysis of a sample of a bright blue "*Koijio*" paper from the Japan Paper Company sample book determined the dye to be Prussian blue, although it has not yet been determined whether an aniline dye had been used in addition, as was often the case at this time. Purple dye was observed under polarized light magnification in addition to blue dye in the *Koijio* fibers.

XRF was used to analyze two papers used by Arthur B. Davies. The first was the navy-blue paper, and the second was a thin turquoise laid paper which closely resembled the Chinese Colored Papers from the Japan Paper Company. Similar results were obtained for both sheets: both contained iron, chrome, and lead, which seems to indicate a mixture of lead chromate and Prussian blue. Both pulp dyes were common for paper in the late nineteenth and early twentieth century, as was the mixture of them, called chrome green, as explained in 1901 by Julius Erfurt in his comprehensive book *Dyeing of Paper Pulp*.

JAPONISME AND THE ARTS EDUCATION REFORM MOVEMENT

As has been touched upon somewhat peripherally, both the explosion of *Japonisme* and the rise of the Arts and Crafts aesthetic were hugely influential on a reformulation of the concept of arts education across the country. The movement was grounded not only in artistic theory, but in a belief in the benefit of an aesthetic education for future generations of Americans, both for the development of a more harmonious, moral, and cultivated society, and for a stronger industrial economy.

Arthur Wesley Dow was an art teacher himself at the Pratt Institute (1895–1903), and other institutions, as well as a teacher of teachers at Columbia University (1904–1922). Dow synthesized the new approach to teaching art by emphasizing creativity, thoughtfulness, and craft with an understanding, foremost among his contemporaries, of Japanese art and materials as conducive to those values. Dow recommended that students use a Japanese brush and papers, largely because he believed that the Japanese artist was closer in his approach to this full appreciation of beauty and nature than the Western artist (fig. 11). Dow explains in his influential book, *Composition*, first published in 1899, that the Japanese brush "responds most readily, gives the widest range of quality, and tends soonest to make the hand obey the will..." (Dow 1903, 8). We see here a value, in addition to artistic capability, applied to the brush: a value of honesty, insofar as the Japanese brush is the most able to authentically and simply represent the artist's thoughts.



LINE DRAWING

II.—JAPANESE MATERIALS AND BRUSH PRACTICE

JAPANESE brushes, ink and paper are to be preferred for exercises in line drawing, tracing, notan massing and washes in grays.

Long brushes are best for long continuous lines, short brushes for sharp corners and broken lines. For lettering, clip the point of a long line-brush. (see p. 55)

Line or Wash — Wash — Various Line-Brushes

Japanese paper for artists' use is made of the bark of the mulberry tree, and is prepared with a sizing of glue and alum. Unprinted wall paper (lining paper) is serviceable for practice work. "Bogus" paper and cover papers can also be used for line or mass.

Japanese ink must be ground upon the ink-stone, a slab of slate. Intense blackness can be secured immediately by using only a few drops of water.

Dry the ink stick, and wrap in paper; never leave it soaking. Ink of good quality, and a clean stone are essential.

Tools perfected by ages of practice in line drawing and brush work, afford the best training for hand and eye. Painting with the Japanese brush leads directly to oil painting. If Japanese materials are not to be obtained or are not desired, the exercises can be carried on with pencil, charcoal, water colors, crayons, and even oil paint.

Japanese ink, and ink-stone

15

Fig. 11. Arthur Wesley Dow, *Composition*, 1913 ed., p. 15, illustrating the use of Japanese brushes and ink

The Prang Educational Company, founded in1882, allied itself with the methods of *Composition* and also sold Japanese art materials through its catalog for teachers. The Prang art teacher's manual recommends (using a common misnomer for *sumi* ink), in its section on materials for drawing, that "India ink of excellent quality can be obtained in sticks directly from Japanese dealers." In the section on brushes, the manual recommends a "Japanese pointed brush", and notes that:

> The constant and laborious drill which Japanese children are obliged to go through in order to learn to write their peculiar characters with ease and freedom and correctness is undoubtedly a great means in developing their mastery of the pencil and brush in their characteristic art (Clark et al. 1900, 59).

Bunkio Matsuki also directed much of his marketing effort towards teachers, and at the end of his catalog, he reveals the closeness of the relationship between education policy-makers, art institutions, and advocates and merchants of *Japonisme*, collaborators with the belief that the values of *Japonisme* would hold tangible benefits for American society. In his catalog, Matsuki quotes a letter he received from the aforementioned Henry Turner Bailey, Massachusetts state supervisor of drawing:

> It gives me pleasure to bear testimony to the great service you have rendered Art Instruction in the public schools, by making it possible to supply the pupils with reference material and objects of beauty for study at reasonable prices. Every school building in the Commonwealth ought to be furnished with a miniature art museum, containing just such beautiful things as you can supply.

JAPONISME AND THE MORAL COMMODITY

While Japanese artists' materials do have unique characteristics that make them well-suited to particular artistic pursuits, we have also seen how they were considered by Americans in relation to many other Japanese consumer products available at the time. From the perspective of a study of material culture, as opposed to an art-historical or biographical basis, the values that we have seen associated with these products mainly relate to their origin—supposedly outside the realm of the American early-industrial economy—with attendant assumptions of quality and honesty.

However, once the desire for such objects becomes common to the general public, the objects become déclassé, tacky, and perceived as dishonest and unauthentic, which was the case as the "Japan Craze" gradually became fodder for cultural satire, as it was by 1885 in England in Gilbert and Sullivan's libretto *The Mikado*. One of the early signs of this condition is the set of warnings we see, such as inferior-quality Chinese imitations of Japanese goods, with the implication that there were two classes of consumers of *Japonisme*: the informed connoisseur and the hopeless aspirant. Such changing perceptions of consumer goods speak to a society's tendency to imbue objects with morals and values, maintaining all the while the belief that the objects themselves confer these morals and values upon individuals.

NOTES

1. Bing published 36 issues of *Artistic Japan (Le Japon Artistique)* monthly between mid-1888 and mid 1891 in French, German, and English.
2. Many thanks to my colleague and native Japanese-speaker Akiko Yamazaki-Kleps for her assistance in this determination.
3. XRF spectroscopy performed by the author using Bruker ARTAX open-beam ED-XRF, at a kVof 50 and μA of 500 through air, using no filter, with a live time of 100 seconds.
4. Raman spectroscopy analyses were carried out by Silvia A. Centeno with a Renishaw System 1000 configured with a Leica DM LM microscope, notch filters and a thermoelectrically cooled charged-coupled device (CCD) detector. A 785 nm laser line was used for excitation and was focused on the different areas of the sample using the 50x objective lens of the microscope attached to the spectrometer, achieving a lateral resolution of $\sim 2\mu$m. In order to avoid changes of the sample materials due to overheating, neutral density filters were used to set the laser power at the sample to .0 mW. A 1200 lines/mm grating was used and integration times were set at 30 seconds.

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Unilateral Nuclear Magnetic Resonance Studies of Oil Stains on Paper

ABSTRACT

Oil stains can be transferred to artworks on paper through handling or close contact with an oily object or media. Sometimes it is not desirable to reduce these stains because they represent historical evidence, or are adjacent to sensitive media. When the stains are visually disruptive or the damage is recent, however, it is important to know the safest and most effective treatment options.

Unilateral NMR (Nuclear Magnetic Resonance Spectroscopy) is a novel technique that allows the measurement of NMR relaxation and diffusion parameters directly on the paper surface. NMR relaxation parameters T_1 and T_2 can be correlated to molecular size and motion and therefore they can be used to monitor the presence of large and small molecules present in the paper matrix and how these change with aging, paper type and the action of treatment. In this work we have applied unilateral NMR on a sample population prepared with five types of paper, ten different oils with iodine index ranging from 90–180 and three approaches to aging. T_2 relaxation measurements were collected and correlated to paper type, iodine index, cross-linking degree and treatment.

The potential of unilateral NMR as a tool to determine effective treatment procedures for oils stains on works of art on paper will be discussed.

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Presented at the Book and Paper Group session, AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

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Characteristics of Japanese Block Printed Books in the Edo Period: 1603–1867

INTRODUCTION

In 2003 I had an opportunity to examine Japanese bookbindings from the Edo period (1603–1867) in the Asian Division of the Library of Congress. Approximately 200 items were reviewed, and the structures, materials, and decoration of the bindings were recorded. The Edo period texts were almost exclusively produced by wood-block printing on Japanese paper bound with a side-stitch into paper covers (fig. 1). While the binding style is relatively simple and seems less diverse than western bindings, I have found wide variations in cover decoration as well as dimensions, both of which are associated with certain genres of writing. Today's scholars of Edo literature agree that students and researchers should be able to identify easily the genre of a monograph by looking at its physical appearance. Therefore, it is important for conservators to gain better understanding of this topic to avoid mistakenly ignoring or destroying such historical evidence.

This paper highlights the results of my article published in the March 2009 issue of the *Journal of the Institute of Conservation (JIC)*, formerly *The Paper Conservator* (Hioki 2009). Since limited subjects and references are included in this paper, those interested are encouraged to read the original article. In addition, two other papers in the same *JIC* issue may be of use (Munn 2009; Song 2009). They discuss traditional Eastern Asian bindings similar to the Edo books.

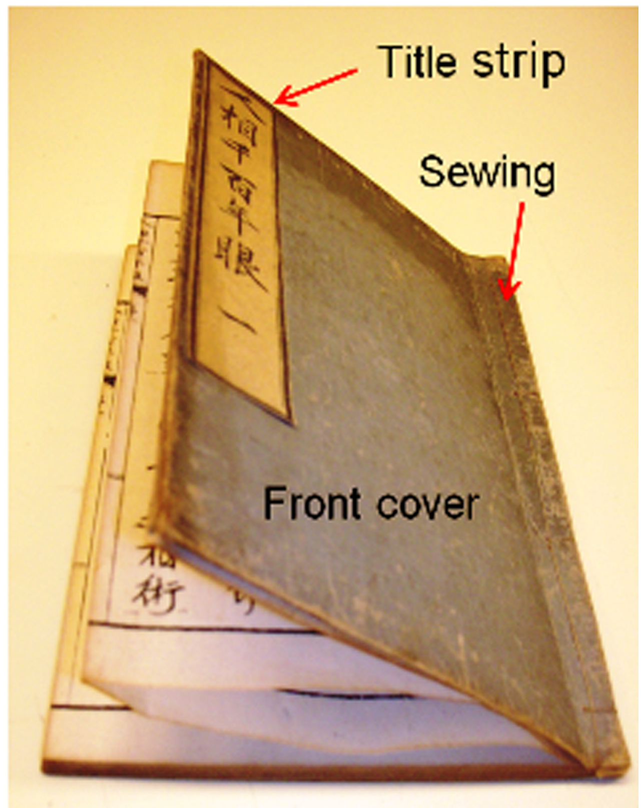

Fig. 1. Example of a side-stitched binding showing the sewn right-hand edge and folded fore-edge of the text block pages

EDO PERIOD

In 1603, Shogun Ieyasu Tokugawa (1543–1616) established hegemony over a country that had seen intermittent warfare between various provincial warlords over much of the past two centuries. The Tokugawa shogunate controlled Japan during the Edo period from 1603 to 1867, bringing stability and prosperity to the society and economy. By early 1700, the capital Edo (now Tokyo) had become the largest metropolitan center in Japan with a population of over one million.

With urbanization, economic growth, and the government policy of rule by law, literacy increased during this period. By the end of the Edo period, approximately 50–60% of the Japanese population was literate. This represented a growth of literacy that was due to the reinvention of print technology and the emergence of the commercial publishing industry.

Presented at the Book and Paper Group session, AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

WOODBLOCK PRINTING

Until the seventeenth century most Japanese texts were circulated in the form of manuscripts. Although printing had been introduced as early as the eighth century, it was practically a Buddhist monopoly for centuries. Buddhist institutions produced very small print runs using woodblocks. Around the 1590s movable type had been introduced to Japan from Europe and Korea. For a half century court aristocrats, political elites, and commercial publishers used movable type technology, and over 500 titles were printed.

However, around the 1640s woodblock printing reemerged because of its financial advantages over movable type. By 1640 there were over one hundred commercial publishers in Kyoto. By 1700 the number doubled. Publishers usually ran a small number of copies at one time and reprinted repeatedly, using the same woodblocks for an extended period—sometimes over one hundred years. The curved wooden blocks could be stored and used again as the market demanded. In addition, both a publisher's right to print and the blocks themselves had capital value, and publishers could make a profit by selling their blocks. The reversed images of both the text and illustrations were carved on the blocks. Each sheet was printed on only one side because of the thin and translucent Japanese paper. The printed paper then was folded in half so the printed side was outermost.

PAPER

During the Edo period, paper became one of the greatest sources of tax income for most local governments or *han.* Under the *han*'s monopoly system and their promotion of papermaking, paper production grew throughout the country. Before the Edo period, paper was expensive and used exclusively for writing. As the demand for paper grew, the production of mass-produced and inexpensive paper increased for different uses such as printed books, stationery, and even clothing.

A type of mass-produced, low-quality paper was recycled paper, which was made from waste paper that was supplied to papermakers by waste business communities that had developed in the slums of Edo.[1] Generally, recycled paper was employed for the covers of books. As shown in figure 2, the waste paper had not been sorted well, resulting in high content of non-paper waste such as fabric, animal hair, straw, and dust. And since techniques of ink removal were not available at the time, the final paper was usually gray in color.

BOOK SIZE

During the Edo period, paper sizes were standardized. As a result, book sizes were standardized. Paper for printed book production was consolidated into two standard sizes: the larger paper, or *mino* size paper, which measured 10–12 inches long by 15–17 inches wide; and the smaller paper, or *hanshi* size paper, which measured 9–10 inches long by 13–14 inches wide. Unlike factory-made twentieth-century paper, the standardization of paper sizes and book sizes was crude, varying slightly from one papermaker to the next. Papers for books would be either folded or cut, and they are categorised based on their dimensions. The resulting standard book sizes are shown in figure 3.

Fig. 2. Example of recycled paper used for a book cover

Generally, the publications in the early Edo period, including serious studies such as classic literature, medical texts, and Buddhist scriptures, were made in the largest size of *ō bon*, which measured approximately 10 inches long by 7 inches wide. As literacy increased, book sizes became smaller and shifted from *ō bon* size to *hanshi bon* size, which measured approximately 9 inches by 6 inches. Then, in the later Edo period, new genres of popular fiction appeared in the

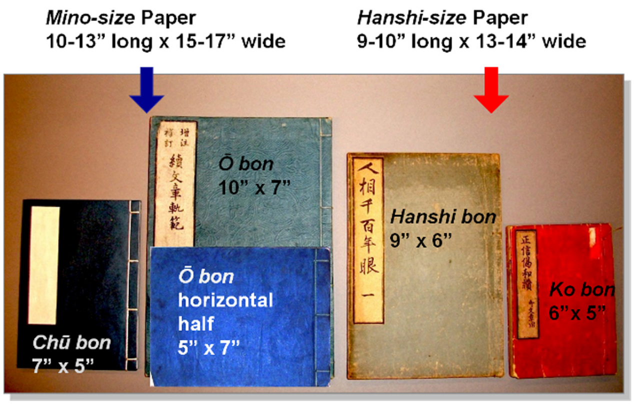

Fig. 3. Comparison of four vertical standard book sizes and one horizontal book. Ō bon (Mino sized book), chū bon (medium sized book) and yoko-chūbon (ō bon horizontal half sized book) are made from mino size paper. Hanshi bon and ko bon are made from hanshi size paper

Fig. 4. Example of a book cover construction showing the laminated layers of recycled paper and a sheet of dyed paper and turn-ins

smaller book size of *chū bon*, which measured 7 inches long by 5 inches wide.

Practical books such as how-to manuals, travel guides, and pocket books were made in the smallest size of *ko bon*, which measured approximately 6 inches long by 5 inches wide. In addition to the vertical format, horizontal books, such as *ō bon* horizontal half, which measured 5 inches long by 7 inches wide, were also often employed for practical books.

COVER CONSTRUCTION

The majority of the covers were constructed of layers of poor-quality paper—generally recycled paper—and an outermost sheet of thin, dyed paper. Layers of sheets were cut to size allowing 1–2 cm turn-ins on all sides (fig. 4).

COVER DECORATION

Before the early eighteenth century, covers usually had simple monochromatic colorsuch as brown or blue, and then brighter colors as well as cover decorations became common. A major reason for this diversity is the flourishing commercial publishing industry. At the peak of the printed culture in the eighteenth century, over 10,000 titles were published and millions of copies were circulated among the population. The total number of commercial publishers exceeded 6,000 during the Edo period. Covers were one of the most effective merchandizing tools. Eye-catching, multi-colored printed images decorated the covers of fiction books; popular *ukiyo-e* artists designed the cover illustrations.

Certain genres of text became associated with certain cover designs as well as book sizes. The below list outlines the selected cover decoration styles for commercially printed books:

- *Chestnut shell color* (*kurikawa hyōshi* 栗　表紙) covers were a plain brown color that was produced by brushing the cover with persimmon tannin juice. This was the most common cover style before 1644 and was usually found on books about Buddhism and medicine. Chestnut shell covers have a lustrous shine from the persimmon juice. Because of its association with older publications, covers from eighteenth- and nineteenth-century editions were sometimes replaced with chestnut shell-colored covers taken from other books to fake authenticity.
- *Burnished* (艶だし,空ずり) cover decoration style started in the early Edo period, and was frequently employed until the early twentieth century. In its process, the verso of a laminated paper cover was placed on a wooden block that had been carved with the cover design. The paper surface was rubbed with a smooth boar's tusk or a piece of ceramic until the burnished design was exhibited lightly on

Fig. 5. The burnished front cover on flower arrangement (left), Enshū-ryū shō fū kaku (1818?) Library of Congress, Control No: 98847039. Close-up of the burnished design (above)

the paper's surface. Various designs, usually small repeated patterns, were used (fig. 5). Burnished decoration is delicate and subtle. It was easily worn out and missed. As shown in figure 6, the recto of the cover seems to have no

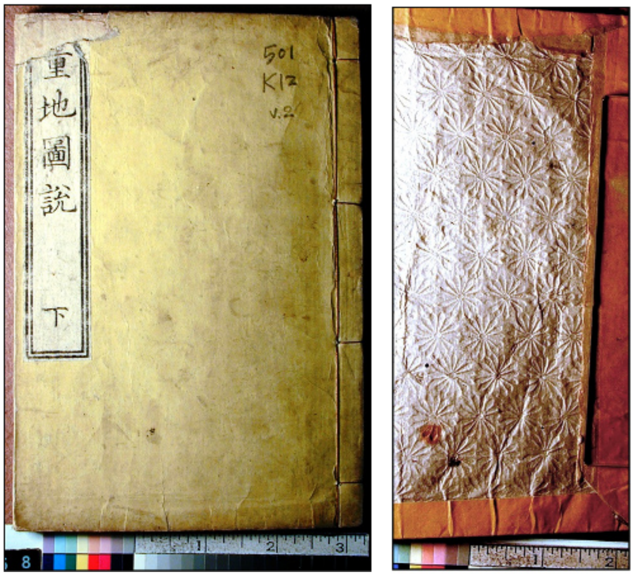

Fig. 6. Recto of burnished front cover (left) and verso of the cover (right), showing the impressions of the small flower design. Ryōchi zusetsu, Koei Kai, Edo (1852?), Library of Congress, Control No: 98847154

decoration, but the verso preserves the impression from the curved block.

- *Embossed covers* became common in the nineteenth century. The paper cover was first moistened before being placed recto-down on the carved wooden block. Pressure was applied to impress the design onto the sheet. Various designs were used, ranging from simple geometric patterns to elaborate drawings that might depict, for example, a landscape and plants (fig. 7).
- *Clove brush line* (丁子引き,刷毛引き) was a decorative cover-style in which horizontal, diagonal lines, or lattice patterns were drawn onto the cover by hand with a brush. Originally, these lines were drawn using yellowish light-brown dye made from the floral buds of cloves. Clove dye was replaced with cheaper by-products of safflower dye, and then gray dye made from ash. This decoration was considered to be one of the cheapest covers and became more common in the eighteenth century (fig. 8).
- *Colored covers*. New popular fiction genres flourished from the late seventeenth century to the end of the nineteenth century. These popular fiction genres included red-covered books, black-covered books, blue-covered books, yellow-covered books, and combined books. They were grouped by the cover color as well as content.

Red-covered books were the earliest group of popular fiction appearing in the late seventeenth century, and had plain bright-red (*niiro* 丹色) covers. The colorants

LEFT TO RIGHT

Fig. 7. Front cover embossed with plant design on a book about geography and industries, Nippon sankai meibutsu zue, Tessai Hirase (not before 1797), Library of Congress, Control No: 98847077

Fig. 8. Front cover showing clove brush line decoration of a book of Chinese poems. Kaifusō, Kyoto (1793), Library of Congress, Control No: 98847008

Fig. 9. Front cover of yellow-covered book. Kamado shougun kanryaku no maki, Hokusai Katsushika, Edo (1800?), Library of Congress, Control No: 2005550672

employed were inorganic pigments, probably red lead (Pb_3O_4) or cinnabar/vermilion composed of mercury sulphide (HgS). Red-covered books were replaced by black-covered books (colored with black carbon ink) and then by blue-covered books, which dealt with more adult subjects. The blue-covered books were replaced by yellow-covered books, which were dyed with saffron yellow or other organic yellow. Yellow-covered books signified satiric and didactic literature, and often had color-printed, illustrated title slips (fig. 9). The color of blue-covered books was actually a yellow-green made by mixing the saffron yellow of turmeric, or a variety of organic yellows, and blue dye extracted from the dayflower. The dayflower dye was so light-sensitive that it faded, leaving behind the visible yellow color only. The result is that existing blue-covered books look the same color as yellow-covered books. It is not clear why red was replaced by black and blue. One theory is that the increase in demand caused the price of the red pigments to rise, resulting in the use of cheaper, alternative organic dyes. Likewise, blue was replaced by yellow probably because the extra step for mixing two colors (yellow and blue dyes) to make blue was eliminated as the demand increased.

The stories, which had consisted of two or three fascicles in yellow-covered books, were gradually lengthened and the number of fascicles in a story increased. In order to minimize the binding cost, several fascicles were bound together to form a combined book (fig. 10). The illustrated title strips used for yellow-covered books gave way to multi-colored, woodblock-illustrated covers to eliminate the extra step of placing title strips on the cover.

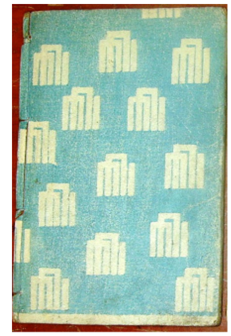

Fig. 10. Front (left) and back (above) covers of combined book. Fūzoku kingyo den, Bakin Takizawa, Edo (1852), Library of Congress, Control No: 98847023

POPULAR FICTION

Books defined as popular fiction, including red-, black-, blue-, yellow-covered, and combined books, were illustrated novels with illustrations on each page. Their text filled the blank spaces surrounding the illustrations, resembling the comic books of today. Texts run vertically. They use little symbols that help readers navigate to the next scene (fig. 11). The monographs appealed to those who had little ability to read.

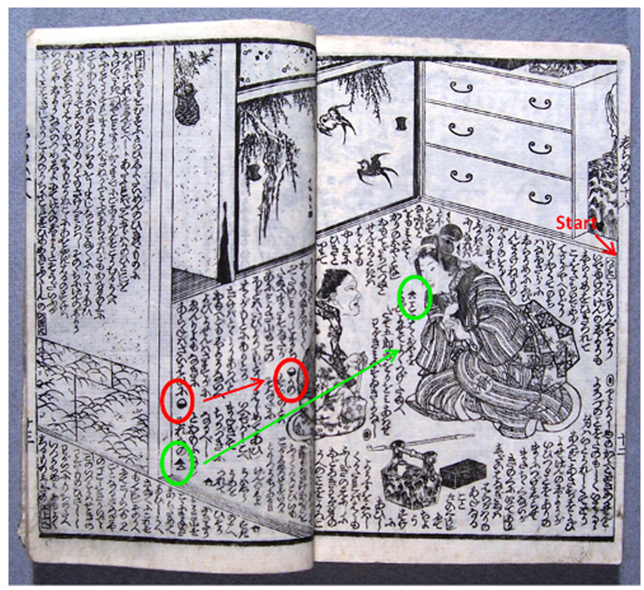

Fig. 11. Text pages of combined book showing the closer visual relationship between the illustrations and text. Text starts at the right top corner and runs vertically. The symbols in circles navigate the readers to the next sentence (see the arrows.) Shiranui monogatari, 17 hen, Tanekazu Ryukatei, Edo, Library of Congress

The illustrated popular fictions were revolutionary publications in book marketing. Before these, most books had been borrowed from lending libraries because of their high cost. The average price for one fascicle of a classical text was $58 in today's converted value. By contrast, a fascicle of a yellow-covered book cost $3 on average, which was affordable for average Edo citizens. The publishers' major clients shifted from dozens or hundreds of lending libraries to thousands of individual customers. The publishers' strategy in the marketing of popular fiction was to achieve low price and high volume, and they published thousands of copies at one time until the woodblocks were worn out, and then made new blocks to continuously release up-to-date texts in fresh and current styles. In order to minimize the cost, the book dimensions became smaller and thinner. Popular fiction was issued in the small *chū bon* size (7 inches long by 5 inches wide), with only five sheets (or 10 pages) to a single fas cicle. After they had been bought and read, these popular fictions were often discarded. The cover was usually made of a single sheet which was trimmed on three or four sides to match the text size.

The combined books, which were serially published, were the last group of popular fiction. Covers to sequential fascicles could be laid side by side to create a panoramic image or scene. Usually two or three fascicles could be joined to create a diptych or triptych. One fascicle alone gave no clue to the scene, and urged the customer to buy the next volume. It was a clever marketing tactic (fig. 12). The publishers often employed a popular artist to design the covers, and printed the artist's name on the cover along with the names of the author and the publisher. In this manner, their names and brands started to bear commercial values (fig. 13).

Fig. 12. Front covers of two fascicles of combined book (left : jo or 1st volume, right: ge or 2nd volume). Shiranui monogatari, 17 hen (jo and ge), Tanekazu Ryukatei, Edo, Library of Congress

Fig. 13. Front covers of two fascicles of combined book showing the names of the artist and author in yellow-circle as well as publisher's name in dash-circle. Kantan shokoku monogatari, 19 hen (jo and ge), Senka Ryutei, Edo (1834–56), Library of Congress, Control No: 00714383

The combined books continued into the 1880s until they were replaced by newspaper novels printed using new western-style metal movable type. By 1890 movable type and the mechanized press ended the dominance of woodblock printing. Binding practices also changed from the soft cover side-stitched binding to the hard cover western binding.

CONCLUSION

The construction and design of a printed Edo book reflect its development, readership, and content, and inform us about the society and culture of Edo. The decrease in book size and the development of a more appealing appearance reflected the increase of readership and the rise in competition within the publishing industry. The association of certain genres with certain book dimensions and cover designs was deeply rooted in the society of Edo. In this society, formalism and appearance embodied content and spirit.

This study has only touched the surface of the vast subject of printed Edo books.

There are many topics that need further research, such as recycled paper production including its process and makers. It is hoped that this paper will encourage further study of the printed book during the Edo period.

ACKNOWLEDGEMENTS

I would like to thank the staff in the Asian Division and Conservation Division in the Library of Congress for their support of my research. My greatest thanks go to Jesse Munn for her inspiration, encouragement and help on this project.

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NOTE

1. Waste business communities were made up of a class of people called *eta, hinin* or "the humble," who lived in the slums of Edo and engaged in waste collection, including waste paper. Their trash business was organized under government control, and the gathered waste

paper was sold to the makers of recycled paper. There were a couple of recycled papermaking villages (communities) in the Edo region. These villages were located very close to the waste business communities (waste paper suppliers).

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New Directions in the Non-Destructive Analysis of Watercolors by John Marin

ABSTRACT

The authors will discuss the application of newly developed analytical techniques to characterize colorants in eighteen watercolors by the American modernist artist John Marin, painted between 1895 and 1929.

This presentation will elaborate on the technical examination of Marin's watercolors presented at the AIC meeting in 2005 which focused on the artist's evolving palette, influenced by prevailing color theories and corresponding developments in optical science. Preliminary analysis for that study using air-path X-Ray fluorescence spectroscopy, color spectroscopy, infrared reflectography, and visual examination with ultraviolet radiation will be integrated with data obtained from relatively newer technologies now being developed for the analysis of works on paper such as X-Ray fluorescence spectroscopy using a helium flush and imaging spectrometry.

The qualitative evaluation of colorant responses to ultraviolet and infrared radiation in the previous study have been superseded by hyperspectral and fluorescence emission imaging techniques that gather narrow band reflectance and luminescence data, allowing the resolution of colorants with similar spectral behaviors, the discrimination of colorants in mixtures, and their spatial distribution across a work to be examined. XRF employing a helium flush has the advantage over air-path XRF in that it eliminates spectral noise and identifies atomic weight elements below potassium, enabling the characterization of mordants and fillers used in the manufacture of watercolors.

These new analysis techniques not only offer the promise of a considerably richer understanding of John Marin's color palette as his technical abilities matured, but may also provide a template of options for colorant analysis on art and historic artifacts where sampling is not permitted.

Presented at the Book and Paper Group session, AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

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A Discussion of the Conservation of "SS-Hygiene Institute" Documents in the Archives of the Auschwitz-Birkenau State Museum

The Auschwitz-Birkenau State Museum, founded on July 2, 1947, has been included in the UNESCO list of World Heritage Sites since 1979.[1] There are currently 878 cultural and natural heritage sites in 145 countries on the UNESCO list. Only twenty of these sites relate to the twentieth century and two of them are monuments to the deaths of people. One of the two monuments is Auschwitz-Birkenau German Nazi Concentration and Extermination Camp (1940–1945) and the other is Hiroshima Peace Memorial (Genbaku Dome). As places of horror and modern barbarism, both are on the list as "representatives" for other burying sites of crimes against humanity. The documents in the archive of the Auschwitz-Birkenau State Museum are also included in the UNESCO list as they are the material legacy of the commemorated period.

There has been another UNESCO program since 1992 termed Memory of the World (MOW).[2] The MOW register contains documents from all over the world. Among these, seven are from Poland and include the Warsaw Ghetto Archives and Solidarność's *Twenty-One Demands.*[3] The aim of the MOW program is to ensure accessibility to culturally and historically significant documents and prevent evidence of outstanding value from disappearing into a collective oblivion or falling victim to destruction.

The Polish National Committee has also included the archive collections of the Auschwitz-Birkenau State Museum on its national list. The documents from the archives are currently housed in the Auschwitz-Birkenau State Museum's archives. The museum is on the site of the former extermination and concentration camp Auschwitz-Birkenau. Camp documents, documents filed by concentration camp prisoners, letters sent from the camp, notes, papers documenting crimes of the Nazi staff within Auschwitz, and other documents such as the files of the Institute of Hygiene of the Waffen-SS are stored in this archive. In order to cover the tracks of the crimes, starting in the third quarter of 1944 the camp authorities ordered the destruction and deportation of most of the documents issued by the SS administration.

Fig. 1. Auschwitz-Birkenau State Museum

The systematic concealment of documents was initially carried out in a rather slow and secret way though it accelerated in the final days of the camp with the open burning

Presented at the Book and Paper Group session, AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

of documents. The chaotic nature of such public burnings allowed some prisoners to get hold of a few documents and hide them from the eyes of their camp guards. Although many written materials could have been saved from the fire, approximately 95% of all documents filed and papers written during the existence of the camp in Auschwitz-Birkenau fell victim to the systematic elimination of documentary evidence within the concentration and extermination camp.[4]

A few weeks after the liberation of Auschwitz-Birkenau by troops of the Red Army on January 27, 1945, members of a Soviet special committee began collecting the camp's remaining documents.[5] Some of them were found in abandoned buildings while others, having been blown by the wind across the compound during the burnings, needed to be rescued from the surrounding marsh. After the termination of the committee's work, the saved documents were brought to Moscow. Two Polish committees were set up to further ensure the safety of the documents and other tangible assets. In February 1957 the museum's archive started to function as an independent department. It was not until 1961 that the Main Committee for the Investigation of Nazi Crimes in Poland handed over the documents of the Institute of Hygiene of the Waffen-SS to be archived in the Auschwitz-Birkenau State Museum.

The Institute of Hygiene had been headquartered in the concentration camp Auschwitz I until spring 1943 when it was relocated to a place called Rajsko, five kilometers away from Auschwitz. The activities of the Institute of Hygiene centered around medical analysis for the SS, the Wehrmacht, the police force, and the concentration camp (including Joseph Mengele, the doctor notorious for his experiments on humans). Laboratory examinations from the Institute of Hygiene included samples of urine, blood, stool, sputum, and throat swabs. The documents from the Institute in the collection of the archive contain approximately 40,000 files from the period between April 10, 1943 and January 12, 1945, of which nine volumes are general ledgers, eight volumes are subsidiary ledgers, and the rest of the papers are arranged in sixty-two folders.

The documents allow for insights into the inner workings of the Institute of Hygiene. They include notes about treated prisoners of Auschwitz and its satellite camps as well as members of the SS troops stationed at Auschwitz. Frequently, these documents have turned out to be the only written record remaining about prisoners. Beyond that, there is also information about members of the administrative ranks of the Institute of Hygiene of the Waffen-SS and prisoners employed there as aides. Furthermore, the documents contain detailed lists of materials and devices ordered by the Institute of Hygiene of the Waffen-SS.

Currently, there are 40,000 documents from the Institute in the restoration workshop. The three-year conservation project started in January 2008. The German state of North Rhine-Westphalia provides funding (€ 630,000) and support for the project. Several conservators from the restoration workshop in the Auschwitz-Birkenau State Museum are there to work on the conservation of the documents. Besides Polish conservators, who are permanently engaged in the restoration workshop, some members of the project team are from Germany.

In order to develop a conservation protocol for the 40,000 documents, conservators of the Auschwitz-Birkenau State Museum tested the proposed treatments on 245 documents contained in one folder. All papers were heavily yellowed and brittle, and they contained wood pulp. The pH value of the sheets was between 4.4 and 5.2. The major problem in applying the aqueous immersion treatments or alkalization of the sheets was the water-soluble aniline inks found on almost all documents in the form of identification markings and stamps, all in various colors. In one folder the conservators examined there were the usual printing inks, a number of colored stamps, and colored pencils. No less than forty items had multiple colors. Some of the documents are typescript while others are carbon copies. As shown by the preliminary tests, the majority of the written material needed to be saved for aqueous immersion treatments. The adhesive strength of writing or stamping inks, which were fundamentally vulnerable to aqueous immersion treatments, depended on the condition of the color and its ionic charge (either anionic or cationic), the consistency of the paper, the thickness of the ink, and how the aqueous immersion treatment was applied.

It proved to be particularly difficult to find a means of fixation that ensured the fastness of all inks. The tests made by conservators, as well as the exact procedures of the conservation process, have been published in the *Polish Biuletyn Informacyjny Konserwatorów Dzieł Sztuki*.[6] The fixative Standard Suspension (Neschen), sometimes used with cyclododecane, showed the best results. Mesitol NBS and Rewin EL were also tested.

The documents also showed signs of mechanical damage most likely from being rolled. The wear was indicated by torn strips of paper on the back of the documents. Today we cannot really reconstruct whether the rolling of sheets was due to space restrictions on transportation or storage. The sheets showed material losses and cracks, especially around the punch holes. The documents were stained and partly covered with pressure-sensitive tapes and adhesive residues. Some areas even displayed rust spots caused by paper fasteners. The printing ink, writing inks, and several other means of writing are in relatively sound condition with only a few visible spots of minor blurring.

The treatment designed by the conservators at the Auschwitz-Birkenau State Museum utilized their testing results, suggesting that individually applied aqueous immersion treatments and alkalization were adequate techniques to

save all of the 40,000 documents. The fixation of the different types of writing inks was followed by a washing process. The sheets then were immersed into water enriched with magnesium carbonate. If the sheets were still moist, losses were repaired and supported by lining Japanese paper on the back of the document. Once cyclododecane sublimated, resizing the paper with methyl cellulose MH-300 in water enriched with magnesium carbonate was carried out via airbrushing. The slightly damp sheets were pressed between blotters for a final drying. The documents were stored in acid-free folders.

It is a novelty that such a conservation effort has been made possible at the Auschwitz-Birkenau State Museum. A profound knowledge of the historical facts surrounding the objects is as important as the elaboration of item-level treatment. The Auschwitz-Birkenau State Museum rejects mass conservation techniques typically applied to archival collections. The State Museum considers their documents too important to be exposed to the flaws associated with mass conservation efforts. To avoid these pitfalls, every single document requires an individually applied conservation treatment. Beginning in the 1990s the Auschwitz-Birkenau State Museum entrusted private restoration workshops with the conservation of their archive collections as there was no on-site restoration workshop at this time. This changed in 2003 when highly qualified conservators were hired to found a restoration workshop on the museum's compound with the help of the U.S.-based Roland S. Lauder Foundation, which donated $2.7 million for the cause. Besides its primary focus on the conservation of objects, the workshop also serves didactic ends. Visitors to the museum such as groups of school kids, soldiers, and others have a chance to take a guided tour of the workshop where they are introduced to the conservation process. This visualization of conservation work makes visitors realize the acuteness and the enormous effort necessary for the restoration of the documents. The material legacy of Auschwitz-Birkenau in the State Museum must be treated individually and with utmost care.

CONCLUSION

The conservation of documents at Auschwitz-Birkenau State Museum is about more than simply fixing sheets paper. It is, above all, about the safeguarding of original documents that provide us with evidence of crimes against humanity.

NOTES

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Where Archival and Fine Art Conservation Meet: Applying Iron Gall Ink Antioxidant and Deacidification Treatments to Corrosive Copper Watercolors

ABSTRACT

Iron-gall ink accelerates both acid hydrolysis and oxidation of cellulose. Stabilization of ink-on-paper artifacts must therefore use both deacidification and antioxidant treatments (chelating agents, radical scavengers, or peroxide decomposers). Calcium phytate (CaP), a chelating agent for iron (II), and calcium bicarbonate (CaB) is an effective treatment for corrosive iron-gall inks; however, this treatment must be applied aqueously and does not address the corrosion caused by other transition metal ions in the inks. New antioxidant treatments, including peroxide decomposer tetra-butyl ammonium bromide (TBAB), are emerging in the literature. TBAB is advantageous, as it is not metal specific and can be applied non-aqueously.

Antioxidants have been studied primarily for use in archival conservation; their use in fine art conservation has not been fully investigated. This research examined the effects of antioxidant treatments on metal gall inks and associated media found in fine art on paper. Ideally fine art media would be unaffected during treatment or perhaps even stabilized by the antioxidants. Two common copper-containing pigments (verdigris and azurite) were studied in a watercolor medium alongside samples of two laboratory-prepared inks (pure iron-gall ink and a mixed iron-copper gall ink). Accelerated aging allowed monitoring of the treatments over time. Results showed that all treatments produced initial color changes in all four media. Under the studied aging conditions, verdigris stability was not greatly increased, but neither was it decreased relative to the untreated control. Generally, however, treatment enhanced the stability of the two inks and the azurite watercolors.

INTRODUCTION: PROJECT IMPETUS AND SCOPE

This paper is based on research conducted in the final year of studies in the Master's of Art Conservation Program at Queen's University in Kingston, Ontario, Canada, completed in 2007. The project began with a problem encountered during a summer internship at Library and Archives Canada: how does one treat corrosive iron-gall ink inscriptions on the backs of watercolor paintings? Over the last ten to fifteen years, a great deal of research has been performed investigating the stabilization of iron-gall ink with combined antioxidant and deacidification treatments. The calcium phytate (CaP) treatment has emerged at the forefront of these investigations (Neevel 2002). Its effects have been well studied with regards to both the ink and its paper substrate for archival documents, but not as to the effects on other surrounding media found in fine art. The question of whether such treatments can be safely used for watercolors warrants further consideration.

In addition, there was the desire to investigate an exciting emerging treatment, tetra butyl ammonium bromide (TBAB). This antioxidant has the potential to affect positive change in corrosion caused not only by iron ions, but also by other transition metal catalysts, in particular copper-induced corrosion. Copper corrosion actually links iron-gall ink and watercolors quite closely. Copper ions are found in iron-gall inks either as impurities in iron salts, or in copper salts called for as individual ingredients in many historic recipes. Similarly in watercolors, several well known pigments, such as malachite, azurite, and verdigris are copper-containing minerals. The least stable of these pigments, verdigris, exhibits well-documented corrosion characteristics very similar to those of iron-gall ink. The parallel nature of verdigris and ink degradation led to the idea that perhaps corrosive copper watercolors would not only be "safe" in iron-gall ink treatments, but also stabilized by these same interventions.

Embarking on a project that takes archival treatments and applies them to fine art objects means that it is necessary to consider the differences in methodology. In archival conservation the ultimate goal is long-term stabilization in order to retain the information. In fine art conservation the artist's intent and the aesthetics of the object must play a much more vital role. Slight color changes that may be acceptable

Presented at the Book and Paper Group session, AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

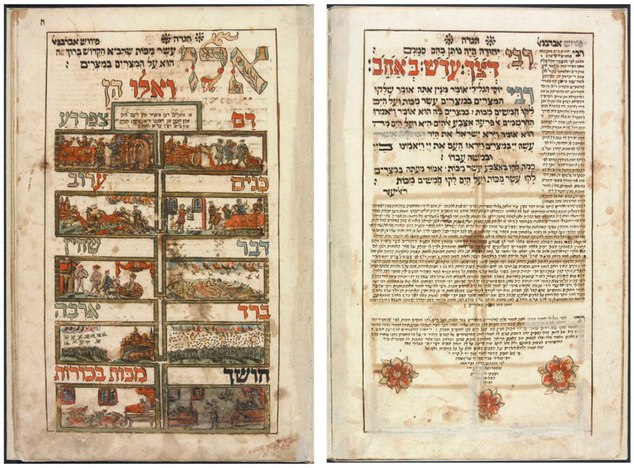


Fig. 1. Recto and verso of a Haggadah manuscript page from Library and Archives Canada, showing both verdigris and ink corrosion (Acc # LOWY MS A229)

in archival conservation become unthinkable in fine art conservation. So called "more invasive" bench treatments may be set aside in favor of more ideal storage and other preventive measures. In cases such as media corrosion, however, the necessity for stabilization is paramount to all objects lest they disintegrate beyond repair. The manuscript page in figure 1 suffers from both verdigris and ink corrosion. Such an artifact would benefit greatly if a successful antioxidant and deacidification treatment could be formulated for both inks and watercolors.

After establishing the basis for the research, the project aims were simply stated in the form of three questions:

- Can mixed media drawings that include both inks and watercolors be safely treated by iron-gall ink treatments (that is, without unacceptable change to the media)?
- Are corrosive copper watercolor pigments and iron-gall inks stabilized by the same treatments?
- Ultimately, are TBAB and CaP treatments effective in fine arts settings?

The theoretical scope of the project is quite large, encompassing the application of several different antioxidant treatments to both inks and watercolors. The actual avenues of investigation were narrowed to two main paths:

1. Testing the iron-gall ink treatments on two copper watercolors (azurite and verdigris) to assess the potential for successful treatment—both aesthetically and in terms of stability.
2. Further testing of the new emerging TBAB treatment on two laboratory prepared inks (one containing only iron, the other containing both iron and copper). TBAB treatment would be compared to the established CaP treatment. The testing of these inks would also serve as a comparison for the magnitude of color change observed in the inks relative to the color change seen in watercolors to help establish acceptable levels of change.

This paper will focus on the results from the first objective, touching only lightly upon the second objective. Despite the emphasis on the watercolors rather than the inks, the chemistry and composition of both media types will be discussed below. In trying to develop treatment methodologies, it is important to understand the commonalities between the degradation of inks and the watercolors.

MATERIALS: PROPERTIES, COMPOSITION, AND CHEMISTRY

Watercolors

Watercolor paints consist of four main ingredients (Cohn 1977; Stephenson 1984). A binder, usually gum arabic, also serves to increase the luster of the media. Next, the pigment imparts color. Almost every known artist pigment (organic or inorganic, natural or synthetic) has been employed in watercolor paints. The right pigment-to-binder ratio (usually close to 1:1) is imperative for the correct working properties of the watercolors. Too much binder makes the paint glassy and likely to crack. On the other hand, too little binder leaves the pigments friable and vulnerable to flaking or abrasion. A third minor ingredient is a humectant, such as honey, sugar, or glycerin. The humectant is a hydrophilic substance that allows the watercolor paints to be remoistened and reworked. The final common ingredient is ox gall, a natural wetting agent that breaks up particle agglomerates and helps the paint flow across the paper.

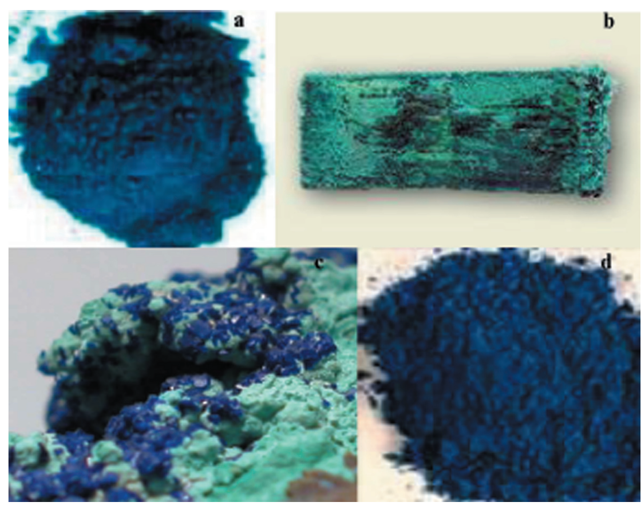


Fig. 2. a) powdered verdigris, b) verdigris as a copper corrosion product, c) azurite (blue) and malachite (green) minerals, and d) powdered azurite

The two pigments chosen for this study are inorganic, copper-containing pigments (fig. 2). Both verdigris, a green pigment, and azurite, a blue pigment, have been known since medieval times, and have been employed in both manuscripts and paintings. The first pigment, verdigris, is a basic acetate of copper. This substance is a corrosion product of copper, and can be made by exposing metallic copper to vinegar (acetic acid) in the presence of oxygen (Bhowmik 1970). The pigment has been historically prepared in a number of ways, and takes numerous, slightly different chemical forms with x, y, and z forming various ratios: $[Cu(CH_3COO)_2]_x \cdot [Cu(OH)_2]_y \cdot zH_2O$ (Kuhn 1993). Verdigris was historically one of the first green pigments and, despite being quite unstable, had very wide application (Gettens and Stout 1942). This green is known to cause paper degradation in a manner similar to iron-gall ink. Initially, the pigmentation seems to migrate to the verso of the support, staining it green. Next, the paper becomes brown. This browning begins locally at the pigmented areas, but spreads as a halo. The green of the pigment decreases as the browning spreads, disappearing entirely after a time. The paper finally becomes so brittle that it fragments and perforates (Mairinger et al. 1980).

The second pigment, azurite, is a natural mineral pigment. A blue basic copper carbonate, it is mined side by side with malachite (a green basic copper carbonate) (Hagadorn 2004). Indeed, azurite exists in a reversible equilibrium with malachite. Unstable under atmospheric conditions, azurite slowly converts to malachite over time (Banik 1989): $Cu_2(OH)_2CO_3$ (malachite) + $CO_2(g) \leftrightarrow 2Cu_3(OH)_2(CO_3)_2$ (azurite) + $H_2O(l)$. Transition metal catalysis occurs in solution with moisture provided from the air. As azurite is much less soluble than verdigris, it is therefore less destructive as a catalyst. Not completely stable, copper carbonates are susceptible to acid attack and decompose upon heating.

Metal Gall Inks

Iron-gall inks are made with three main ingredients (Krekel 1999). The first is a tannin source. Tannins are traditionally taken from the gall nut from an oak tree. Chemically, these tannins are a glucose structure with m-digallic acid groups rather than hydroxyl groups. In the ink formation reaction, the low pH levels result in hydrolysis of the tannins to produce the gallic acid that reacts to form the ink colorant. Modern ink recipes can simply start from purified tannic or gallic acids, rather than oak gall extracts. Next a metal source is needed. Typically iron (II) sulfate, called green vitriol, was used. This salt generally contains other metal impurities, such as copper or manganese. In addition, many recipes call for copper(I) sulfate, or blue vitriol.[1] Finally, gum arabic from the acacia tree acts as a binder for insoluble particles. The gum also modifies the viscosity of the ink to produce optimal flow for writing or drawing.

To understand what it is about iron-gall ink that is corrosive, it is necessary to understand how the ink colorant is formed. It is also important to note that not all iron-gall inks are unstable. The right ratio of iron to tannin (3.6:1) produces a balanced ink (Banik 1997).

The formation of the ink colorant is a two-step chemical process (Krekel 1999). In the first step, iron (II) sulfate reacts with gallic acid to produce a colorless, soluble iron (II) gallate complex. Secondly, oxidation (reaction with atmospheric oxygen) forms an iron (III) gallate complex. This final compound is insoluble and colored (initially a blue-black color). This chemistry is illustrated in the reaction scheme in figure 3.

Ink degradation occurs by two main mechanisms, the first of which is acid-catalyzed hydrolysis. Here, sulfuric acid (a byproduct in the first step of the ink colorant formation reaction), as well as other acids such as wine or vinegar found in ink recipes, break down not only the cellulose (resulting in a decreased degree of polymerization by breaking the glycosidic bond) but also the ink colorants themselves. As the ink breaks down, phenolic degradation products (fig. 4) result in the brown color associated with iron-gall ink (Krekel 1999). These brown-colored molecules are slightly soluble, and may cause haloing during aqueous treatment.

The second form of degradation comes from oxidation reactions. Soluble iron (II) ions catalyze two separate processes, shown in figure 5 (Neevel 1995). Focusing on the R groups in the reaction scheme, one can see the breakdown of

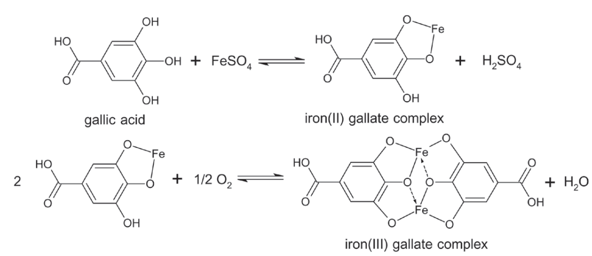


Fig. 3. Reaction scheme for the formation of the black iron (III) complex from the colorless iron (II) complex

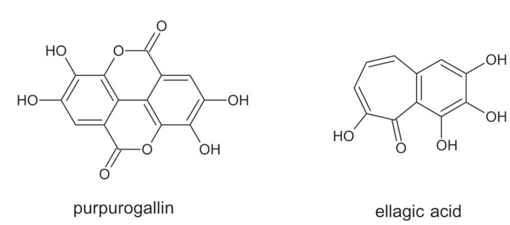


Fig. 4. Chemical structures of oxidative iron-gall ink degradation products

$$
\begin{aligned}
\text{a)}\quad & Fe^{2+} + O_2 \rightarrow Fe^{3+} + \cdot O\text{-}O^- \\
& Fe^{3+} + \cdot O\text{-}O^- + RH \rightarrow Fe^{2+} + HOO\cdot + \mathbf{R\cdot} \\
& R\cdot + O_2 \rightarrow \mathbf{ROO\cdot} \\
& ROO\cdot + R'H \rightarrow ROOH + \mathbf{R'\cdot} \\
\\
\text{b)}\quad & Fe^{2+} + HOO\cdot + H^+ \rightarrow Fe^{3+} + H_2O_2 \\
& Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^- + \mathbf{OH\cdot}
\end{aligned}
$$

Fig. 5. The oxidative degradation of cellulose catalyzed by iron (II): a) the direct production of organic radicals and their subsequent oxidation, b) the Fenton reaction, or the production of hydrogen peroxide and its decomposition to a hydroxyl radical and a hydroxide ion

cellulose to produce the bolded organic radicals and peroxides. The formation of these highly reactive species results in a decreased degree of cellulose polymerization. In the second reaction (the so-called Fenton reaction) the key molecule is the bolded hydroxyl radical. The hydroxyl radical is a highly reactive species; it has the ability to extract a hydrogen atom off of almost any molecule, while propagating the chain reaction of radical production.

Looking at figure 5 it is important to realize that any metal with multiple oxidation states can also act as a catalyst in these reactions. Copper, chromium, manganese, and other transition metals are therefore also detrimental catalysts. In particular, copper has a higher catalytic activity than iron, especially under alkaline conditions. At a pH of 8.0, copper is twenty times more effective a catalyst than iron, while at a pH of 9.0 its catalytic activity has increased to two hundred times that of iron (Kolar 2004).

Preservation Methodology

Conservation of iron-gall ink has developed over the last one hundred years into a three-pronged approach (Schäfer 2004). Antioxidant treatments are used to either deactivate transition metal catalysts or to interrupt the catalytic cycle by destroying peroxides or capturing radicals. Deacidification is employed to neutralize sulfuric acid, as well as other organic acids. Finally, physical reinforcement, generally with Japanese tissue, is often required to strengthen the now-brittle paper substrate.

Another consideration in forming a preservation methodology is the issue of aqueous versus non-aqueous treatments (Reissland 1999). Aqueous treatments offer all of the benefits found in washing paper, from removal of degradation products to reformation of cellulose hydrogen bonds. The swelling of paper fibers, however, is a double-edged sword. While swelling encourages penetration of antioxidant and deacidification agents into the paper fibers, it also introduces dimensional stress. The degraded areas of the ink line are more hydrophobic than the surrounding paper, leading to differential wetting and added stress. Aqueous treatments also pose problems for soluble media, and objects like books where aqueous treatment is all but impossible without rebinding. Non-aqueous treatments offer none of the benefits of washing, but pose little risk of dimensional stress or bleeding of media. Additionally, media color change is likely to be smaller with non-aqueous treatments.

Antioxidants

Two classes of antioxidants can combat the destructive effects of the reactions shown in figure 5 (Malšič et al. 2005). Preventive antioxidants remove or incapacitate the transition metal catalysts and tend to be metal-specific. For instance, phytate is a preventative antioxidant for iron ions. Chain-breaking antioxidants, on the other hand, are not metal specific. Rather than targeting the metal catalysts, they act as peroxide decomposers or radical scavengers, removing the propagating species from the catalysis reactions. TBAB is an example of a peroxide decomposer, while lignin can act as a radical scavenger.

Phytate (myo-inositol hexakisphosphate) treatment is perhaps the most well-researched antioxidant treatment. Salts of phytic acid coordinate with iron (II), blocking the coordination of peroxide, and preventing the propagation of the Fenton reaction (Neevel 2002). Phytate treatments have been established as an effective method for archival conservation. Studies state that, when coupled with deacidification, phytate treatment theoretically doubles the lifetime of iron-containing paper (Kolar et al. 2003). However, this treatment is only really effective aqueously, as it relies heavily on washing the iron-phytate complex out of the paper. Additionally, it only effectively coordinates iron (II) ions by blocking all coordination sites. Other transition metal catalysts are left active and are still able to cause damage.

Tetrabutyl ammonium bromide (TBAB) acts as a chain-breaking antioxidant (Kolar et al. 2005). It is the halide, experimentally shown as more effective when coupled with a large cation, that causes peroxide decomposition (Malšič et al. 2005). TBAB is advantageous in that it is not metal specific, while also being effective when applied non-aqueously (Kolar et al. 2003). However it is still a very new treatment and does not have a proven track record.

Deacidification

The deacidification treatment most commonly used with iron-gall ink is calcium bicarbonate ($Ca(HCO_3)_2$ or CaB) as it maintains a relatively neutral pH throughout treatment. Too high a pH leads to other alkaline mechanisms of ink degradation. The pH tolerance of the pigments must also be considered. Deacidification is therefore done for the benefit of the cellulose, at the possible detriment of the coloring matter.

EXPERIMENTAL: SAMPLE PREPARATION, TREATMENT AND ANALYSIS

Sample Preparation

Laboratory samples (fig. 6) were prepared of two ink formulations and two watercolor paints of different pigmentation. The inks were corrosive formulations. Ink 1 was an iron-gall ink (5.5:1 iron to tannin ratio), while ink 2 was an iron/copper gall ink (5.5:1 metal to tannin ratio, 0.7:1 copper to iron molar ratio). The watercolors were made from two unstable copper pigments, verdigris (basic copper acetate) and azurite (basic copper carbonate). Whatman filter paper no. 1 ($86.0gm^{-2}$, DP: 2630, RSD 0.74%), a pure, unsized, cellulose cotton linter formation, was chosen to ensure that interactions with fillers or additives present in other papers would not skew the results of the study.

Each sample consists of a row of five circles of 2.5 centimer diameter, spaced apart by 1 centimer. The paper of the sample is held sandwiched between a 4-ply mat board window (with 1 centimer-wide edges) on the verso and four strips of 1-centimer-wide 4-ply mat board on the recto held in place by mini binder clips. This frame served to keep the samples from cockling while they were created and aged. A Mylar template of the sample layout was prepared with voids cut out at each of the sample circle positions to ensure the spacing and size was consistent between samples. A constant volume of ink and constant mass of watercolor was transferred to each circle with an Eppendorf pipette and a synthetic paintbrush, respectively. The five circles allowed for three locations for color measurements and two locations for pH measurements.

The samples were aged prior to treatment, to simulate old artifacts, at approximately 75% RH and 55° C in a Despatch LEA 1–69 oven for four and a half days. After treatment the samples were submitted to twelve days of accelerated aging at 65% RH and 80º C to monitor the effects of the treatments over time.

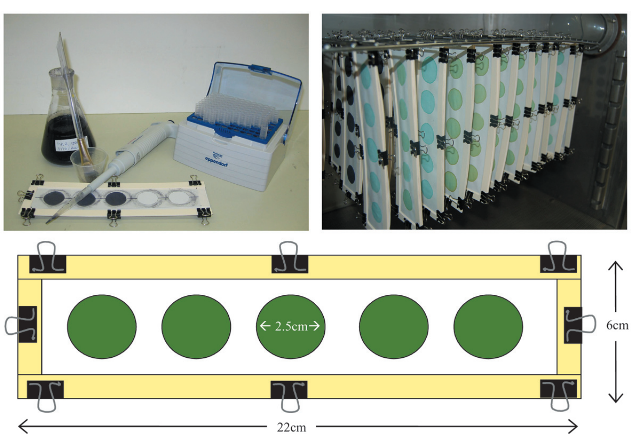


Fig. 6. Sample preparation (top left); samples arranged in an RH/temperature oven for accelerated aging (top right); sample schematic (bottom)

Antioxidant and Deacidification Treatments

Ten samples of each of the four media types were prepared to allow for several treatment groups, as well as the necessary controls. Note that, unless specified, each sample received both pre-treatment and post-treatment aging. The sample groups are as follows:

- No treatment and no aging
- Pre-treatment aging only (no treatment, no post-treatment aging)
- Pre-treatment and post-treatment aging (no treatment)
- CaP (20 minute immersion), CaB (20 minutes)
- TBAB (20 minutes), CaB (20 minutes)
- Water immersion control (40 minutes)
- TBAB in ethanol (20 minutes)
- Ethanol immersioncontrol (20 minutes)
- CaB (20 minutes)
- Water immersioncontrol (20 minutes)

CaP was prepared by the following recipe, outlined below for the preparation of 500mL of 1.75mmol/L CaP (equivalent to 0.116% phytic acid) (Neevel and Reissland n.d.):

- 1.44g 40% phytic acid solution was weighed into a beaker using a pipette.
- 0.22g $CaCO_3$(s) was slowly added, stirring with a glass rod to make a homogeneous paste. Note that CO_2(g) evolved from the paste.
- Approximately 50 mL of distilled water was used to dissolve the paste; distilled water was added to make a total volume of 450 mL.
- While stirring and monitoring the pH, approximately 4 mL of ammonia water (1.25%) was added to turn the solution slightly turbid and reach a pH between 5.5 and 6.0.
- Water was added to make the total volume 500 mL, ensuring that the pH remained between 5.5 and 6.0.

For the preparation of CaB ($Ca(HCO_3)_2$), half a liter of a saturated solution at pH 5.88 can be prepared by bubbling CO_2(g) through a mixture of 0.55 g $CaCO_3$ in 500 mL distilled water while stirring (Neevel n.d.).

TBAB was used both aqueously and non-aqueously. The aqueous treatment was a 0.03mol/L solution prepared with distilled water. The non-aqueous treatment was prepared at a concentration of 0.03 mol/L in ethanol.

Instrumentation and Analysis

Among other analyses, pH measurements and colorimetry were employed to compare the various treatments. A ROSS Ultra flat surface pH electrode was used for both

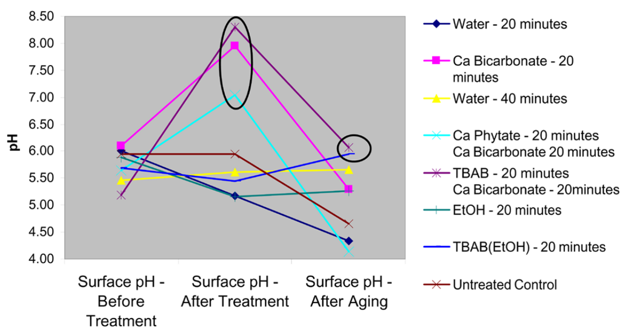


Fig. 7. Surface pH readings for azurite recorded before treatment (after pre-treatment aging), after treatment, and after twelve days of post-treatment aging

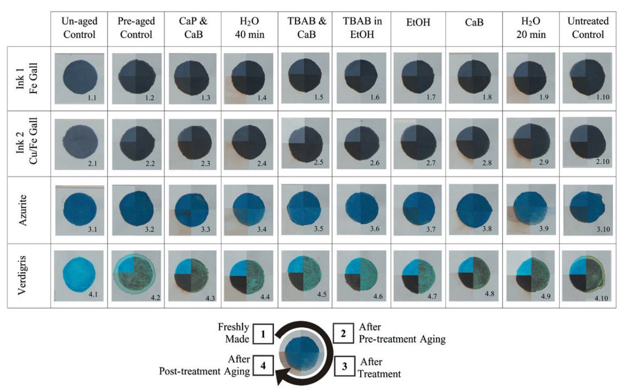


Fig. 8. Composite photographs of one spot on each sample compiled from photographs taken when the samples were: 1) freshly made, 2) after pre-treatment aging, 3) after treatment, and 4) after twelve days of post-treatment aging (see the visual key just above). These photographs qualitatively demonstrate what happens to both the media and to the paper substrate over time

surface pH and cold extraction measurements. Using the L*a*b* color space, a Minolta Chroma Meter CR-300 colorimeter was used to record and average three color readings at each of three locations per sample. These readings were recorded after pre-aging, after treatment, and after day one, day three, day six, and day twelve of post-treatment aging.

RESULTS AND DISCUSSION

Colorimetry

The colorimetry results (fig. 8) are perhaps easiest to digest and visually comprehend by looking at what happens to the paper support. Qualitatively, this is done by looking at the browning of the support around each media circle. The conclusions drawn from this qualitative analysis are as follows:

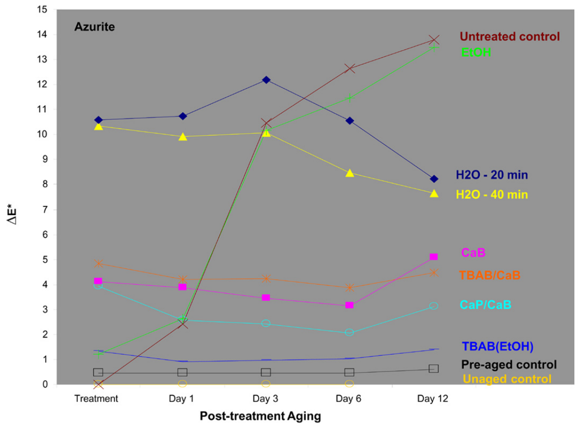
- With simple water washing, soluble transition metal ions are spread through the paper.[2]
- CaB is slightly better, as it hinders acid hydrolysis, but not oxidative degradation.[3]
- CaP with CaB treatment is effective, but only completely for the pure iron-gall ink (ink 1). For the watercolors and the mixed metal ink, it is either partially effective (ink 2) or not effective at all (both copper pigments).[4]
- Both non-aqueous and aqueous TBAB treatments are quite effective at preventing discoloration from spreading through the paper with treatment; best results are with non-aqueous treatment. This of course does not equate to stabilization, as illustrated by examining the last sample (the post-treatment aged sample). This sample underwent both pre- and post-treatment aging with little browning of the paper support. These results therefore simply indicate that TBAB treatments are safe to use. TBAB treatments seem to counter the effects of any ions that they spread throughout the paper.[5]

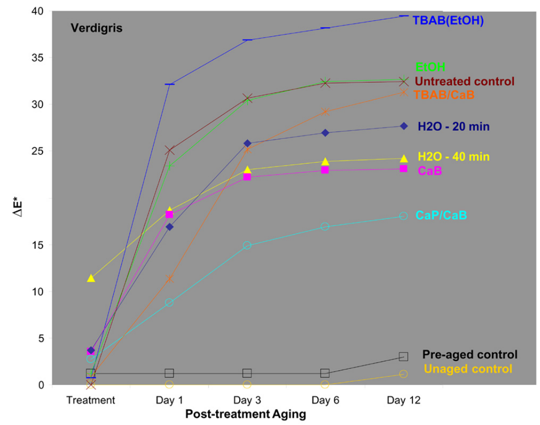
The total color change ($\Delta E^\star$) is considered perceptible to the human eye if greater than one. An optimal treatment would have an initial color change less than one, indicating that treatment does not change the appearance of the object. As the object ages, an optimal treatment would also prevent drastic color change, indicating that oxidation and hydrolytic pathways had been effectively slowed or blocked. For azurite (fig. 9) it can be seen that the best treatment, in terms of $\Delta E^\star$, is $TBAB_{(EtOH)}$. This treatment has very little initial color change with treatment, as well as an almost flatline rate of color change over time.

For verdigris (fig. 10), however, while both TBAB treatments ($TBAB_{(EtOH)}$ and TBAB/CaB) cause little initial color change in treatment, there is drastic color change on days one through three of aging. It is possible that this dramatic color change is not due to oxidation by reactions mechanisms that produce peroxides (and hence are unaffected by the presence of TBAB). The color change could instead be associated with oxidation of the pigment to various copper oxides; the reaction rates of such pathways were likely increased by the high levels of heat used in the accelerated aging regimes.

Watercolor Surface pH

While figure 7 displays the results for azurite, very similar results were also obtained for verdigris. The surface pH readings show that initially, after treatment, CaB treatments are the most effective at creating an alkaline pH. However, in time, the TBAB treatments seem to be the most effective at maintaining a near neutral pH. This may raise questions about the sufficiency of the buffer deposited by calcium bicarbonate treatments.





LEFT TO RIGHT

Fig. 9. Total color change (ΔE^*) after treatment and over the course of post-treatment aging for azurite

Fig. 10. Total color change (ΔE^*) after treatment and over the course of post-treatment aging for verdigris

CONCLUSIONS

The project began with an outline of three questions. The first of these questions was whether watercolors can be safely treated by iron-gall ink antioxidant treatments. To be "safe" a treatment should not alter the media by changing it physically or chemically. While all of the antioxidant and deacidification treatments produced an initial color change in all of the media, over the course of aging, they each showed drastic improvement over no treatment at all. In proceeding with treatment, the conservator is therefore presented with some choices. After spot testing to ensure that the media is stable, the first choice is aqueous or non-aqueous treatment. With non-aqueous treatment, there is less color change in the media; however, aqueous treatment is much more beneficial to the paper. The conservator must then decide if this initial color change is acceptable for fine art conservation. This is a question of short-term change for long-term stability, and must be judged on a case-by-case basis.

The second question posed by this research was whether corrosive copper watercolors could actually be stabilized by the same antioxidants as iron-gall inks. From the surface pH it was shown that initial stability (judged by a neutral pH) was achieved by all CaB treatments. Over time, however, it is the TBAB treatments (with or without deacidification) that show the greatest promise in maintaining neutral pH levels. Colorimetry on the recto of the samples showed that for azurite, while all antioxidant and deacidification treatments produced increased color stability, TBAB in ethanol was the best treatment. The verso tells a different story. While the color change on the verso was not quantitatively measured, qualitative analysis clearly shows that the treatment producing the most enhanced stability is TBAB/CaB (fig. 11). TBAB in ethanol produces the second most effective stabilization. For verdigris, results were not as promising. It may be that higher concentrations of antioxidants are required, or simply that thermal aging is not an accurate way to judge the long-term aging properties of verdigris samples.

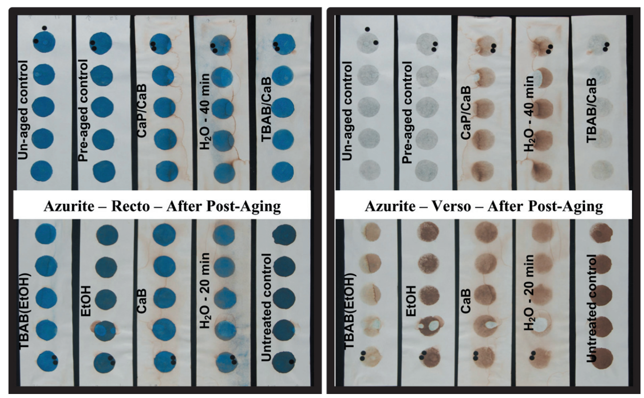


Fig. 11. Photographs of each of the azurite samples, first recto then verso, after post-treatment aging. These photographs can be used to visually compare the difference between the unaged, treated, and untreated samples

Finally, the question of the efficacy of TBAB and CaP for fine art was posed. Experimental results demonstrate that for CaP combined with deacidification the pH remains higher than untreated samples. Over time, the media color change is much less than untreated samples. From the colorimetric results, the aqueous nature of the treatment was confirmed to spread copper corrosion from the watercolor media to the surrounding paper. For TBAB, one can conclude that this treatment is effective in treating azurite. When comparing the verdigris results to the untreated control, it can at least be concluded that TBAB treatments caused no harm.

FURTHER RESEARCH

A project such as this tends to lead to many more avenues of investigation. It would be interesting to analyze the immersion baths for removed ions. Does the chelating treatment of phytate remove more ions than simply washing? Additionally, it would be very instructive to monitor the verso of the samples. As seen particularly with the azurite samples the verso gives a good indication of what is happening to the paper,

rather than simply monitoring the media itself. Analyzing the paper for color, pH, degree of polymerization, and brittleness would be informative. Additionally, varying the concentration of TBAB in the treatment of the watercolors, while also using more dilute watercolors, would help to determine optimal treatment parameters.

ACKNOWLEDGEMENTS

Many people provided invaluable input throughout the course of this research project. Thanks to Season Tse (Canadian Conservation Institute) and Maria Trojan-Bedynski (Library and Archives Canada) for aid in formulating and executing the project. Additional thanks to various key players from Queen's University for providing materials and assistance: Alison Murray, Bernard Ziomkiewicz, Barbara Klempan, John O'Neill, and Mark Reitsma. Finally, I thank my fellow MAC students for their support and solidarity during my years of study.

NOTES

1. Recipe by Ribeaucourt (1792): 2 oz. galls, 1 oz. ferrous sulphate, 0.25 oz. copper sulphate, 1 oz. gum, 1 oz. logwood, 24 oz. water. Retrieved from figure 2 in Banik, 1997, p. 21.
2. The spread of ions is seen by the brown coloration that covers the paper after aging. This is compared to the untreated control, where the paper remains white with aging.
3. In the CaB samples, the browning is less intense than in the water immersion controls, as acid hydrolysis is limited by the neutralized pH levels.
4. The efficacy of this treatment is judged on the degree of browning in the paper. For ink 1, the paper remains white, indicating that CaP/CaB works. For ink 2, the browning is less than that seen in the washing controls. For azurite and verdigris, the washing controls and the CaP/CaB brown discolorations are at the same level.
5. Both TBAB treatments prevent any brown discoloration from spreading to the paper support.

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Abrasion of Digital Reflection Prints: The Abrasiveness of Common Surfaces and the Vulnerability of Print Processes

INTRODUCTION

Very little is currently known about the care and handling of digitally printed materials. Even within the industry the manufacturers know very little about the care of the very things that they produce and sell. As a result, Image Permanence Institute (IPI) undertook its Digital Print Preservation Portal Project (DP3). The purpose of this paper is to describe two aspects of the abrasion studies done as part of the DP3 project and their impact on storage recommendations for digital print collections.

THE DP3 PROJECT

The DP3 project started in late 2007 with the understanding that the digital printing field was still evolving, although by 2007 the industry seemed to be relatively mature in its development. The project is intended to result in a web site dealing with all aspects of stability and care of digitally printed materials. However, rather than addressing the particular stability issues of printing products by brand, this research project looked at stability differences and similarities by generic product type. If necessary, product specification may go as far as an ink type and paper type. Therefore, the web page might be more specific than referencing generic product type alone. If greater specification becomes necessary, the user will have to be able to identify digital print processes.

DIGITAL PRINTS

Within the confines of the DP3 project it was necessary to define what IPI meant by the term "digital print." We have found that many people have different views on what materials this term includes (Burge et al., *Archival Outlook,* 2009), so, for this project, "digital print" refers to both text and images on digital press, inkjet, dye sublimation, electrophotographic, and digitally printed chromogenic prints.

Presented at the Book and Paper Group session, AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

ABRASION

Initially, the interest in abrasion testing was undertaken because it was one of the most common handling problems found in traditional photographic collections. Remarkably, a survey of the field undertaken as part of the DP3 project in the summer of 2008 showed that abrasion was the number one problem observed in collections of digitally printed materials. Forty-two percent of respondents from libraries, museums, and archives said that they had observed abrasion in their collections of digitally printed materials. Assuming that the survey respondents represented a good cross section of institutions in the field, this result had a maximum error of 7.3% to a 95% confidence. Given that digitally printed materials have been kept in collections for only the past fifteen years or so, 42% is a very serious result (Burge et al., *Archiving*, 2009).

Abrasion differs from scratching in both form and cause. Scratches tend to appear as discrete furrows in the surface of the print from which material has been removed. They are caused by relatively large, sharp objects (large asperities) being pushed across the surface of the print (or *vice versa*, the print may be pushed across the sharp objects). Large, relatively dull objects may also produce a furrow, but in this case the print material will be pressed in and not scraped out. Scratch damage may be reproduced in the lab by scraping a needle or stylus across the surface of the print. Abrasion, on the other hand, results from a material surface with small asperities being pushed across the surface of the print (or *vice versa*).

PRELIMINARY STUDIES

Currently, the International Organization for Standardization (ISO) has no standard abrasion test method for digitally printed images, although there are a variety of scratch tests for traditional photographic materials. In fact, work has

just barely started on an abrasion test method standard for images. A test method was determined at IPI that will be presented to ISO for consideration. It is important to note, however, that it is very difficult to characterize any individual storage or transportation situation and, therefore, nearly impossible to relate the absolute results of the lab tests to any real-life situation.

In previous work a variety of abrasion testing devices were considered, and the Sutherland rub tester, a motorized rub tester, was settled on as the best abrading device. This device consists of a base to support the test sample and an arm on which an abrading surface can be mounted. The arm sweeps the abrading material back and forth across the test sample at a programmable speed and for a programmable number of cycles. A number of weights are available so that the abrasion can be varied across a broad range of pressures. A similar manual device was also considered, but it was decided that the electric motor made the Sutherland easier to use and largely eliminated operator differences as a source of noise in the test.

Preliminary tests showed that abrasion manifested itself in several ways: density loss in dark patches as colorant was scraped off, changes in surface gloss, and smearing of colorant from dark patches to adjacent white patches. The objective monitoring of samples for damage would have to track these manifestations of abrasion damage. For example, in samples with smudging it was found that visual ranking of samples correlated well with percent density change and average gray value change in the adjacent white patch with Spearman's rank correlation coefficient values of 0.90 and 0.89 respectively (Salesin et al. 2008). It was interesting to see that other parameters—such as density change in the black patch and gloss change with Spearman's rank correlation values of 0.62 and 0.27 respectively—did not correlate as well to visual evaluation. By far, smudging of colorant into the adjacent white area was the most objectionable effect of abrasion. Even when gloss change values or density change values were quite large, the visual change in the sample was usually not considered to be very significant.

METHOD

The remainder of the paper deals with two aspects of the project: the relative effects of common abrasive surfaces and the relative vulnerability of a range of print types and papers to abrasion. This method section as well as the analysis section will therefore be divided into two parts.

Abrasive Surfaces

Sample materials included the following:

- Digital press on glossy paper (Samples L, M, N)
- Offset printing on glossy paper (Sample O)
- Black-and-white electrophotography (laser prints) on office copy paper (Sample G)
- Color electrophotography on office copy paper (Sample H)
- Solid inkjet on office copy paper (Sample F)
- Dye inkjet on porous-coated and polymer-coated photo papers (Samples A and B)
- Dye inkjet on office copy paper (Sample D)
- Pigment inkjet on photo paper (porous-coated) (Sample C)
- Pigment inkjet on office copy paper (Sample E)
- Dye sublimation, also called dye sub, dye diffusion thermal transfer, or D2T2 (Samples I and J)
- Digitally printed chromogenic photographic paper (Sample K)

Dye sublimation printing requires a mated paper for the printer and therefore has no paper type specified.

Polymer-coated paper, also called swellable paper, has a swellable polymer coating, usually gelatin or a combination of gelatin and polyvinyl alcohol. This type of paper is a direct descendent of traditional photographic paper. It is currently used only for dye-based ink. The ink is absorbed into the coating, causing the coating to swell. This paper has the disadvantage of being slower to reach touch-dryness, as the coating remains sticky until the water and additives in the ink have dried. As a result, printer through-put is necessarily slower.

Porous-coated paper, also called instant-drying paper, is produced by mixing an inorganic material, typically silica or alumina, with a polymer to make a paper coating that contains pores. Macro-porous paper has the largest pores and is only available in a matte surface. Micro- and nano-porous papers can be quite glossy. This paper acts by absorbing the water from ink into the pores and holding the water in a lower layer of the paper. The surface becomes dry to the touch almost instantly, and therefore printer through-put can be quite fast without the risk of stacked prints sticking together (IPI 2008).

In all cases, three replicate specimens were run.

Four common abrading surfaces were used for comparison: the back side of an identical print paper, a typical envelope paper, a typical interleaving paper, and polyester film, such as is used to make sleeves. A very small amount of very fine silica is added to one or both sides of the polyester film as an anti-blocking agent to prevent sleeves from sticking together when they are stacked. This silica has not been found to be a significant cause of abrasion with conventional photographic materials.

The abrading surface was mounted to the moving arm of the Sutherland rub tester, and the test prints were abraded with two pounds of weight on the arm, producing a pressure of 1.7 kPa or 0.25 psi for 100 cycles.

Print Vulnerability

In this part of the experiment a wider range of print types and papers was used. A total of 57 printer/paper combinations were tested including multiple brands representing the following generic combinations:

- Liquid toner digital press on glossy paper
- Solid toner digital press on glossy paper
- Offset lithography on glossy paper
- Black-and-white electrophotography on laser-print-specific office paper
- Black-and-white electrophotography on non-recycled office copy paper
- Black-and-white electrophotograph on 100% recycled copy paper
- Color electrophotography on color laser-print-specific office paper
- Color electrophotography on non-recycled office copy paper
- Color electrophotography on 100% recycled copy paper
- Solid inkjet on color laser-print-specific office paper
- Solid Inkjet on non-recycled office copy paper
- Solid inkjet on 100% recycled copy paper
- Dye inkjet on photo-coated paper
- Pigment inkjet on fine art paper
- Dye inkjet on inkjet-specific office paper
- Dye inkjet on non-recycled office copy paper
- Dye inkjet on 100% recycled copy paper
- Pigment inkjet on inkjet-specific office paper
- Pigment inkjet on non-recycled office copy paper
- Pigment inkjet on 100% recycled copy paper
- Dye sublimation prints
- Digitally printed chromogenic prints

These samples were abraded with envelope paper only using the same pressure and number of cycles as for the abrasive surfaces study. Again, three replicate specimens were run for all printer/paper combinations.

MEASUREMENTS

Objective measurements were made on all specimens using the following three devices:

The BYK Gardener Micro-TRI-Gloss meter measures gloss at three angles to the surface normal. Highly glossy materials are measured with the incident light and detector at 20° to the surface normal. Moderately glossy materials are measured at 60°, and matte surfaces are measured at 85°. The rule of thumb for measurement is that one starts with the 60° angle and if the reading is less than 10, then the surface is matte and 85° should be used. If the 60° reading is greater than 70, then the surface is glossy and 20° should be used.

The GretagMacbeth Spectrolino spectrophotometer was used to make optical density and colorimetric readings. Density measurements conformed to ANSI/ISO status A (ISO 1995a, 1995b).

ImageXpert image analysis software takes a specified area of a scanned image and assigns an eight-bit brightness level to each pixel in the area of interest, ranging from 0 (black) to 255 (white). The average brightness level for all of the pixels in the area of interest is called the *average gray value*. For the one-half inch by one-half inch square used in this project, there were 4,481,689 non-random measurements averaged.

With both the gloss meter and spectrophotometer, three random readings were taken on each of the three replicate specimens before and after abrasion.

ANALYSIS

In all cases, it was assumed that the direction of change in gloss, density, or average gray was not important, but the degree of change was. Therefore, absolute change values for these three measurements were used in the analysis.

Abrasive Surfaces

Analysis was performed in three ways: visual examination, comparison of average change by printer/paper combination per abrader per measurement parameter, statistical calculation using the Whitney-Mann U test or equivalent rank sum test to compare pools of data containing all absolute change values for all printer/paper combinations for each test parameter and abrading surface. One pool might contain absolute change values for gloss measurements with polyester film as the abrading surface. Another might be average gray value change in the black patch with interleaving paper as the abrading surface. This kind of general analysis was potentially problematic for the print verso abrading surface, since the abrader changed with the paper used. Other abrading surfaces remained constant over all of the paper/printer combinations. Comparisons between data pools produced one of three outcomes: the two were equivalent to a 95% confidence, A was worse than B to a 95% confidence, or B was worse than A to a 95% confidence. These paired results were then combined to produce an overall rank order for the abrading surfaces by test parameter.

The general result was that polyester film was less abrasive than envelope paper, interleaving paper, or print verso, and that print verso was apparently the most abrasive surface (table 1). However, the values that went into this result were highly variable by printer/paper combination so the result has to be considered in conjunction with the results from the print vulnerability study (tables 2–4).

Abrading surface	Average gray in black patch	Average gray in white patch (smear)	Gloss
Verso	1	7	3
Polyester	0	0	1
Envelope Paper	1	6	2
Interleaving Paper	0	6	3

Table 1. Average changes in three parameters across all samples for abrasive surfaces study

	Sample														
Abrading surface	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
Verso	0	0	3	1	1	1	2	0	0	1	1	0	2	1	0
Polyester	0	0	1	0	0	0	0	0	0	0		0	0	0	0
Envelope paper	0	0	2	1	2	1	1	0	1	0	0	0	3	6	0
Interleaving paper	0	1	1	1	1	1	0	0	1	0	0	0	0	0	0

Table 2. Changes in average gray in the black patch by abrading surface and sample for abrasive surfaces study

	Sample														
Abrading surface	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
Verso	0	2	13	40	4	1	33	2	0	1	0	0	1	1	1
Polyester	0	0	0	0	1	1	2	1	0	1	0	0	0	0	0
Envelope paper	3	0	16	33	13	2	17	1	0	0	0	1	2	2	1
Interleaving paper	2	0	5	59	14	1	10	0	0	0	0	1	0	1	1

Table 3. Changes in average gray in the white patch (smear) by abrading surface and sample for abrasive surfaces study

	Sample														
Abrading surface	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
Verso	2	0	2	2	1	3	9	1	4	7	4	0	5	2	0
Polyester	1	2	0	0	0	1	0	2	2	3	4	0	0	0	0
Envelope paper	0	7	1	1	1	4	4	0	1	0	2	1	1	5	1
Interleaving paper	11	8	1	2	1	5	4	1	6	0	2	1	0	0	0

Table 4. Changes in gloss by abrading surface and sample for abrasive surfaces study

Print Vulnerability

As with the abrasive surface study, analysis of this part was performed in three ways: visual examination, comparison of average change values by parameter and printer/paper combination, and rank sum test.

Some general conclusions could be drawn from this analysis, although results could vary considerably from paper to paper for the same printer. Pigment inkjet generally performed quite poorly with regard to abrasion, although it was no surprise, since the colorant sits on top of the paper surface. Color electrophotography was one of the best products. On all three papers, it was in the top eight printer/paper combinations. This is also not necessarily a surprising result, even though the image sits on the top surface as the pigment inkjet does. The difference is that, in laser prints, the colorant is bound in a polymer that is melt-extruded into the paper. What was surprising was that the best black-and-white electrophotography/paper combination was worse than the worst color electrophotography/paper combination. Chromogenic, offset lithography, dye sublimation, and liquid digital press rounded out the top-performing processes. Printer/paper combinations that were in the middle were either mediocre

	Average Gray Value Change in Black Patch		Average Gray Value Change in White Patch (Smear)		Gloss Change	
Print Type	Average	Standard Deviation	Average	Standard Deviation	Average	Standard Deviation
Inkjet fine art 1	2	0.4	36	11.8	2	0.4
Inkjet fine art 2	2	1.0	5	7.1	4	1.7
Inkjet dye office	1	0.5	13	15.9	3	2.9
Inkjet pigment office	2	0.4	21	12.5	3	2.4
Inkjet photo	0	0.2	1	1.3	4	2.8
Inkjet solid	1	0.2	1	0.2	5	1.3
Black-and-white electrophotographic (EP)	1	0.6	9	8.6	4	2.9
Color EP	1	0.9	1	1.3	1	1.0
Dye sublimation	0	0.1	0	0.4	3	2.5
Chromogenic	0	0.0	1	0.5	2	2.4
Digital press liquid	0	0.1	1	0.2	0	0.2
Digital press solid	4	2.3	2	0.9	3	2.2
Offset lithography	0	0.0	1	0.4	1	0.8

Table 5. Average and standard deviation of three parameter changes by generic print type for print vulnerability study

performers in all test parameters or they were excellent in some parameters and poor in other parameters.

However, as with abrasive surface study, results were quite variable and care must be taken before jumping to conclusions based on generalized results (table 5). This table is mostly divided by generic process with measurements averaged over all of the test papers (where used). However, in a couple of cases, specific products were broken out to show differences that were considered to be important. One difference was that digital press with liquid toner was the second-best performer while digital press with solid toner came sixth from the bottom (out of 23). A second difference was that the two fine art papers performed quite differently, although neither was especially resistant to abrasion. All three fine art printers used pigmented inks, but apparently the papers had an impact on how well the pigment adhered to the paper.

RESULTS

From the abrasive surface study, the general conclusion is that polyester film makes the least abrasive surface and the print verso was, relatively speaking, the most abrasive surface overall. As expected, the abrasiveness of the print verso varied quite a bit from printer/paper combination to printer/paper combination, but overall, it was the worst surface for abrasion. However, results were quite variable, so one must consider the results of the print vulnerability study as well.

Results from the abrasive surfaces and print vulnerability studies together led to the following recommendations:

- Archival materials that are quite vulnerable to abrasion damage, such as pigment inkjet prints, should be stored in a polyester enclosure (one print per enclosure) or protected with a polyester cover sheet or interleaving.
- All fine art prints, prints for which even small changes in gloss might be intolerable, should also be stored in polyester enclosures or protected with a polyester cover or interleaving. Care must be taken that no pressure is applied to the polyester.
- Archival materials that are resistant to abrasion damage should be fine if handled and stored with reasonable care. This means that these materials would benefit from a protective enclosure, but a conservation treatment report with images, for example, printed on a color laser printer doesn't need to be rebound with each page in a sheet protector. Similarly, photo books printed on a liquid digital press or chromogenic system probably don't need to be rebound to fit interleaving sheets.

These recommendations are made based on relative rankings of abrasive surfaces and print processes. At this time it is not possible to evaluate the absolute abrasiveness of a print surface or the absolute sensitivity of any digital print process with respect to real life.

The outcome of this research will require that the people charged with the care of these objects be able to identify print processes.

ACKNOWLEDGEMENTS

The authors would like to thank Jessica Scott and Nino Gordeladze for their contributions to this project.

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A Technical Study of a Relatively Unknown Printing Process: Mixografia

ABSTRACT

This paper is a summary of a project undertaken to investigate the materials, manufacture, and subsequent treatment options for a collection of prints produced using the Mixografia process. The Mixografia printing method when compared to traditional techniques such as etchings, woodcuts, or lithographs is very unusual. The originators of this process still hold the patent; little is known about the materials and mechanism used to create these works.

To understand the development of this printing method, an art historical survey was conducted on the originating artist, Rufino Tamayo, and the printing studio, Mixografia Workshop, as well as the owner Luis Remba. Information on the events leading up to the collaboration and conception of the process has been well documented, but specific information on the actual printing process and materials used were limited. The descriptions provided by the Mixografia Workshop raised more questions than were answered.

A technical investigation was conducted to determine details regarding the manufacture of these prints. During this study some of the materials associated in making these works of art were identified, and a reconstruction of the printing process was devised.

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Digital Surrogates: A New Technique for Loss Compensation of Graphic Works on Paper

ABSTRACT

Historically and presently, restorers and conservators engaging in loss compensation have used fine hand skills to recreate convincing design elements in addition to paper color and texture. These techniques have been documented and taught. In contrast, the focus of this paper is the use of digital surrogates as an alternative for loss compensation of graphic works using digital images and high quality printers. By employing this method, the lost design is reconstituted without compromising the artist's original intent.

The process of producing a digital surrogate will be explained using a case study of a recent treatment of Jacques Callot's *The Siege of La Rochelle*, a monumental print from sixteen plates. While producing a facsimile image might seem relatively simple, there are several details in production that can be adjusted to produce a high quality surrogate. Using digital images from a complete print for the reproduction, two methods of production were identified: printing the appropriate design on a paper similar in quality to the original and printing the illusion of paper texture, color and design on a high quality inkjet optimized paper. This paper will focus the techniques used for the final digital surrogate with a discussion on image capture, digital manipulation, paper selection, and surface coatings.

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Presented at the Book and Paper Group session, AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

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Place in Wales: Reconstructing Drawings from a Sketchbook by Francis Place

ABSTRACT

Amgueddfa Cymru—National Museum Wales holds eighteen works on paper by Francis Place (1647–1728). During routine conservation it became apparent that ten of the drawings were from the same sketchbook. These ten drawings form an important part of the collection as they are the earliest images of Wales carried out on the spot (dated 1678). Place was a gentleman amateur artist who was one of the earliest English artists to specialise in landscape.

On the removal of secondary supports from two works, drawings were discovered on the verso, enabling some order of the drawings in the sketchbook to be established. Research revealed, among other facts, that a second sketchbook from his tour of Wales also survives.

Perhaps the most fascinating information to be revealed during the project were the annotations and marks that Place used. Pages fold over to join up with the landscape on the verso; ink marks in the form of crosses found on the edge of a double page spread join up with sketches on the verso to extend the panorama further. These intriguing, subtle marks are an insight into Place's working techniques and his use of a sketchbook, transforming it from a simple drawing surface into an instrument that conveys space.

From this new research it will now be possible, using the manipulation of digital images, to "recreate" the sketchbook and view the panoramas as Place saw them. Through the museum's website and gallery interactive, access to the drawings will now be possible in a way that could never be achieved on a gallery wall or in a study room.

INTRODUCTION

Amgueddfa Cymru—National Museum Wales has eighteen drawings in total by Francis Place (1647–1728) (although there were nineteen until one was recently re-attributed to William Lodge). Of these, fifteen are views in Wales, ten of which are from the same sketchbook (the size of the paper and the watermarks all being the same), and it is these ten that this article centers on. The ten sketches (dated 1678) are important as they are the earliest images of Wales carried out on the spot.

The group came to the Museum in 1931, purchased from a dealer who had bought them a few months earlier from a sale at Sotheby's. The sale at Sotheby's was of the collection of Patrick Allan-Fraser Art College in Arbroath, Scotland, including drawings, prints, pottery and the only known oil (which is a self portrait) descending directly through the family from Place.

Works by Francis Place came up in my work schedule as they were going on display in the new Welsh Landscape Gallery: The Power of the Land. This gallery is a project that is part of a gallery refurbishment program that followed extensive roof repairs. This gallery marks a new approach to display by the Museum in that it is not arranged chronologically but by the regions of Wales, and works on paper hang alongside oils.

THE DRAWINGS

The drawings are all made from at least two separate sheets either joined by overlap or butted together and then strengthened on the verso by a strip of paper. Each was adhered to a secondary support that appears to be a cut-down album page of early wove paper, suggesting late eighteenth- or early nineteenth-century. Some drawings that obviously extended over the page had been left 'loose' on the album page to allow the viewer to lift the page and view the verso, but others had been adhered down around all four edges to the secondary support—the sketch on the verso seen as inconsequential. Figures 1 and 2 were taken before conservation and are the same work. The secondary supports are still attached and visible. In these two images one can see how the joined sheets were attached to the album page along the centre, and

Presented at the Book and Paper Group session, AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

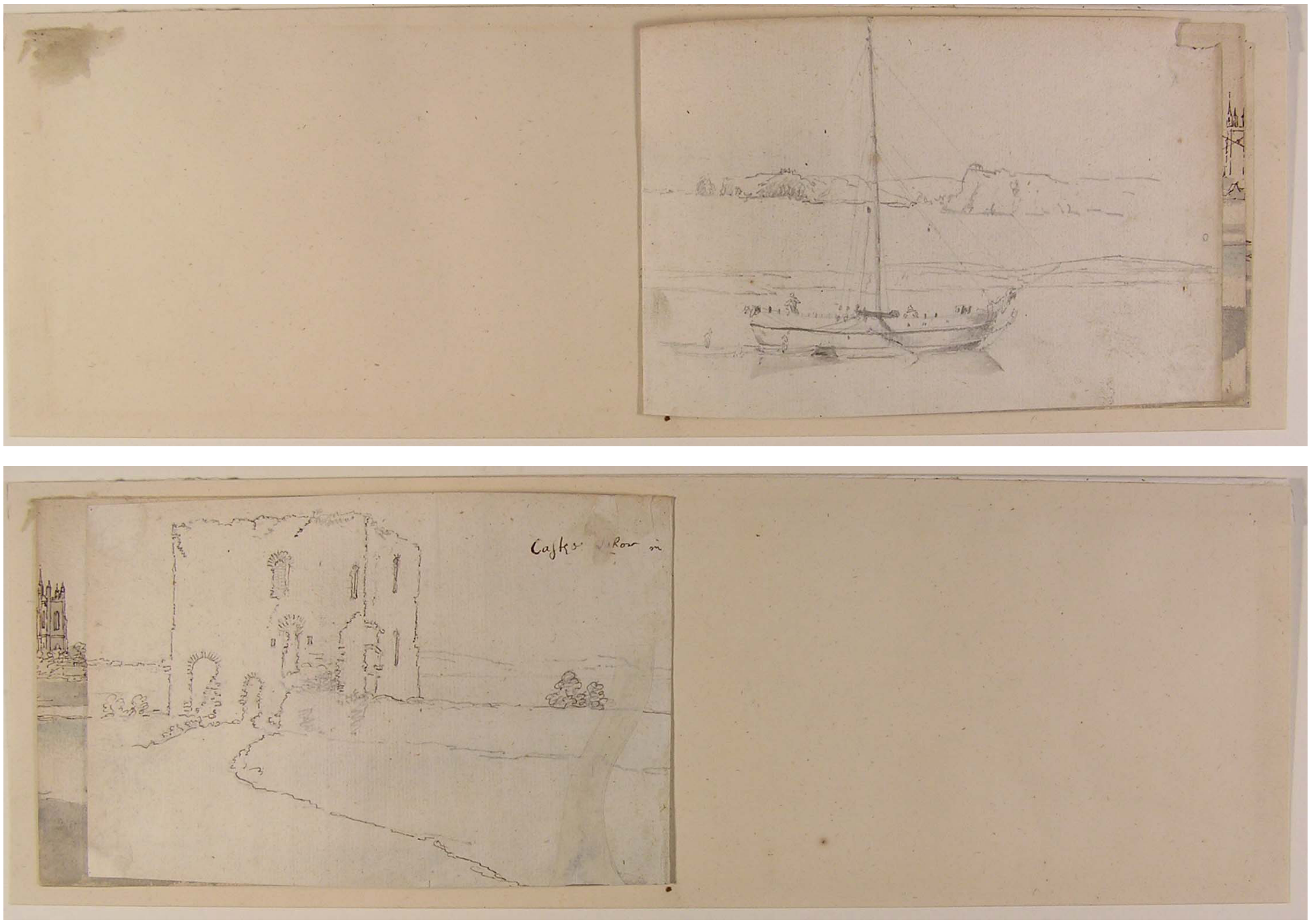

Fig. 1–2. Francis Place, *Cardiff 1678*, NMWA 16367 (verso)

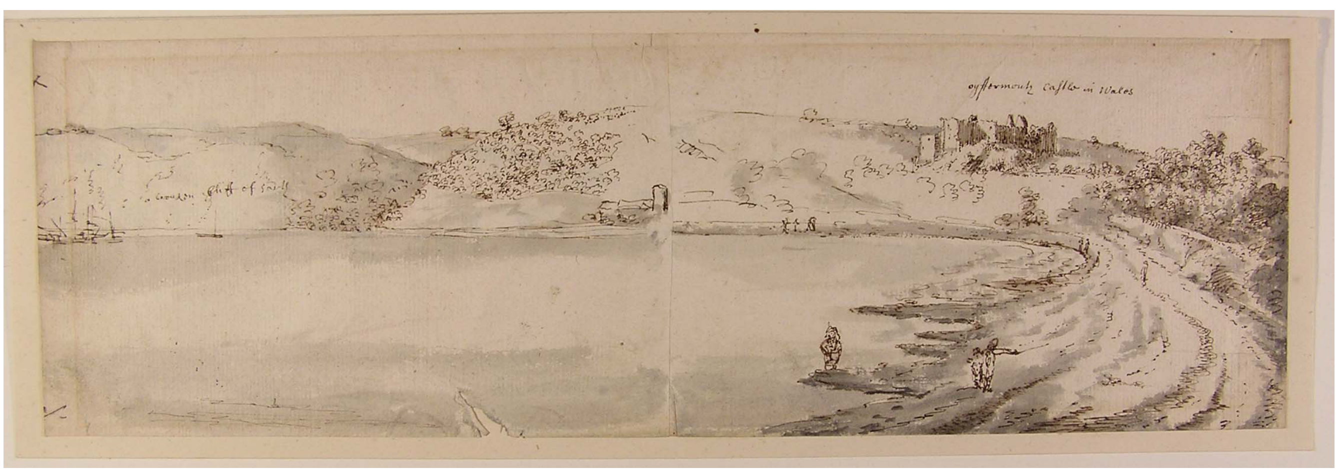

Fig. 3. Francis Place, *Oystermouth Castle*, NMWA 16368

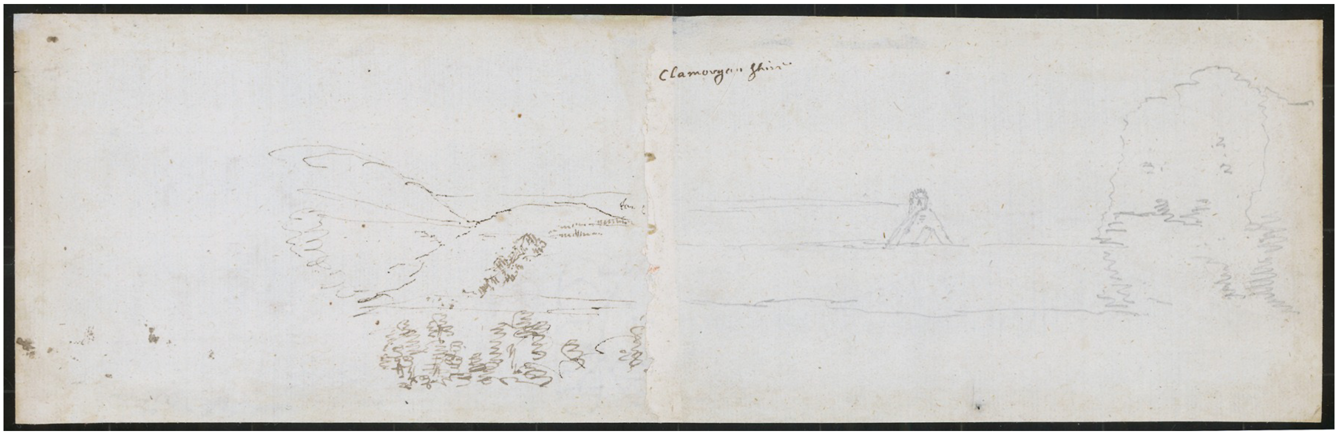

Fig. 4. Francis Place, *Tenby*, NMWA 3355 (verso)

Fig. 5. Digitally stitched image created from the versos of NMWA 16367 and 3355

by turning over the sheet the sketches on the verso can be revealed on the left (fig. 1) and right (fig. 2).

I discovered that in the last twenty years four sketches had been removed from their secondary supports to facilitate mounting. In consultation with curatorial staff I made the decision to remove the remaining sketches from their secondary supports. This then revealed further sketches on the versos.

I removed the secondary-supports/album-page remnants and adhesive residue and also decided to remove some of the repair paper only when it obscured part of the image below. On some I made no attempt to remove the repair paper as it was causing no damage or discoloration, so I saw it as important to retain as evidence of the object's history.

On removing the album page debris, some drawings were also found to have blue paper remnants beneath at the corners—evidence that they were on a previous secondary support. Some drawings had suffered from light damage and from being displayed (fig. 3). I carried out a little judicious surface cleaning but no aqueous treatments were considered, mainly because of the lightness, subtleness and wear of the shading and because the sketches are double sided and the ink and pigments were sensitive to water. Cleaned and pressed, the sheets were scanned.

WHO WAS FRANCIS PLACE?

Francis Place was a Yorkshire man, born into a wealthy family in 1647 the last of ten children. It was his father who decided that he should follow in his footsteps and at the age of seventeen or eighteen he entered Gray's Inn in London to study Law. Francis made his dislike for law known and the Great Plague in London (1665) gave him his excuse to finish with his studies and return home. A short time later he returned to London and worked with Wenceslaus Hollar who introduced him to printmaking and print selling in London.

American colleagues may be thinking that his name sounds familiar and there *is* an American connection: The Historical Society of Pennsylvania has two small chalk drawings by Place, which they claim are the only surviving images made from life of Pennsylvania's founder William Penn and his second wife Hannah Collowhill Penn. They are signed and were bought in 1957 from the descendents of Place.

There is some suggestion that Place received some or all of his inheritance before his father died in 1681, and it would have been this that enabled him to follow his passion for art and angling. The seventeenth century was the age of the *Virtuosi*—likeminded men with money and leisure who were interested in art, science, and philosophy; many of whom went on to form the Royal Society in 1660. In the first half of the century only royalty and those closely associated learned and practised the art of drawing and painting, the main reason being that to study perspective gave them insights into fortification and painting so that they could collect.

Towards the end of the century it was then taken up by the landed gentry and their sons and daughters. There were many reasons for this including filling their time so they didn't go off the rails or slip into the abyss that was "melancholy." They always studied to acquire knowledge which was very different from men who had to make a living from it. There is evidence that, although ostensibly an amateur, Place was paid for work, particularly early on.

So Place fits in very well with the gentlemen of the time; he had the time and the money and, from what survives of his work, he dabbled in many media including early experiments in porcelain. He was a member of the York Virtuosi who included, among others, Martin Lister, Henry Gyles, Thomas Kirke FRS and William Lodge. It may have been through this group that he earned commissions and also learned the "secrets" of the day. A good example of one of these secrets is the art of mezzotint, which was developed by these amateurs to its full potential before it got out into the real world where it was used for more commercial purposes. It is also unsurprising to note that the earliest examples of mezzotint can be found in the collections of these virtuosi.

With his fathers money, Place travelled far and wide in the UK to sketch and practise the art of angling. We know from correspondence of the period that our sketches were carried out on a tour of Wales and the West Country in 1678. He was travelling with his great friend and fellow York Virtuosi William Lodge. Travelling at this time was not without its dangers as this was the time of the Popish plots, and it is known that while in Wales they both spent one night in jail under suspicion of being Jesuit spies.

PUTTING THE DRAWINGS IN ORDER

With all the sketches detached from the album pages it also became apparent that a drawing on the back of one joined up with the drawing on the back of another to create a new double-page spread. With this revelation I began matching up and seeing a sketch unseen since the original sketchbook had been taken apart at least 200 years ago. It was in this way that I also managed to work out the order of the sketches in the original sketchbook. Figure 2 joins up with figure 4, and figure 5 shows the two digitally stitched together.

I quickly realized that there was limited potential in showing these historical views of Wales in a traditional gallery setting, so I worked with our photography department to scan and digitally stitch the images back together again. Photography staff also gave me some insight into Place's working methods, remarking that in putting together the separate pages no manipulation was required to allow horizons to match up, as they all did perfectly. Testimony indeed to Place's ability as a draughtsman. Although as to whether he used a camera obscura—at this time being developed and used by the virtuosi—remains unsubstantiated.

Another interesting observation, which made it much easier to see where drawings continued over the page, was Place's use of marks and devices to extend a panorama and thus his use of the sketchbook to create wide open space. On many of our works there are crosses or arrows which show where the panorama is extended over the page on the verso. Note on figure 3 the crosses on the very left edge which show that the coastline is extended on the verso of the sheet.

On a view of Pembroke Castle (fig. 6) a section of the sheet on the very right folds over. Notice the crease where the panorama continues on the verso (fig. 7). Figure 8 shows it digitally stitched together. This image is very puzzling as the extra section on the verso is noted as St David's, which, geographically, would be technically impossible to see from this view. As a point of interest, National Museum Wales also has another version of the whole panorama—not what you would call finished, but slightly larger than the sketchbook sketches and executed on a single sheet.

RESEARCH

The curator of prints and drawings, Beth McIntyre, and I embarked on research into Place and the history of these drawings. The original Sotheby's sale in 1931 is where the bulk of his original work, now in institutions, comes from. Various lots from this sale found their way to the collection of the Victoria & Albert Museum in London. We were particularly interested in a number of mounted drawings and two sketchbooks from their collection.

The first sketchbook consists of eighteen sheets that are exactly the same size as ours and bearing exactly the same watermarks. Were our drawings from this sketchbook? We think so. Tantalisingly, there is a list at the front of the sketchbook that corresponds to the views in the sketchbook (missing a few irrelevant or unidentifiable places), and the list continues with places in Wales that correspond with the order I have established from our sketches. Unfortunately, the list is in someone else's handwriting but is still of some age. Maybe it was a descendant of Place who made the list before they extracted the best sketches to mount in their prized album?

More evidence to back up our claim is found in the second sketchbook where there is a sheet pasted onto the back

Fig. 6–7. Francis Place, *Pembroke*, NMWA 16371, recto (top) and verso (bottom)

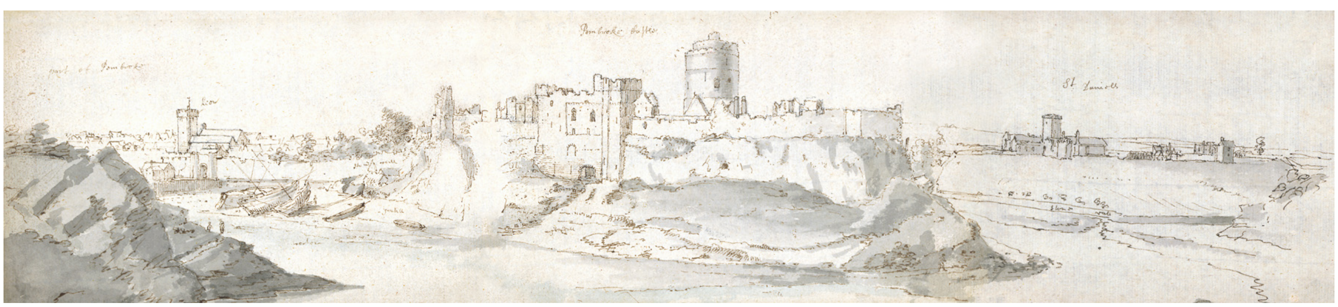

Fig. 8. Digitally stitched image created from recto and verso of NMWA 16371

inside cover. This sheet joins up with the last sketch in the first sketchbook. Unfortunately, one third of this last page is pasted down but on the small part that can be lifted a definite pencil line can be seen—potentially this pencil line, or horizon, matches up with that on one of our sketches.

In the larger, second sketchbook there is evidence of how Place used a double page to create his panorama. The horizon is drawn across from one page to the next, and presumably the potential is to then fill the drawing in, but this piece is unfinished or abandoned.

CONCLUSION

It has been a very pleasing project to work on and immensely satisfying. I have a few blank pages, but I have been able to establish the order of our sketches in a sketchbook and even fit them into an existing sketchbook.

Digital technology has been imperative to this ongoing project. Only a few years ago we would have been physically cutting out photographs to put these sketches back together. Technology, though, has not changed the way in which I

treated these objects but it has greatly enhanced our knowledge of the works by giving deeper insight into the working methods of the artist.

Technology has also allowed us to make this knowledge available and accessible to our visitors. We are working on a computer interactive available in the gallery and also an article for the "virtual museum" on our website.

ACKNOWLEDGEMENTS

Thanks to the Anna Plowden Trust for a mid-career training grant to assist me in attending and presenting this paper at the AIC annual conference.

Thanks also to staff at National Museum Wales, particularly Beth McIntyre and Becky Brumbill.

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Monitoring Aging Processes of Archival Documents by Means of Quantitative Hyperspectral Imaging: A Part of the Hyperspectral Project at the Nationaal Archief (National Archives of the Netherlands)

ABSTRACT

Archival and library institutions are constantly faced with the great challenge of finding and maintaining a good balance between the public access of historical documents and their conservation. Many tools are used in the conservation field to assess and monitor the suitability of storage and exhibition environments, but a direct verification of the effects of these environments on original documents is still difficult. The Bihanne Project, started in 2006 by the Nationaal Archief (National Archives of the Netherlands), as a branch of the Hyperspectral Project started in 2004, aims at exploring the applications of the hyperspectral imaging technique for monitoring the aging process in documents. Within the Bihanne Project data are taken from both original documents (naturally aged) and specifically prepared samples (artificially aged) to compare aging behaviors of a large number of materials exposed to different conservation treatments and aging conditions. This paper will provide a general introduction to the project and the working principle of quantitative hyperspectral imaging. The procedures followed for the measurement and the analysis are described in detail, and initial results are presented. The high sensitivity of the technique in detecting spectral changes is demonstrated using sample documents artificially aged through light exposure. The results have provided important information for the definition of the application ranges of quantitative hyperspectral imaging not just as an analysis method but as a monitoring tool.

INTRODUCTION

The use of hyperspectral imaging in the archive field is a relatively new subject, although many of its potential applications have already been experimentally addressed using multispectral imaging (Delaney et al. 2005; Kubik 2007, Fischer and Kakoulli 2006). The two techniques are in fact based on the same working principle, however, hyperspectral imaging provides more detailed spectral information and a better reproducibility of quantitative data if a correct calibration procedure is applied. As a consequence, results extracted with a multispectral imaging system can generally be obtained also with a hyperspectral system covering the same spectral range, but not vice versa. The application of this technique on documents has shown to improve the quality and quantity of information obtained by the analysis of the data, opening new perspectives to the diagnosis and study of archival items. This paper concentrates on one particular application, namely the monitoring of aging processes, which represents at the moment an almost unexplored subject of using hyperspectral imaging in the field of cultural heritage.

The research project is dedicated to the memory of our colleague Bihanne Wassink, who prematurely passed away on the 21st of April 2008.

The majority of this contribution focuses on the challenges faced during the practical implementation of the technique as a monitoring tool and on the initial results of the experiments currently carried out. Although a brief introduction of the hyperspectral imaging technique is given here, the reading of introductory texts such as Campbell's *Introduction to Remote Sensing* (2002) and Chang's *Hyperspectral Data Exploitation* (2007) is suggested for a more detailed and technical description of the technique itself.

THE BRANCHES OF THE HYPERSPECTRAL PROJECT

The start of the Bihanne Project can be traced to three particular challenges faced during other research conducted at the Nationaal Archief: 1) The direct verification on documents of the positive effects of air pollutants filtration in storage rooms (Havermans and Steemers 2005); 2) The identification of iron-gall inks in a non-invasive way (Havermans et al. 2003); and 3) The quantification of the effect of temporary and permanent exhibitions on documents.

Presented at the Book and Paper Group session, AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

Application tests started out using the multispectral technique and they fully proved the high potential of simultaneous recordings of spectral and spatial information of original documents (Havermans et al. 2003). In fact, on one hand, this technique could allow the classification of certain kind of inks and pigments, and in some cases enhance the readability of damaged and erased texts (Goltz et al. 2007). But, on the other hand, it was found unsuitable to distinguish materials with similar spectral characteristics and it was totally insufficient to produce an accurate quantification of spectral changes caused by aging processes. For these reasons multispectral imaging was replaced by hyperspectral imaging, which has much greater potential to achieve all the research goals.

The first applicability studies initiated the division of the Hyperspectral Project in two main branches. The first one is concerned with using the technique to analyze documents, i.e., a single measurement of an object area analyzed for purposes such as materials discrimination, text enhancement, and diplomatic research. The second branch (covered by the Bihanne Project) focuses on exploring the technique's potential as a monitoring tool, i.e., repeated measurements of the same document, performed at different times, are analyzed and compared for purposes such as the monitoring of aging processes, the evaluation of conservation treatments, and environmental conservation actions (Muñoz Viñas 2005).

THE HYPERSPECTRAL IMAGING TECHNIQUE

Hyperspectral imaging is a remote-sensing technique that enables the acquisition of spectral and spatial information of an object, or parts of it, without getting directly in contact with its surface (Elachi and Zyl 2006; de Jong and van der Meer 2004). The same general principle is used in photography and in human vision, where spectral and spatial information are combined to produce an image of a certain scene. For example, when a digital image is acquired by a standard digital camera the end result is generally a color image composed of three overlapping images that are obtained in three discrete spectral channels (RGB). Therefore a digital camera acts as a three-channel multispectral camera, being able to simultaneously acquire all the spectral information of a certain scene in three specific portions of the electromagnetic spectrum. If more than three channels are used, potentially more information about the recorded scene can be obtained (Feller 2001).

Differences between Multispectral and Hyperspectral Systems

On the basis of the number of channels used in the recording, their contiguity, and their spectral separation, it is possible to define the difference between multispectral and hyperspectral imaging techniques. In a multispectral imaging system the number of independent spectral channels is typically not more than ten, and they are not necessarily contiguously distributed. The amount of spectral information acquired with such systems is therefore limited and the classification of certain materials is only possible if significant spectral differences occur.

As compared to multispectral imagers, a hyperspectral imaging system provides a much greater number of spectral channels, which cover the spectral range in a contiguous way, and therefore provide much more complete spectral information. Basically every individual pixel of a hyperspectral imaging recording carries the information of an entire spectral curve, which enables a more efficient target discrimination and further analysis (Grahn and Geladi 2007).

The Development of a New Instrument

The main challenge of applying hyperspectral imaging, or in fact any other analytical technique, to the study of original documents is to obtain the highest amount of information from the analyzed units while limiting any potential damage provoked by handling the documents and by the instrumentation used. Therefore, during the first part of the project great attention was given to the development of a dedicated hyperspectral imager that could fully satisfy the requirements of accuracy, reproducibility, and non-destructiveness. The instrument, a quantitative hyperspectral imager named SEPIA, was developed in collaboration with the company Art Innovation B.V. (Klein and al. 2006). After its construction, periodic measurement commenced for a number of selected original documents, kept in the Nationaal Archief and in some cases provided by other institutions.

This instrument is composed by two wavelength-tunable light projectors, named TULIPS, which illuminate the document from two sides under an angle of 45°. The TULIPS can be tuned through seventy consecutive spectral channels covering the entire range from the ultraviolet (UV, shortest wavelength 365 nm), via the visible (VIS), into the near-infrared (NIR, longest wavelength 1100 nm). The document is imaged by a 4-megapixel, charge-coupled-device (CCD) camera that is mounted overhead at a distance of about 40 cm. In order to avoid any interference by external light the entire setup is enclosed in a climate-monitored darkroom cabinet. The positioning of the optical spectral filters inside the TULIPS is driven by built-in electronic components, which themselves are controlled by software running on an external personal computer. A graphical user interface gives access to all important settings for each spectral channel such as exposure times, gain, and focus position of the camera, and it provides readings for the operating status of the light sources and the environmental conditions inside the cabinet. For more technical information on the operation of the instrument and the results of performance tests please refer to "Quantitative Hyperspectral Reflectance Imaging" by Klein et al. (2008).

In conventional multispectral imaging, the recorded object is illuminated with a powerful white-light source and the different spectral channels are discriminated by placing

spectral filters in front of the camera. In contrast, the developed hyperspectral imager achieves spectral discrimination by filtering the light in the light sources, before it hits the object. The great advantage of this approach is that during the recording the document is illuminated not by the entire spectrum (white light), but only by the narrow spectral component used to acquire a particular image. As a result, the document area receives the minimum light energy required for the measurement. This is of highest importance especially if multiple recordings of the same document have to be carried out, for example to monitor aging process, since the recording itself could locally induce accelerated aging if a full-spectrum illumination was used for prolonged times.

The Datacube

Data obtained from both hyperspectral and multispectral imaging is generally represented in a three-dimensional reference system, where the so-called spectral data cube is represented. For every point in this datacube two of the three axes (e.g., X and Y) describe the spatial coordinates of the recorded data (i.e. the surface positions) and the third axis (e.g., Z) describes the optical wavelength (fig. 1).

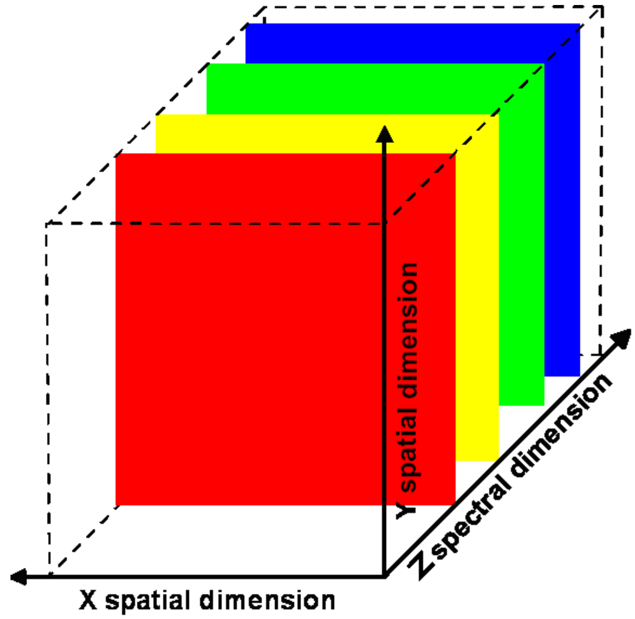

Fig. 1. The three-dimensional shape of the hyperspectral datacube

From the recorded spectral images, the data cube is composed as follows. The pixels of each digital image form a (two-dimensional) matrix of numerical values. After proper calibration of the recorded data, the value of each pixel lies in the range of 0 to 1 and describes the portion of light that is reflected from the corresponding spot on the object at the particular wavelength at which the image was recorded. Each pixel value thus describes a physical quantity, namely the local spectral reflectance of the object.

The entire datacube contains for each pixel the series of numerical reflectance values from all spectral channels, i.e., effectively an entire spectral reflectance curve. For the purpose of a mathematical analysis often an alternative representation of the numerical information contained in the data cube is chosen. The spectral reflectance values of a pixel can in fact be regarded as the elements of a particular vector in a high-dimensional mathematical space. Although being much less intuitive than spectral curves, the representation as such abstract spectral vectors makes it possible to apply a vast array of mathematical tools, developed, for example, to distinguish certain target materials and map their occurrence on the object with pixel accuracy.

THE BIHANNE PROJECT

The Bihanne Project is divided in two main sections: 1) the study of natural aging process of original documents, and 2) the accelerated aging process induced on sample materials. These two branches of the research are currently used to build two databases that give valuable information about the possible application ranges, limits, and practical procedures to be considered when applying hyperspectral system as a monitoring tool. In the specific case, of the internal exhibition room at the Nationaal Archief, the natural aging of the exhibited documents should be minimal, because each item is allowed to be exhibited for a maximum period of four months per year (53 hours per week) at an illumination intensity of 50 lux. Considering the relatively low light dose accumulated in a single four-month period (42400 lx·h) only very small spectral changes are expected, even for photosensitive materials (Thomson 2000). This is why considerable effort was put in the technical development of both the instrument and the recording procedure to achieve maximum reproducibility and sensitivity of the hyperspectral measurements.

Measurement reproducibility

One of the major challenges in the Bihanne Project has been to achieve the required reproducibility of the hyperspectral measurements to compare recordings of the same documents made at different times. Over the past three years, the recording and calibration procedures, which are crucial for the measurement reproducibility, have been constantly improved. In particular, the hardware and the control software of the instrument were adapted and a special recording procedure was adopted to minimize the thermally induced drift of the measured values. In order to verify the achieved reproducibility between several measurements of the same document, reference targets are placed within the recorded area. Differences observed between spectral data cubes, recorded for the same document area, can now be related to actual changes of the physical and chemical condition of the document rather than reflecting the imperfections of the apparatus itself.

Another important element taken into consideration for the reproducibility of hyperspectral recordings of documents was the surface of the stage that supports the analyzed sample and thus serves as the optical background of the measurement. In fact, many writing substrates can be translucent to some degree so that the reflectance spectra measured by the instrument are slightly influenced by the background. To give a practical example, when the page of a bound volume is recorded a small amount of the light is transmitted through the measured page and reflected by the underlying pages of the textblock. In a subsequent measurement of the same page, the underlying sheets may have shifted slightly or their reflectance characteristics may have changed, resulting in an interference source for the measured page. In our case the document support received a finish of specially selected flat black paint that is highly light-absorbing over the entire spectral range used by the SEPIA instrument.

The Reference Targets

Generally the calibration of hyperspectral datacubes is based on the comparison of the data obtained from the measured object (having unknown spectral characteristics), to the data obtained from one or more reference targets (having known spectral characteristics), when both are exposed to the same recording conditions. For example, in the airborne scanning of the Earth's surface, reference measurements are carried out by using special target sheets, placed inside the recorded area, or by collecting *in loco* spectral values of homogeneous areas of the ground (Elachi and Zyl 2006). The reference measurements used in the calibration process make it possible to compensate for varying recording conditions (e.g., sun illumination and atmospheric changes) and for possible instrumental errors. Moreover the pixel values in the hyperspectral data cube can be associated with a specific physical quantity such as spectral reflectance.

In our case using a spectrophotometer to obtain reference data from the measured object itself would not provide sufficient accuracy, because such a spot analysis would not produce an effective evaluation of the recording conditions over the total recording surface. In fact the typical inhomogeneity of document surfaces would seriously impede the reproducibility of such type of reference measurements. As an alternative, data sets are recorded directly with the instrument from three types of targets that cover the recorded area in part or completely.

Primary Reference Target—The primary reference target used is a 128 x 128 mm Spectralon tile that covers the entire field-of-view of the SEPIA instrument (fig. 2). It is provided by the company Labsphere and it has been chosen for its high diffuse reflectivity, as in fact more than 99% of the light is reflected from its surface over a wide range of the light spectrum (230 to 2000 nm). In addition, this calibration standard is provided with a certificate stating its reflectance values according to the standards set out by the National Institute of Standards and Technology. The Spectralon tile is used as the primary reference to correct instrumental errors and provide an absolute, long-term calibration enabling a pixel-per-pixel calibration of the datacubes and a correct evaluation of inhomogeneities of the light distribution over the recording area.

Recording Verification Target—In order to detect any major recording malfunctions during the analysis of the investigated documents or test samples, a reference target is placed inside the recorded area of both the documents and the Spectralon (figs. 2–3). This target was realized with a strip of Whatman paper grade number1 (230 x 20 mm) fixed by the two short sides on a 1 mm thick preservation board

Fig. 2. The Recording Verification Target (bottom strip) and the Reproducibility Target (top strip) during the recording of a test document

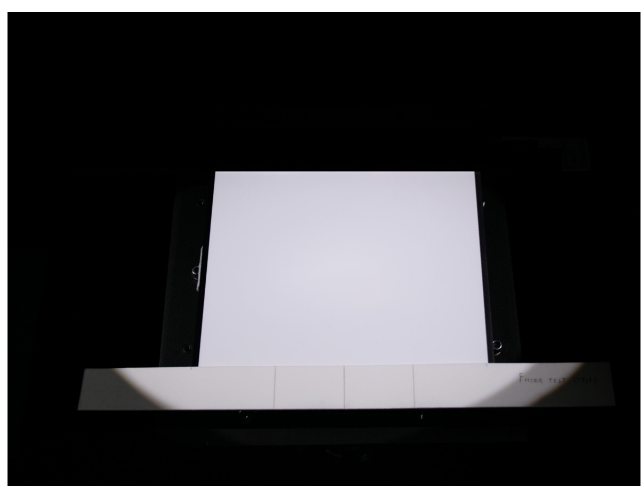

Fig. 3. The primary target (Spectralon) with the Recording Verification Target placed partially on top of it, in the position used for the recording of the document

(with identical dimensions) using Evacon-R adhesive. The fixation of the paper was necessary because its translucency, in combination with an inhomogeneous background, could have caused unpredictable variations of its recorded spectral characteristics, as discussed above. To avoid any changes of the quality of the Whatman paper the adhesive was only applied to the border of the strip, in an area external to the field of view of the imager.

Reproducibility Target—This target is inserted only in the recording of the monitored document or sample test (fig. 2). It is used as a reference to verify that eventually observed spectral changes, found during the comparison of multiple recordings of the same document, would not be caused by a measurement error but by an effective changing of the physical and chemical characteristics of the document. This target is realized in multiple copies with the same procedure used for the Recording Verification Target (described in the previous paragraph) and each of them is associated to one single document recording. Because these targets are constantly preserved in darkness, under optimal climate conditions, and reused only when a new recording of the same document area is performed, they provide a direct reference for the reproducibility of the measurements in time.

The Recording Procedure

The recording procedure used in the Bihanne Project, for monitoring of both accelerated and natural aging processes, is based on daily recording periods of approximately eight hours in which a total of four investigated documents can be analyzed. Each section is divided in thirteen independent recordings of thirty-five minutes. The first two are used to warm up all the components of the instrument and their data are not stored. In the following, a document recording is always calibrated by interpolating the reference data from four Spectralon recordings, namely two recordings made before and two after the document recording itself (table 1). This is why the session starts and ends with two subsequent recordings of the Spectralon target, and between these pairs the documents and the Spectralon are recorded alternately.

Monitoring Natural Aging

At the moment only a small number of selected original documents are monitored using the hyperspectral technique. These documents, due to their national and international importance, are constantly exhibited in the internal exhibition room of the Nationaal Archief or they are sent to external institutions for temporary exhibitions. The accumulation of data started in the year 2006 and continues to the present time, resulting in more than fifty document areas that have been repeatedly measured before and after their exhibition periods, keeping record of the conditions in which documents were exhibited. The analysis of the data stored in this growing database is still in development and many improvements are constantly performed in order to extract more information from the monitored documents. For this reason, and also because it was necessary to wait a sufficiently long period of

Recordings	Uses	Storage
WU1	Warming up	Erased data
WU2	Warming up	Erased data
01_SPECTRALON-S99	Target used to calibrate Document 1	Stored data
02_SPECTRALON-S99	Target used to calibrate Document 1 & 2	Stored data
03_DOCUMENT 1	Recording of the investigated Document 1	Stored data
04_SPECTRALON-S99	Target used to calibrate Document 1 & 2 & 3	Stored data
05_DOCUMENT 2	Recording of the investigated Document 2	Stored data
06_SPECTRALON-S99	Target used to calibrate Document 2 & 3 & 4	Stored data
07_DOCUMENT 3	Recording of the investigated Document 3	Stored data
08_SPECTRALON-S99	Target used to calibrate Document 2 & 3 & 4	Stored data
09_DOCUMENT 4	Recording of the investigated Document 4	Stored data
10_SPECTRALON-S99	Target used to calibrate Document 3 & 4	Stored data
11_SPECTRALON-S99	Target used to calibrate Document 4	Stored data

Table 1. Daily recording section

Substrates	Writing products	Adhesives	Conservation treatments	Original documents
Whatman paper Grade nr.01	Iron gall ink	Methylcellulose (MC)	Reducing (Tert-butyl-amine borane)	Original document with typewritten text (1941)
Romandruk paper (wood-pulp based)	White lead	Carboxyimethylcellulose (CMC)	Oxidative (Hydrogen peroxide)	Printed book with foxing (late 19th c.)
Eucalipto paper	Copper green	Tylose	Deacidification (Bookkeeper)	Document with iron gall ink (1920's)
Silversafe paper	Brazil wood	Evacon-R		Document with aniline ink (1920's)
Calf skin	Saffron yellow	Gelatine		
Goat parchment				
Cotton				
Linen				

Table 2. Materials actually investigated in the Bihanne Project

time in order to record natural aging processes, preliminary results cannot yet be presented. From September 2009 new analytical tools will be applied to speed up the managing of these data, improving also the quality of the results.

Monitoring Artificial Aging

The acquisition of hyperspectral data from original documents is of extreme importance to understand the natural aging process of their component materials. But in order to predict the long-term effect of certain exhibition or storage environments it is necessary to produce specific aging models by applying accelerated aging to test samples. For this reason within the Bihanne Project a set of artificial samples was manufactured and aged in different ways to create aging models of materials generally found in the production and restoration of archival documents. The hyperspectral measurement of these artificially aged samples is expected to provide valuable information about the type and the intensity of spectral changes that can be expected. In addition, these measurements will help to establish the optimal recording and analysis procedures to be used in standardized monitoring methods in archive institutions. The selection of the materials investigated in this branch of the Bihanne Project is shown in table 2. They are divided in five main categories: substrates, writing products, adhesives, conservation treatments, and original documents. Great attention was given to the use of standardized materials and their production recipes in order to create reproducible samples to be combined in different ways and exposed to different artificial aging process.

Accelerated Aging—All the materials tested until now have undergone three types of artificial aging procedures. The first procedure addresses accelerated light aging of the samples as can be expected to occur during an exhibition period in the internal exhibition room of the Nationaal Archief. In fact, a specifically designed artificial light aging cabinet (ALAC-2) imitates as closely as possible the actual illumination conditions in which original documents are exhibited. Conventional artificial light aging of sample materials is usually performed by using climate chambers fitted with UV filtered Xenon lamps. As opposed to this, the ALAC-2 features four UV free lamps identical to the ones used in the exhibition room (Whitestar UV-P, 12v 50W 4200K). In addition, the same glass cover as used in the showcases, is inserted in the light path between the lamps and the samples. In this way it has been possible to recreate in a laboratory environment the identical spectral characteristics of the illumination used in the exhibition room but with a higher intensity (circa 1200 lux instead of 50 lux). The lamps are fitted with a forced-air cooling system and the temperature at the sample documents is constantly monitored during the aging process in order to exclude any influence of thermal aging of the samples.

The second aging procedure is based on the variation of climate conditions (temperature and relative humidity) to induce aging process on the tested materials. This type of accelerated aging was applied to the samples by using either of two different climate chambers: Heraeus-Vötsch VTRK 150 or the MMM Group Medcenter Climacell-111. Both were programmed to provide different sets of aging cycles, in order to verify differences in their effect on the spectral characteristics of the tested materials.

In the VTRK 150 cabinet the samples were kept at a stable temperature of 80° C for 15 hrs while the relative

humidity was cycled three times between 35% and 80%. The second cabinet was programmed to provide a stable temperature of 70° C for 15 hrs and three full cycles of the humidity parameter between 35% and 75% (Bogaard and Whitmore 2002).

The third aging procedure addresses the effects that occur when combining environmental stress and exposure to light. A complete aging cycle according to this third procedure is implemented by sequentially applying to the samples the first aging procedure described above (light) followed by the second (temperature/humidity).

The Recording of the Samples—Each test sample of the used materials was shaped in a rectangle of 120 x 180 mm. This rectangle was then divided in four identical strips of 30 x 180 mm. Each one of these strips underwent a different aging process: natural aging (kept in darkness and used as a reference strip); light aging; climate aging; and light plus climate aging. Recordings of the samples were always carried out before and after every aging cycle in order to be able to measure a progressive changing in the spectral characteristics of the tested materials. To minimize any possible error caused by the positioning of the samples in the area measured by the instrument, a special sample holder was designed (fig. 4).

To reduce the effect of back reflection for translucent materials this holder provides a homogeneous, flat-black surface onto which the samples are mounted. Using eight screws, four sample strips are fixed next to each other on the holder. On top of the samples a grill is placed to press the borders of the sample strips on the holder surface, keeping them flat. This is important because some sample materials, such as parchment, can be considerably deformed by the climate aging. On top of the grill four reference strips with a gray pattern are arranged to verify the working of the instrument. The same grill is also used for the Spectralon calibration recordings, where spacers are used to place it at the same height and position as during the sample recordings.

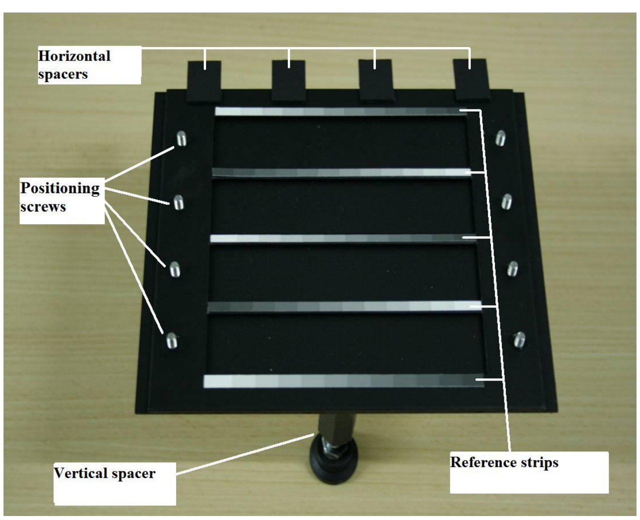

Fig. 4. The sample holder developed to reduce positioning errors of the artificially aged samples when undergoing the recording procedure. A grill fixed on the recording plane with eight screws reduces problems caused by the possible deformation of the samples

RESULTS

The materials tested up until the present time underwent five aging cycles and they were recorded before and after each cycle using the procedure described in the "Recording Procedure" section. The corresponding six recordings are labeled "R0" and "R1" to "R5, where "R0" denotes the zero-measurement before any aging was performed and "R5" the measurement after the fifth aging cycle. An additional recording, labeled "R4-1," was taken after having stored the recorded samples for three days in darkness (i.e., between the fourth and fifth aging cycle). This was done to verify whether any measurable processes occur while the samples are stored in darkness under optimal environmental conditions.

It is beyond the scope of this paper to present and discuss all the results obtained for the tested materials and the different aging parameters. Therefore, only one example was selected to discuss the analysis methods and demonstrate the type of information that can be obtained by using the quantitative hyperspectral imaging technique for monitoring induced aging processes. From the selected sample shown in figure 5, which was cut from a discarded paper document dated 1941, spectral data was extracted from four regions-of–interest (ROIs), which in this case were all defined in the paper substrate in the four differently aged areas. This particular sample was measured seven times over a period of approximately six months, during which it underwent five aging cycles. Each color represents a particular ROI from which the mean values of the pixels, for all the seventy spectral channels, were extracted resulting in a mean spectral curve for each ROI. The ROIs are denoted as follows: R = reference strip; L = light aging; RH/T = climate aging; and RH/T/L = climate aging followed by light aging.

The first and very important step of the analysis of data was to verify the reproducibility of monitoring measurements. This was done by comparing the mean spectral curves extracted from all six data cubes for the ROI defined on the reference strip (not aged). This part of the document is not artificially aged at all and it is stored in darkness under optimal climate conditions. For this reason a minimal change of the spectral reflectance curve is expected for the ROI defined on this sample. As shown in figure 6, the spectral curves extracted from the six recordings (R0-R5) of the reference strip (R) show indeed only very small differences. They are probably the result of a combination of residual measurement errors of the instrument,

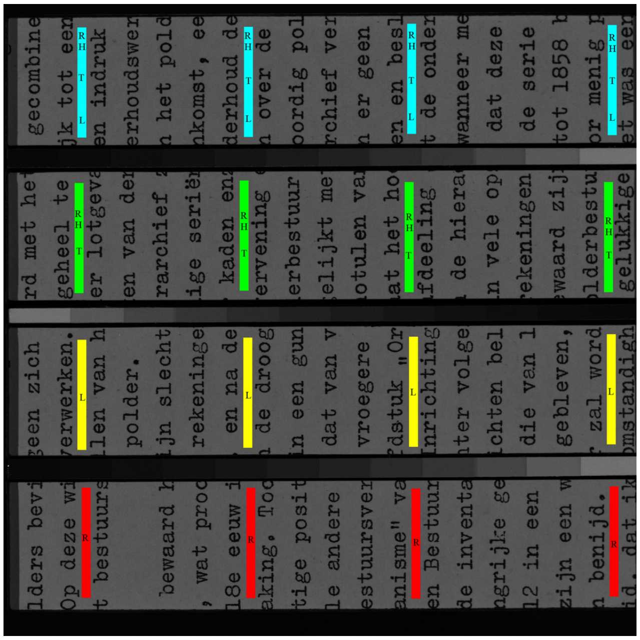

Fig. 5. Selection of four regions-of-interests (ROI) in a hyperspectral datacube of a discarded paper document dated 1941. Each ROI is defined in areas of the document that have undergone different aging process (R: not aged; L: light aged; RH T: climate aging; and RH T L: combination of light and climate aging)

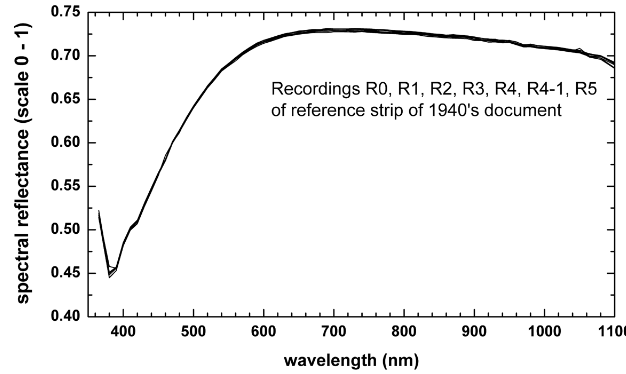

Fig. 6. Mean spectral curves of the not-aged reference strips for the six recordings of the artificial aging tests. All six curves lie very close together, confirming the high reproducibility of the measurement

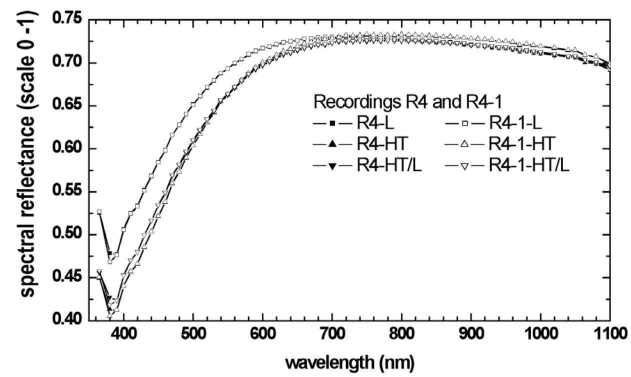

Fig. 7. Comparison of the spectral curves of the aged samples before and after three days of storage in darkness. For all three types of artificial aging: by light (L), by humidity/temperature (HT), and by the combination of both (HT/L); no significant effect of the storage period was measured

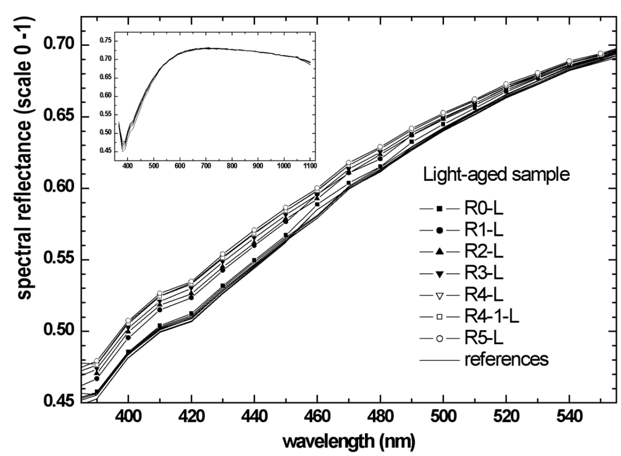

Fig. 8. Spectral curves of light-aged samples for different numbers of aging cycles applied. For comparison, the corresponding curves of the reference sample are plotted as plain lines. Insert: Overview of entire wavelength range. The most significant changes are in the blue-green range of the visible part of the spectrum

imperfections of the measurement procedures, and possible changes of the sample itself, even when stored under optimal conditions. If in the same measurements larger differences are observed for the spectral curves of the other aged sample strips, this will confirm that this type of change could be caused only by the differences in the aging process of the paper.

Once the reproducibility of the measurement was verified it was also necessary to test the stability of the spectral characteristics of the sample after a period of darkness (fig. 7). In fact it has been reported in the literature that some materials that have undergone accelerated aging can show changing in their spectral characteristics when stored in darkness (Lee et al. 1989; Strilič et al. 2004). This effect is explained by the progression of the chemical reactions, induced by the exposure of certain materials to external energies (such as high temperatures and light), even when the exposure is interrupted (Haillant et al. 2004; Mukherjee 1978). This type of information is thus useful to quantify how long after each artificial aging cycle the corresponding measurement can be performed before the progression of the chemical reactions modify the resulting spectral values (Bukovský and Trnková 2003). In order to verify this, the same document was measured directly after the end of an aging cycle (recording R4) and a second time after three days of storage in darkness (denoted as R4-1 recording). Figure 8 shows that for this type of paper, during such a short period of darkness,

the spectral values have not changed in a relevant manner. In fact, the ones extracted from the three different aged areas (L; RH/T; RH/T/L), from recordings R4 and R4-1, overlap almost perfectly over the entire spectrum.

In order to verify possible spectral changing of these values after longer periods of darkness, the same samples will be recorded again after six months of storage. Figure 8 shows the spectral curves of the light-aged sample strip for the recordings R0 to R5. The light aging applied between two subsequent recordings corresponds to approximately one month of exposure in the exhibition hall of the Nationaal Archief. With progressive aging the curves show a small but systematic increase of the reflectance values of the paper in the visible region of the spectrum, whereas the values in the infrared remain practically constant. The total change in the reflectance curves between the R0 and the R5 recording is only about 2% in the visible range; however, this is still about five times more than the variation observed for the same recordings on the reference strips. From this it can be concluded that with the quantitative hyperspectral technique it is possible to identify spectral changes incurred after an exhibition period of only one or two months.

Since the reflectance curves mainly change in the visible violet-blue region of the spectrum (400–500 nm), the effect of the light aging is potentially visible to the human eye as bleaching of the paper (Neuvirt 2005; Schaeffer 2001). Due to the high spectral resolution of the hyperspectral imager, the spectral curves can be used to calculate the CIELAB color indices $L^\star$, $a^\star$, and $b^\star$ and, from those, the color difference (ΔE) of the sample between any two recordings. For the standard illuminant D65, the difference in the spectral curves of the sample in the R5 and in the R0 recording corresponds to a color difference of $\Delta E = 1.35$ for the equivalent four months of exhibition. For a trained person, in direct comparison and at sufficient light levels, a color difference of $\Delta E >= 1$ can be distinguishable whereas for the average human observer a threshold of $\Delta E >= 3$ is more typical. This means that after applying light aging corresponding to only one or two months of exhibition, the induced spectral change can already be measured with the instrument while it is not yet visible, even for a trained person in good lighting conditions.

CONCLUSIONS AND CURRENT DEVELOPMENTS

Preliminary results obtained by the Bihanne Project, with both natural and artificial aging, have already shown that quantitative hyperspectral imaging has a great potential as a monitoring tool for archival documents. The most promising result of this research is the possibility to measure minute changes of the reflectance spectra even after short exhibition periods in optimal environmental conditions and provide a map of their distribution. The experiences accumulated until now with this project have also shown that the quality of the data, the accuracy and sensitivity of the instrument used, and above all, the reproducibility of the recordings over a period of several months, are crucial parameters when applying hyperspectral imaging as a monitoring tool. A considerable part of the research described in this paper addresses the development of suitable recording procedures that minimize the risk of measurement errors. Such procedures are still being improved in order to obtain a final standardization for the application of the hyperspectral imaging technique in the normal working flow of archives and libraries.

Current research within the Hyperspectral Project at the Nationaal Archief addresses the further improvement and simplification of the recording procedure so that more samples can be measured without compromising the accuracy. A second important and very challenging issue is the development of new, efficient methods for comparing, with maximal spatial resolution, the data cubes of multiple recordings of the same object taken at different times. The goal is based on the measurement of the original and the artificially aged samples to develop aging models of different material systems and identify the most representative spectral channels to be used for monitoring purposes. Such information will provide conservators with the knowledge required to apply the quantitative hyperspectral imaging technique in the most efficient way to monitor the condition of historic documents even with less advanced and easily affordable instruments.

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Using Tycore Board as Mounting Panel for Oversized Charcoal Drawing

ABSTRACT

The oversized charcoal drawing *Passers-by on the Square* by Taiwanese artist Ching Jung Chen was donated to the Kaohsiung Museum of Fine At. This drawing, approximately 339 cm in width and 230 cm in height, had previously been folded. It was also torn and had been repaired with tape. Treatments such as consolidation, tape removal, humidification, and flattening were carried out to stabilize the piece. Due to its enormous size, it was impossible for the museum to store this drawing in flat format. The alternative was designed and executed using three pieces of Tycore board as backing panel. The boards were hinged with Japanese paper and then glued together into one large piece. The backing panel was then pasted onto layers of bast-fiber papers. The drawing with paper-strip extensions was then mounted onto the panel. After framing, it was stored vertically and secured onto a wire frame. This paper will describe the procedures of the whole project and the challenges encountered during its execution.

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Presented at the Book and Paper Group session, AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

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Effect of Aqueous Treatments on Nineteenth-Century Iron-Gall-Ink Documents: Assessment Using Hyperspectral Imaging

ABSTRACT

Five original, nineteenth-century, iron-gall-ink documents were subjected to eighteen separate aqueous stabilization treatments. The effectiveness of eight of these treatments was evaluated after extended exposure to heat and humidity, high-intensity light, and elevated humidity at room temperature. Heat aging results confirmed the effectiveness of phytate-bicarbonate treatments. High humidity did not cause mold growth on phytate-treated samples. Continuous exposure to fluorescent lights (with UV) caused fading of the papers and some fading of the inks. Visible hyperspectral imaging was carried out using a NuanceTM Imaging system with a liquid crystal tunable filter (LCTF). The usefulness of hyperspectral imaging to evaluate the effect of aqueous treatments on one set of sample is discussed.

INTRODUCTION

The efficacy of iron-gall ink treatments is often tested on model papers and prepared inks in order to control the variables during experimentation. Evaluating these treatments on originals helps identify unforeseen problems and confirms their effectiveness (Neevel 2000; Reissland 2000; Kolar and Malešič 2005). The calcium phytate (Ca-phy) calcium bicarbonate ($Ca(HCO_3)_2$) treatment was consistently found to be effective in protecting model papers and inks during artificial aging. This study uses original documents (circa 1841–1875) donated by a Québec archive, and compares the effect of the phytate-bicarbonate and modified phytate treatments to other aqueous treatments. Phytate treatments are sometimes modified to eliminate crystal formations after treatment and/or the possibility of mold growth (Homolka 2001).

A total eighteen treatments were carried out on nine original documents (Orlandini 2006; Orlandini 2009). The effectiveness of eight of treatments on five documents was further tested by exposure to heat and humidity, high- intensity light, and elevated humidity at room temperature. Changes were evaluated against unaged or untreated controls. Methods of evaluation include hyperspectral imaging, color measurement, pH, iron (II) testing using bathophenthroline test strips (Neevel and Reissland 2005), and microfade testing. Some of these results have been previously reported (Tse et al. 2006).

Hyperspectral imaging combines spectroscopic information with the spatial information of the sample. In hyperspectral imaging spectral information is collected in narrow bands (10 nm) that are contiguous. In this sense spatial information about an object is obtained in two dimensions (x, y) and spectral information is obtained in a third dimension (z), which allows the storage of information in a three-dimensional data cube.

The important advantage of using hyperspectral imaging is that an accurate digital record can be acquired of an historical object (Kubik 2007). This makes it a very powerful technique for identifying and mapping pigments in paintings (Berns 2002; Baronti 1998). For written documents hyperspectral imaging has been used for enhancing the legibility of faint text (Goltz 2007), as well as revealing text that was over-written with another ink (Attas 2004).

This paper highlights the usefulness of hyperspectral imaging and image analysis techniques to evaluate the effect of aqueous treatments on iron-gall ink.

EXPERIMENTAL

Samples

The nine documents were previously donated for testing by an archive in the province of Québec, Canada, and subjected to eighteen different aqueous treatments. Details of the treatments have been described by Orlandini (2006; 2009). Five of the documents and eight of the treatments were subjected to further accelerated aging. A description of these samples can be found in table 1. Results from bathophenanthroline test strips were recorded using a calibrated color chart developed at the Canadian Conservation Institute (CCI),

Presented at the Book and Paper Group session, AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

Sample #	Sample description	Ink condition				
		ICN Condition Rating	Bathophenthroline Test strips		FTIR-ATR	μXRF
			Fe^{2+}	Fe^{3+}		
1	1856 cream ledger, wove cotton rag paper with thin blue lines; ink dark brown	1	50+	50+	gelatin	Fe, K
2	1849 greyish cream ledger; wove cotton rag paper with thin blue lines; ink light brown	1	~10	50++	No gelatin	Fe, K
3	1864 blue ledger; laid cotton rag paper with visible chain and laid lines; ink light brown with dark strokes	1	~25	~25	gelatin	Fe, Ca, S
6	1846 cream ledger; wove cotton rag paper with no lines; ink thin dark brown strokes	1	50	50+++	gelatin	Fe, K
9	1846 green ledger; wove cotton rag paper with no lines; ink light brown	1	~10	~10	No gelatin	Fe, Cu, Mn, Ca, Cl, K

Table 1. Description of treated inked documents

where 1 = detectable; 10 = weakly positive; 25 = positive; and 50+ = strongly positive (fig. 1) (Vuori and Tse 2005).

Treatments

An untreated portion of each original document was kept as a control, and the treatments are described in table 2.

Methods of aging

One set of untreated and treated samples was kept for unaged controls. Heat aging was carried out in a Despatch Environmental Chamber LEA-69 at 80° C and 65% RH for eight weeks. Light aging was done with a bank of fourteen fluorescent lights (40-watt, 4-foot 1157 Vita-lite), vertically mounted, 15–20°C, 40±5% RH for ten weeks, without UV filters. Averaged accumulated irradiance was 5564 kJ/cm^2. The total light exposure was 3.71Mlux-hr. Humidity exposure was carried out in a desiccator maintained at 85% RH with a saturated KNO_3 solution, at 22°C, for twenty-two weeks. All the samples were mounted separately during aging. The samples after treatment and after aging are shown in figure 2.

Methods of evaluation

Visual evaluation was conducted by eleven paper conservators. Color measurements before and after aging were done using a Minolta 2022 spectrophotometer. The presence and recurrence of the ferrous (Fe (II)) and ferric ions (Fe (III)) in all aged samples were tested using bathophenanthroline indicator strips, with and without ascorbic acid, and compared to a calibrated color chart developed at CCI. Attenuated Total Reflection (ATR) IR spectra of selected ink and paper were obtained using the Travel IR ATR spectrometer (SensIR Technologies, Smiths Detection). Micro-extracted pHs of selected papers and inks were

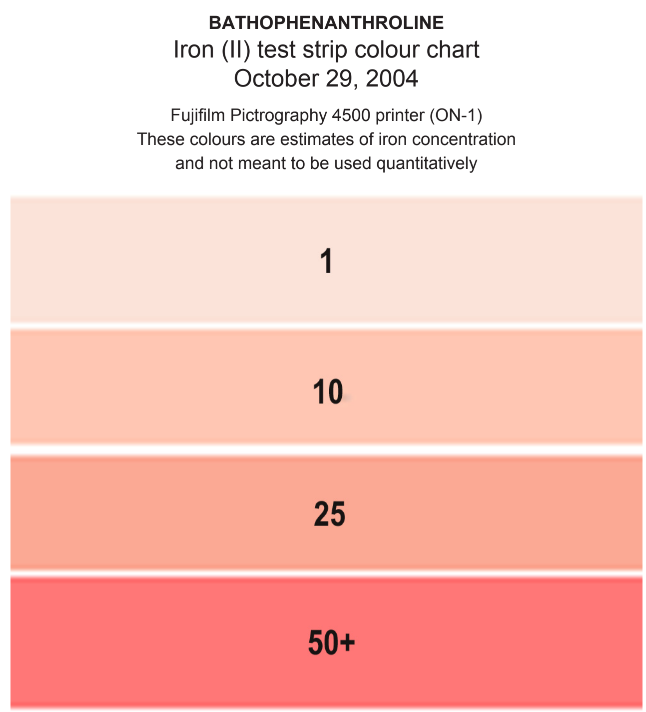

Fig. 1. Color chart developed by CCI for documenting bathophenanthroline Fe (II) test strip results

measured using a IQ-240 ISFET pH meter with microprobe. Calibrated Scan Mate F8 flatbed scanners, conventional, UV-fluorescence, and IR photography were used to document the appearance of the samples.

In 2008, visible hyperspectral imaging of the aged samples and controls was carried out at the University of Winnipeg, using a Nuance MSI (420–720 nm) multispectral imaging system

	Treatment Sequences & Duration
1	Untreated control
2	Alkaline wash: pH 8.5 $Ca(OH)_2$; 20min
3	RO water + 0.011M $Ca(HCO_3)_2$; 20min each
4	Reverse osmosis (RO) water + 0.086M $Mg(HCO_3)_2$; 20min each
5	Ethanol (EtOH) immersion + alkaline water simmer: pH 8.3 $Ca(OH)_2$; 90°C; 15min
6	Pre-wet with EtOH spray; Ca-phy + 0.011M $Ca(HCO_3)_2$; 20min each
7	Ca-phy + 0.011M $Ca(HCO_3)_2$; 20min each
8	Ca-phy diluted 1:1:1 with water and ethanol + $Ca(HCO_3)_2$; 20min each
9	Pre-wet with EtOH spray; Ca-phy (20min)+water rinse (3x10min)+$Ca(HCO_3)_2$ (20min)

Table 2. Treatments applied to original documents

(Channel Systems and CRI) equipped with a liquid crystal tunable filter, optics, and a charge-coupled-device (CCD) detector. The image sensor pixel count of the CCD is 1.3 megapixels and images were 1248 x 960 pixels. Images were acquired from 420 nm to 720 nm at intervals of 10 nm.

For visible imaging, Solux bulbs (3500K, 35W) (Tailored Lighting Inc.) were placed at approximately 0.5 m from the documents, at 45 degrees relative to the imaging camera. For visible imaging with an ultraviolet light source, a low-pressure fluorescent light source was used. This was a broad-band light source with a maximum intensity of 365 nm. The imaging camera was mounted in a face-down position at a distance of approximately 50–100 cm from the samples. An AF Micro Nikkor 105 mm f2D lens, mounted to the imaging camera with a C-mount, was used. An f-stop of 5.6 was used for visible imaging with the visible light sources and a fully open f-aperture (f/2) was used with the UV light source.

The white-reference standard used for this work was a 20 x 20 cm halon panel (98% reflectance at 550 nm). The data cube of the document was flat-fielded by dividing the pixel intensity of the document by the corresponding pixel intensity from the white cube reference image. The flat-fielded images were then processed with units of optical density (absorbance):

$$\text{optical density} = -\log (I_{image} \div I_{white})$$

All of the spectra are presented in units of optical density except where indicated (i.e., fluorescence imaging). Data from imaging experiments that were saved as cube files could also be treated statistically using algorithms in an imaging software package known as ENVI (Environmental Visual Imaging, version 4.2).

3 RESULTS

Extended exposure to heat and humidity

Artificial heat and humidity aging was used to evaluate the effectiveness of treatments to protect the inked samples. Visual evaluation, color measurements, and Fe (II) tests of heat-aged samples showed that the calcium phytate-bicarbonate combination—with or without ethanol spray, even with rinsing—gave the most effective protection. Dilution of Ca-phy with ethanol and water (1:1:1), without repeating treatment, reduced the effectiveness. Simmering, a controversial treatment for iron-gall ink (Tse et al. 2005), was found to be effective immediately after treatment. The color of the paper and some inks became lighter after treatment. After heat aging, simmered papers remained the brightest, but there was some haloing around some ink lines, and recurrence of soluble Fe (II) ions. Alkaline water wash and deacidification alone were the least effective, as there was significant yellowing and recurrence of soluble iron ions.

Extended exposure to high-intensity light

Exposure to high-intensity, unfiltered, fluorescent light was used to study the fading of the inks. Color measurements using the Minolta hand-held spectrophotometer (with a 8mm sample port) showed that the paper was bleached after light exposure. Evaluation of the change of ink color was not satisfactory because of the relatively large port compared to the thin ink lines. The Oriel Microfade tester will be used to determine the light-sensitivity of the inks and measure the average color change with and without light exposure. FTIR-ATR results showed that simmering removes some sizing and binder from the ink. Other aqueous treatments do not result in a detectable change in the compositions of ink or paper.

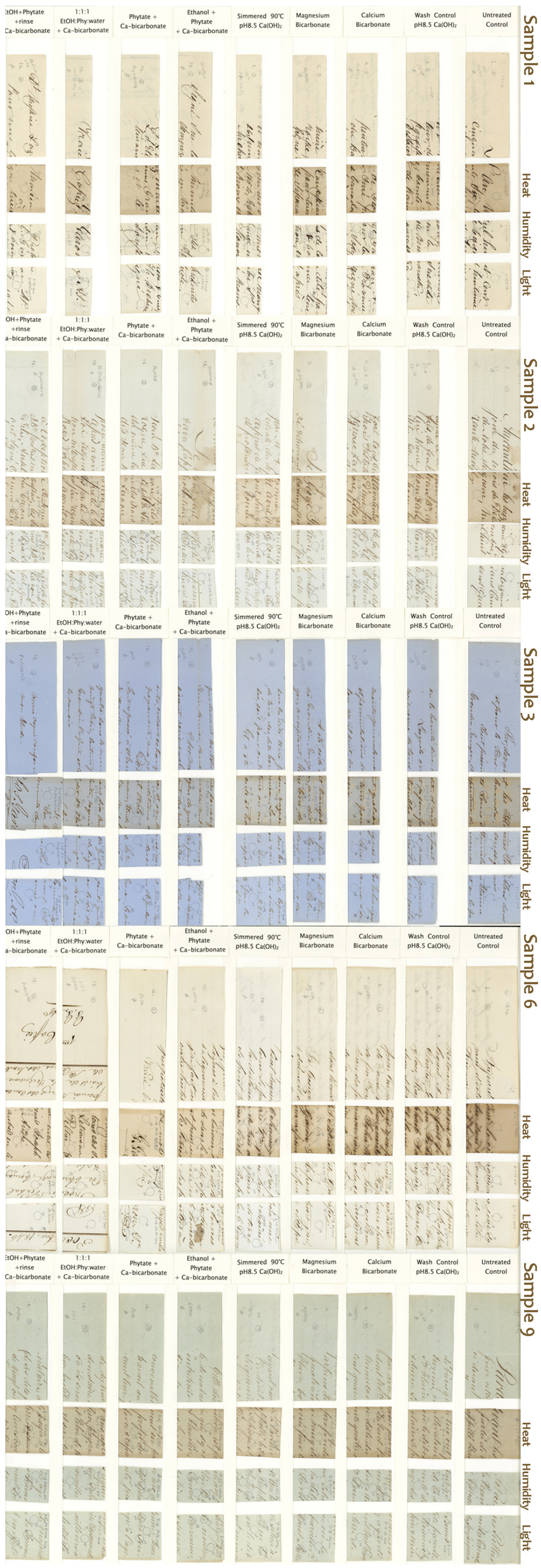

Fig. 2. Five sets of aged and treated iron-gall ink documents. Each column represents a different aging method: 1) unaged controls; 2) heat; 3) humidity; 4) light. Each row of samples represents a different treatment described in table 2

Extended exposure to high humidity

A high-humidity environment is not suitable for iron-gall ink documents, not only because of an increased rate of acid-catalyzed hydrolysis, but also because of the likelihood of ink migration—both laterally and also through the thickness of the paper resulting in "strikethrough" (Eusman and Mensch 2000; Reissland 2000). (Assessment of ink migration is discussed further in section 3.5.)

A secondary concern with high humidity is the possibility of mold growth for phytate-treated documents (Homolka 2001). In this study, the phytate treatment is modified by dilution with ethanol and water, as well as inclusion of a rinsing step, and their effectiveness is evaluated. Exposure to 85% RH for twenty-two weeks did not result in mold growth in any of the samples, including all the phytate-treated ones. This suggests that in a clean environment, not infested with active mold or spores, calcium-phytate treated samples do not become more susceptible to mold growth. For conservators who are still concerned that phytate treatment may be a problem, these results suggest that rinsing the artifact after phytate treatment, to remove excess phytate, would not diminish its effectiveness.

Feasibility of visible HSI for monitoring ink changes after treatment and after exposure to heat, humidity, and light

The most important advantage of using hyperspectral imaging (HSI) is the quantity of spectral data that can be collected in a relatively short time. One image cube contains more than 10^6 pixels, and each pixel represents a visible spectrum (420–720 nm) for a given spatial location. For ink studies this is especially important. Most conventional color measurement instruments have sample ports that are much too large (typically 3–8 mm in diameter) for the width of the ink line (typically less than 1 mm). Therefore it is not possible to obtain spectra of ink alone without the surrounding paper, making it impossible to follow changes in the ink as a result of treatment or aging.

With these samples, single wavelength images were collected from 420 to 720 nm at 10 nm intervals using both ultraviolet (365nm) and visible light sources. The highest contrast between paper and faint portions of ink was achieved at short visible wavelengths. Therefore images under UV illumination were collected at 430 nm with various exposures and are shown in figure 3.

Using visible HSI, we are able to measure spectral changes (fading, darkening, and changes in hue and chroma) in the ink as a result of treatment and accelerated aging. It is,

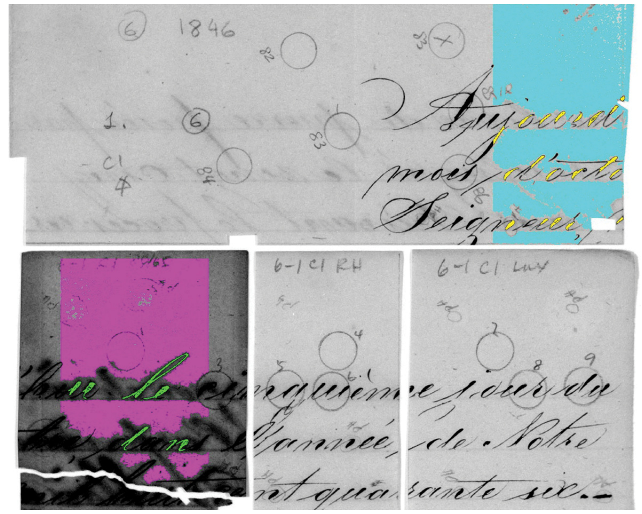

Fig. 4. Four regions of interest (ROI; 1000 pixels) for sample 6

however, limited by the variation (non-uniform nature) of the ink lines, typical of any original ink documents. So it can be difficult to determine whether differences in the ink are a result of treatment, aging, or simply sample variation. The criteria for selecting pixels becomes critical.

Figure 4 shows four regions of interest (ROIs) that were selected for sample 6. In this figure, the blue (control) and purple (artifical aging) ROIs represent pixels selected for paper, and the yellow (control) and green (artificial aging) pixels represent ink pixels. This figure illustrates the specific ROIs of the document that were selected for generating spatially accurate visible spectra of paper and ink shown in figure 4. Apart from examining visible spectra, measuring change in the visible spectra before and after treatment is also a useful tool. Determining differences in the visible spectra can be achieved by subtracting the average pixel optical density of inks that have undergone aqueous treatments from the control ink. The results are summarized in figure 5.

Spectral change occurred in most of the iron-gall inks after accelerated aging. By selecting a large number of ink pixels for each paper, the differences in ink thickness can be minimized. The ink and paper that were exposed to UV and humidity showed less change in optical density than those after heat aging. All of the heat-aged inks became darker, with an increase in optical density of 0.1 to 0.2.

Even when the largest number of pixels was selected (>1000), incorrect conclusions are possible, especially when treatment comparisons are made between different inks on

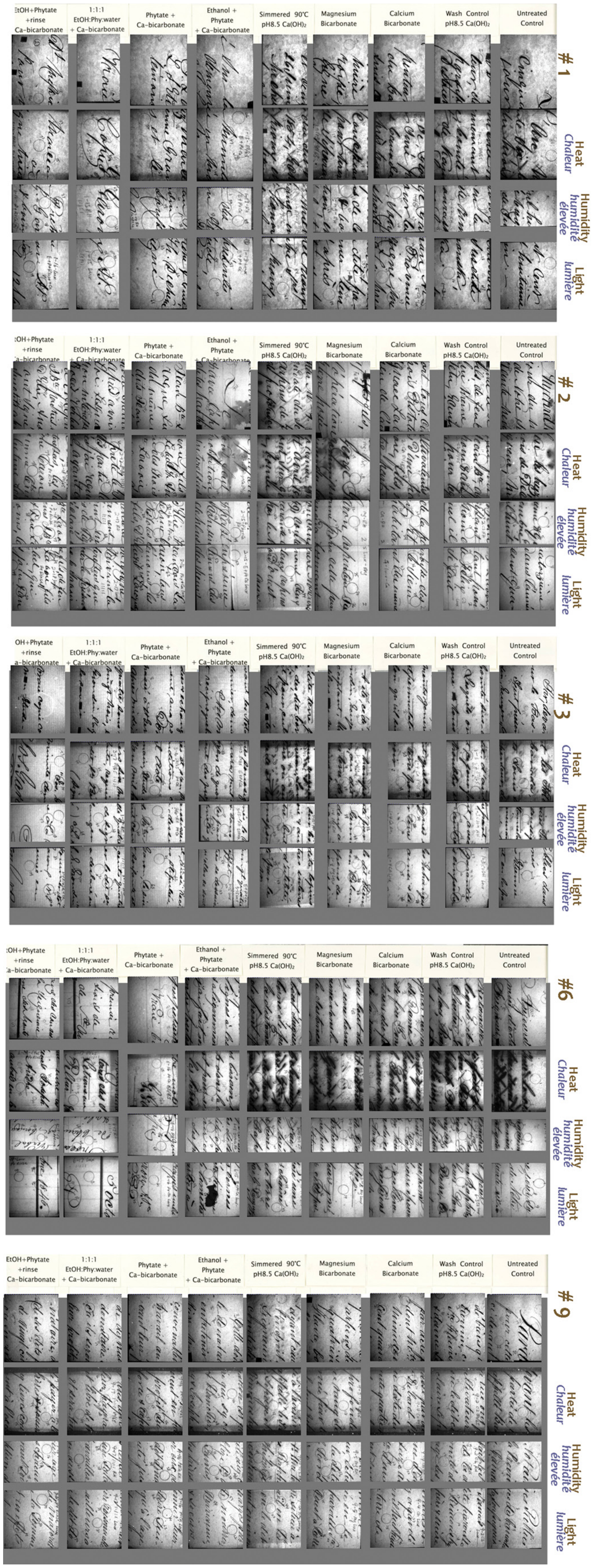

Fig. 3. Five sets of aged and treated iron-gall ink documents under UV illumination, examination at 430nm. Columns: 1) unaged controls; 2) heat; 3) humidity; and 4) light. Each row of samples represents a different treatment described in table 2

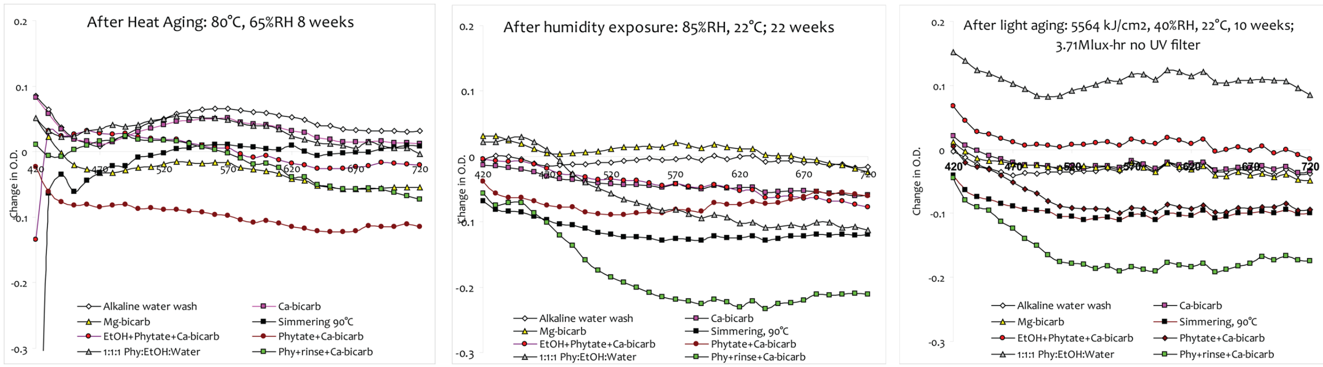

Fig. 5. Spectral differences between untreated control and treated samples after heat, humidity, and light aging for sample 6

different papers. The most reliable approach is to measure optical density of the same ink before and after a treatment.

Feasibility of visible HSI for documenting migration of ink components

The negative effects of high-humidity environments on iron-gall inks are well recognized. UV illumination has been used to visualize ink migration (Reissland 2000). In this study, visible HSI with UV illumination (365nm) was used to document and compare the impact of treatments on different inks after exposure to high relative humidity. The visible images in this report are collected at 430 nm.

Migration of ink components was clearly visible, especially with inks that have a lot of available ferrous ions (bathophenanthroline test results $[Fe^{2+}]>50+$). Of the five documents that were imaged, sample 6 has the highest $[Fe^{2+}]$[1], therefore any migration would be most visible with this set of samples. Under UV illumination, migrated components appear as shadows or halos around ink lines after water washing, deacidification, and simmering. Exposure to very high humidity (85% RH) also resulted in lateral migration of some components of this ink, presumably Fe (II) ions. An example of darkening away from a primary ink line is shown in figure 6. For the purpose of obtaining visible spectra, three regions of interest (ROI) are shown in this image. The visible spectra of each of these ROIs (paper, ink, and migrated ink) are shown in figure 7. The visible spectra obtained from the UV source show a very prominent peak λmax = 520–540 nm. This peak arises from the excitation of π bonds of the paper. Because of the high fluorescence of the paper, the spectra of the ink halos contain some of the spectral features of the paper. Compared to the minimal fluorescence in the primary ink lines, this contrast means that the UV light source provides a very sensitive method for assessing the extent of haloing of the ink. Comparing illumination using 430 and 520 nm wavelengths, 430 nm showed a higher contrast between paper and ink and is more sensitive for assessing the extent of haloing of the ink. The decreasing signal at longer wavelengths is indicative of greater transparency of the migrated ink components above 600 nm.

Initially there were some puzzling results from bathophenanthroline test for Fe^{2+} and Fe^{3+} among the untreated inks after exposure to high humidity. All the inks tested negative for both types of iron ions. Initially this was believed to be caused by iron ions being converted to a water-insoluble form such as iron oxy-hydroxide, but UV examination showed that ink components, presumably Fe (II) ions, have migrated to the surrounding areas on the paper as a result of high humidity. The most migration occurs with untreated inks that have a very high initial Fe (II) content.

All phytate treatments, with or without modification, show no migration after treatment or when exposed to high humidity. This showed that phytate is effective in complexing the Fe (II) during treatment, preventing these ions from migration. Ink with less available Fe^{2+} also showed less migration after non-phytate treatments or humidity exposure.

CONCLUSIONS

The most important advantage of using digital imaging is that spatially accurate information can be acquired. In this sense it is possible to acquire visible spectra of a thin ink line without significant contribution by the surrounding paper. In these experiments, HSI was introduced after treatment and aging; the variation naturally existing in these original samples reduced the sensitivity of the technique to quantify change, making it difficult to distinguish, for example, between strikethrough (inks from the verso) and light-color inks. Even with this limitation, HSI was shown to be useful for assessing spectral changes of inks due to treatment or aging.

For the assessment of the migration of ink or ink components, the ultraviolet light source at 365 nm worked very well, especially when compared to images acquired with a visible light source. For visible imaging with the UV source, shorter (430 nm) single-band images were more useful than longer (600 nm) single-band images. Surprisingly, statistical approaches such as Principle Component Analysis (PCA) did

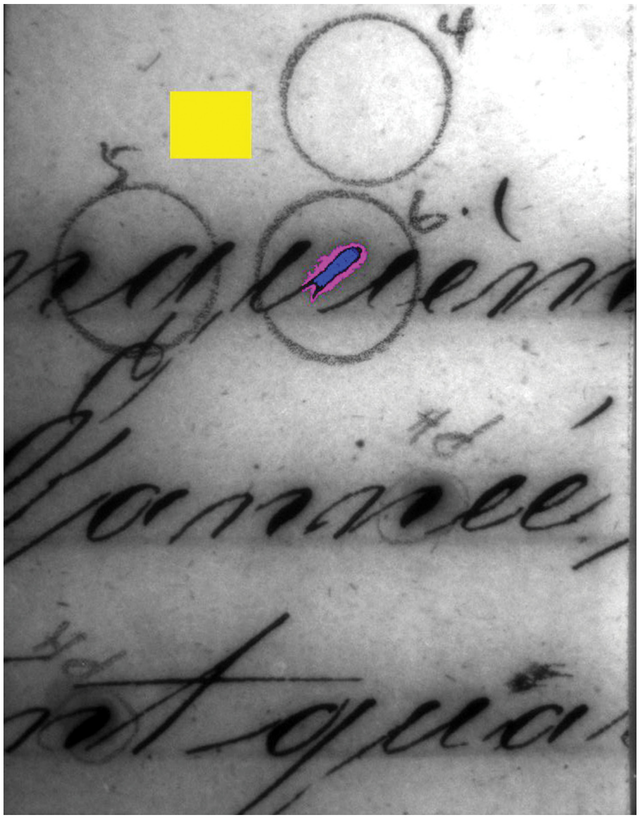

Fig. 6. An example of ink migration from a primary ink line as a result of high humidity exposure. Sample 6 untreated after humidity exposure (85% RH, 23° C; twenty-two weeks), three ROIs: yellow = paper; blue = ink; purple = migrated ink component

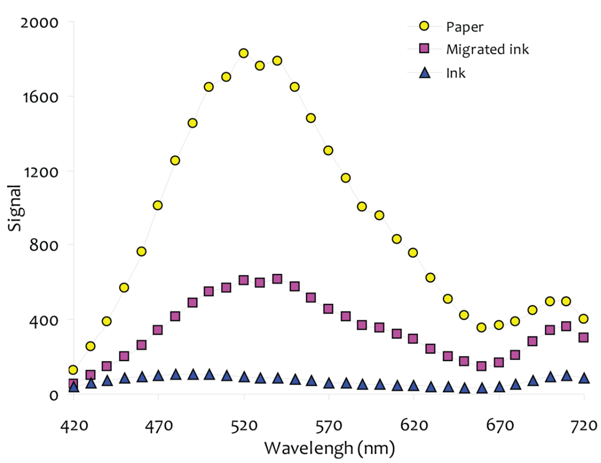

Fig. 7. Visible spectra of paper, ink, and ink migration components with UV Illumination. Sample 6 untreated after humidity exposure, three regions of interest (ROIs) corresponding to fig. 6 : yellow = paper; blue = ink; purple = migrated ink

not improve the visiblity of the migrated ink components and therefore were not used for assessing ink migration.

From a treatment evaluation viewpoint, UV examination showed that inks that have a lot of available Fe (II) ions (tested using bathophenanthroline strips) are most at risk of migration of ink components when subjected to aqueous treatments without phytate or when exposed to high-relative-humidity conditions. All ink samples treated with phytate showed much less or no ink migration compared to other non-phytate treatments. This showed that phytate is effective in complexing the Fe (II) ions, keeping the ions in the ink areas, and preventing lateral migration or "running ink." The immediate benefit of phytate treatments in preventing ink migration is less obvious with inks that do not have have high concentration of Fe (II) ions.

NOTE

1. High concentration of available Fe (II) ions can be a result of excess iron ions in the ink as well as the presence of gelatin size on the paper keeping the ink on the surface of the paper instead of penetrating into the matrix.

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YANA VAN DYKE

Sacred Leaves: The Conservation and Exhibition of Early Buddhist Manuscripts on Palm Leaves

ABSTRACT

During the summer of 2007, a collection numbering over a thousand leaves of rare and important Indian paintings on palm leaf and paper were rediscovered during a renovation and storage relocation project within The Metropolitan Museum of Art's Asian Art Department. The first exhibition, in a series of permanent collection rotations, focused on a remarkable select group of forty-five early palm leaves that cite the Buddhist Sutra of *The Perfection of Wisdom in Eight Thousand Lines (Ashtasahashrika Prajnaparamita).*

Historic evolution of the structure, the sacred ceremonial functionality and utility of the manuscript traditions, and technical background, material composition, support preparation, painting techniques, pigment analysis, condition assessment, and inherent deterioration mechanisms of both the support and media will be examined and described. Conservation treatment materials and procedures—such as structural stabilization of the aged palm leaf, including ethical considerations in compensation and reintegration of design, mounting, housing, display and storage, and topics pertaining to the care and understanding of these early Indian paintings on palm leaf—will be discussed in detail.

INTRODUCTION

Every art lover knows that many works of art contain a special inner force. It cannot be defined in the usual esthetic terms because it goes beyond the representational, symbolic, and decorative. Some civilizations refer to this force as the so-called magic content or aura. If magic is too strong, we might speak of this indefinable force as the mysteriously enticing power within the object. Let us begin by turning the leaves and allowing the knowledge and the sacred lure of these objects to be understood.

Presented at the Book and Paper Group session, AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

HISTORICAL BACKGROUND

Illustrated Buddhist manuscripts of South Asia are books that were produced in abundance throughout the eleventh to thirteenth centuries. These illustrated Buddhist books are monastic products transcribing Buddhist sutras, and nearly all of the manuscripts in existence cite the *Ashtasahashrika Prajnaparamita* or *The Perfection of Wisdom in Eight Thousand Lines* (Leidy 2008, 137), one of the most important and earliest Mahayana texts put down in writing as early as the second century B.C.E. This vast body of literature referred to as the *Prajnaparamita* is essentially a compilation of philosophical and metaphysical teachings on the nature of reality and the universe. The basic premise is a radical non-dualism, in which every and any dichotomist way of seeing things is denied, so phenomena are neither existent, nor non-existent, but are marked by *sunyata*, emptiness, and an absence of any essential unchanging nature. The sacred character of the manuscripts is revealed not only in the writing but also through beautifully illustrated representations of important deities to whom the text was dedicated and who were brought to mind while reciting the texts. The illustrations contained in the manuscripts embody the earliest surviving Indian painting in existence and date from the late Pala period (1000–1200 AD) (fig. 1).

The illustrations provide rare glimpses for historians, following the stylistic development of the paintings by comparing them to extant sculptures, with their sinuous and flowing line, as well as to rare and fragmentary paintings that survive in archeological temple and monastic complexes, such as at Ellora and Aganta. They are truly rare keystones and fundamental sources in the understanding of the development of Indian painting. From at least the tenth century, the manuscripts were beautifully illustrated, typically with an expansive pantheon of deities whose spirits were evoked through its recitation and study. Narrative themes such as the scenes from the life of the Buddha occur more rarely (narrative scenes are found more often on the book covers) and the painting style in these earliest surviving manuscripts evolved out of styles developed in Indian temples and monastic mural

Fig. 1. *Enshrined Image of Bodhisattva Maitreya. Folio from a manuscript of the Ashtasahasrika Prajnaparamita (Perfection of Wisdom)*. Photograph by Yana van Dyke (all photographs taken by author). Image copyright the Metropolitan Museum of Art (2001.445g)

painting now almost completely lost (Guy 1982, 14–17). The paintings are typically of a linear iconic style; images of deities placed alongside the text are there to increase the spiritual potency and magical power of the book by their sheer presence. The images are not necessarily illustrative, but their auspicious and protective presence alludes to their talismanic power and incites the mind to associate the images with the attributes of enlightened ones (fig. 2).

The Astasaharika Prajnaparamita, or *The Perfection of Wisdom in Eight Thousand Verses,* the most illustrated Buddhist text of the eleventh through thirteenth centuries and one of several texts explaining the development of perfect insight, states that the text itself should be an object of veneration, and valued as much as, if not more than, relics, sculptures, and other icons that also allude to the presence of Buddha.[1] The teaching in the last chapter of the *Prajnaparamita* does not mention much about the philosophical concepts of emptiness but rather emphasizes the importance in the worship of the book. The Buddha instructs:

> When through the Tathagata's sustaining power it has been well written, in very distinct letters, in a great book, one should honor, revere, adore, and worship it, with flowers, incense, scents, wreaths, unguents, aromatic powders, strips of cloth, parasols, banners, bells, flag and with rows of lamps all round, and with manifold kinds of worship. This is our admonition to you Ananda. For in this perfection of wisdom the cognition of the all-knowing will be brought to perfection (Conze 2001, 299).

The sacred power of the manuscript also lies in the fact that it is the embodiment of the Buddha, the words spoken by him. Evolving into objects of veneration in their own right in the Pala period, the cultic status of the *Prajnaparamita* (*Perfection of Wisdom*) manuscripts became so prominent that they became objects of worship, featured among the ritual offerings, evidenced in surviving pedestal sculptures of the period. Devotees of the book cult could direct their devotion to the wise goddess of the same name, *Prajnaparamita*, who represents the personification of the text; one of her principal attributes is a sacred book in hand (Kim 2007).

Fig. 2. *Green Tara Bestowing Boons*. Folio from a manuscript of the *Ashtasahasrika Prajnaparamita Perfection of Wisdom)*. Image copyright the Metropolitan Museum of Art (detail 2001.445i)

Fig. 3. *Bodhisattva Padmapani offering protection*. Folio from a manuscript of the *Ashtasahasrika Prajnaparamita (Perfection of Wisdom)*. Image copyright the Metropolitan Museum of Art (1986.25.4)

Cults of different kinds have been born out of Buddhist devotees, and the worship and veneration surrounding books of wisdom assumed and still assume an important role in the temple ritual. The public recitation and sanctification of texts, as well as the display of the manuscript itself—the physical vessel of the teaching—still form an important part of Buddhist worship. The emergence of a book-cult was certainly modeled after the *stupa*-cult (the *stupa* serving as a protective container of Buddha relics and often adorned with

flags), and in a parallel way acknowledges the physicality of the book as a physical container of the teaching that is equated with a dharma relic.[2] Goddesses such as *Prajnaparamita* and *Tara* are symbols of the texts and the advent of the Mahayana female deities, and their bearing on the book cult cannot be underestimated; the idea of a female Bodhisattvas such as *Tara* was being developed during this time (fig. 3).

The Pala Empire was a dynasty in control of the northern and eastern Indian subcontinent, mainly the Bihar and Bengal regions, from the eighth to the twelfth century. The Palas were followers of the Mahayana and Tantric schools of Buddhism. They often intermarried with the Gahadvalas of the Kannauj region. They created many temples and works of art and supported the Universities of Nalanda and Vikramashila. Their proselytism was at the origin of the establishment of Buddhism in Tibet.

Colophons often survive and document the patron who commissioned the manuscript's production, providing dates and monastic place of creation, artisans and monks who made the book and performed rituals around it, and the subsequent footsteps of its provenance, tracing later owners and donors who safeguarded the book. Following its history over hundreds of years and many generations, the book continues to be bequeathed, read and studied, proofread with scrupulous corrections of the writing (text), and repaired time and time again. Because such manuscripts were considered sacred objects and were heavily used both for teaching and as a focus of devotion or meditation, rededications and repairs were common (Leidy 2008, 136).

Small, lightweight, and easily transportable objects of veneration, they have survived through times of danger, persecution, peril, and threat.[3] Sometimes, although rarely intact in their protective wooden enclosures, the sacred leaves live to tell the tale. As is often the case of dispersed manuscript folios containing abstruse, mysterious, and impenetrable scripts from diverse cultures—now living in a museum collection or in private hands—text blocks have been discarded along the way and the images themselves taking center stage, often responsible for the survival of the singular leaves. Valued within the art museum setting for their pictorial style, they provide evidence that is reflective of the evolving philosophies of the culture from which they are born and take their place in the evolution and development of structure and style of later Indian miniature painting to follow. Twenty-first-century technology that has rapidly embraced digitization and preservation projects worldwide is enabling researchers and scholars to share previously isolated esoteric information. Transmissions from the powerful tool of the World Wide Web will facilitate the linking together of dispersed fragments, translations of previously impenetrable texts, and a host of other puzzles yet to be solved in relation to these early manuscripts.

LEAF BIOLOGY AND PREPARATION

Substances used for the reception of writing in the ancient world were numerous: wooden tablets, lead tablets, papyrus, bamboo strips, paper birch bark; whatever was most conveniently available could be used for writing (Diringer 1982, 354–62). The inherent instability of these biochemically decaying materials consequently results in an incomplete understanding of the materials used in and the volume of writing produced from the ancient world. Although it is difficult to say exactly when the palm leaf first began to be used for writing, there is ample literary evidence that in both Greece and Rome, the use of leaves for writing was quite familiar. In his *Naturalis Historia*, Gaius Plinius Secundus (23–79 AD), better known as Pliny the Elder, documents the use of palm leaves for writing materials.[4] The word "leaf" still holds its place in the descriptive lexicon of manuscripts today—as with the term "folio," from the Medieval Latin *folium*, meaning a leaf—and defines many things related to books, pages, and paper within a book, as do the terms "foliation" and "to foliate."

Critically important to understanding these manuscripts is the primary substance from which they are built. All elemental and fundamental attributes are linked to the natural structure of the palm leaf itself: the book's unique shape, form, and design layout; orientation of the script, collation and direction; size and placement of images; media interaction and adhesion; binding style; state of preservation; how they have survived; how they have aged; and the conservation challenges they present.

Arecaceae, also know as the palm, is a family of flowering plants belonging to the monocot order, *Arecales*. There are roughly 202 known genera containing an expansive 2600 species of palms. The taxonomic group of the *Corypha* is a genus of six or seven species of palms native to India, Indonesia, and the Phillipines. They are the fan palms with leaves containing long petioles terminating in a rounded fan of numerous leaflets. *Corypha umbraculifera* is one of the largest palms in the world with gigantic petioles or stalks measuring from two to five meters in diameter, with thirty to forty leaves per stem each measuring up to fifteen feet in diameter and reaching heights of 20–40 meters. It is cultivated throughout southeast Asia and north towards southern China (Wilson 2000).

Although gymnosperm, dicot, and monocot leaves are all used in a variety of artifact construction, the monocot leaves are the most versatile and heavily utilized. Reasons for this can be attributed to their greater size and durability, but more importantly to their strength, suppleness, and ease of splitting imparted by their long parallel fiber and fibrovascular bundles. From the monocot leaf, the leaf fibers comprise the vascular bundles (xylem, phloem, sclerenchyma fibers) and fiber bundles. They are often referred to as hard fibers, as distinguished from the soft fibers obtained from the inner bark

of herbaceous and woody plants. They may in fact be harder as they tend to be more heavily sclerified and lignified, but they are also mechanically stiffer. As the fibers grow in discrete fiber bundles, leaves are usually processed to extract the whole bundle, which is a compact complex of many cells. The strength, color, and abrasion resistance of monocot leaf fibers vary greatly depending on inherent characteristics and on processing methods. Generally they tend to be smooth fibers with a surface sheen that in book production is often further enhanced and beautified through burnishing. Waxes control water permeability and excessive swelling of surrounding mechanical tissues, such as fibrous sheaths around vascular bundles in palm. Pectin is another important structural component that is protected by cuticular waxes and cutin in leaves (Florian et al. 2001, 112–16).

The leaflets from these fan palms are separated from one another by a thick rib that extends from the hastula along the leaflet, creating a corrugated structure with remarkable mechanical and physical strength. Under magnification key elements of the structure become evident and, like all plant forms, they are abundantly more complex than your naked eye perceives.

Three regions are clear and distinguishable, namely the epidermis, the mesophyll, and the vascular bundles or veins. The outer membrane, known as the epidermis, covers the lower and upper sides of the leaf and protects the internal tissue from injury. Each side is composed of a single layer of closely packed rectangular cells, covered with a cuticle on the outer wall, preventing water loss. There are many stomata, formed by pairs of guard cells, perforating the lower surface (more on the lower than upper epidermis) whose function is to allow for gaseous exchange for photosynthesis and respiration as well as to regulate excess water. The vein system of the leaf consists of branched vascular bundles containing xylem and phloem and surrounded by a bundle sheath. The veins strengthen the lamina, the xylem conducts water and dissolved ions to the mesophyll tissue, and the phloem conducts organic food such as glucose from the mesophyll to other parts of the plant (Kern 1947) (fig. 4).The way in which veins are arranged is known as "venation" of the leaf and can contribute to the species identification of certain genera of palms. Manuscripts have been written on leaves from only a few varieties of palm trees: *Borassus flabellifer* Linn and *Corypha umbraculifera* Linn.[5]

Production processes differ across regions: the palm leaves can be processed by retting, soaking, and softening; boiling in water; smoking; wafting over a charcoal fire; and air, sun, or kiln drying, after which the leaves are then polished and wiped with a cloth of oil. In Indian states, leaves are 1) dried in the shade and rubbed with gingili oil to make them smooth; 2) hung in smoke for several days, then the surface smoke deposit is wiped off, sometimes followed by rubbing with turmeric paste; or 3) dried in the sun, buried in pond mud for ten to fifteen days, cleaned, dried, and rubbed with turmeric paste. In Sri Lanka, fresh, young leaves are boiled in water or lime water for a few hours and dried in the shade. In Thailand, the golden leaf from the Lopburi region is preferred. Leaves are dried in the shade, the midrib is removed, and the blades cut to uniform size. Bundles of leaves are placed in a kiln for twenty-four hours, causing a black oil to exude from the leaf edges. The oil is wiped off, the leaf is held over a fire for a few minutes, and finally polished. The removal of the oil is believed to make the leaves more durable (Agrawal 1984, 27–31).

Fig. 4. Photomicrograph (20x) of underlying mesophyll and vascular bundles structure

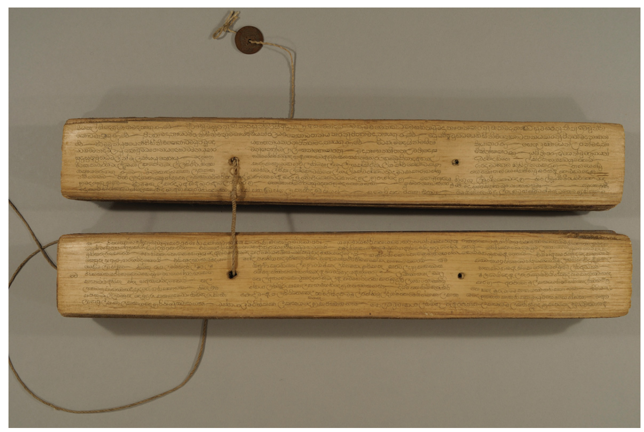

Fig. 5. Bound manuscript of palm leaves. Image copyright the Metropolitan Museum of Art (1953.123)

CONSTRUCTION OF THE MANUSCRIPT

The manuscripts are made of long and narrow leaves of various fan palms, the average size being 22 ½ by 2 ½ inches. The loose leaves are held together between two wooden cover boards, with holes being pierced either through the center, or with two at each end of the leaves and boards. A

Fig. 6. One of a pair of manuscript covers with Buddhist deities. Nepal, Karnata-Malla period, eleventh to twelfth century, distemper on wood. Image copyright the Metropolitan Museum of Art (1983.555.2)

cord or cords are passed through these holes and wrapped around the bundle.[6] The leaves are read from left to right, top to bottom, and then lifted, turned, and stacked face down one leaf over another (fig. 5). Such openness in the format of the binding structure has certainly made it easier to disperse the text block without initially causing physical damage to singular leaves, although this leads to textblocks found mixed and out of order, making it especially challenging to analyze, contextualize, and collate them.

Additionally, this practice has invariably led to covers being separated from their textblocks and there are many examples of detached and decorated wooden cover boards in the Metropolitan's collection (fig. 6). The manuscripts' cover boards are usually of wood, with finishes that range from simply stained and waxed to elaborately composed narrative painting programs that are highly polished and lacquered. Closed bundles were traditionally wrapped in several layers of cloth and stored in chests. Palm leaves may also be joined by tying at the edges so that they can be folded and unfolded in accordion fashion.

CALLIGRAPHY

Palm leaves have traditionally been written upon in two ways: either the text has been incised into the surface or written on the surface. However, there are no examples of inscribed leaves in the group of early Buddhist manuscripts that were selected for the exhibition.[7] The other technique of writing on the surface is with a pen, most likely made of reed, utilizing an ink composed of flame carbon with a binder. The smooth, slick, oiled, flat, and dense epidermal layer allows for the reed pen to flow smoothly and distribute the ink fluidly and uniformly on its surface. Unlike other organic writing supports, this ink does not penetrate, embed, or stain a fibrous matrix as in birch bark, paper, or parchment. In general, the writing ink is chemically stable and only inherently susceptible to microscopic instability or losses due to mechanical abrasion in the stack. This also holds true for the paint layers, as the combination of the processed, oiled surface and natural cuticular waxes act as a protective barrier on the surface of the epidermis, inhibiting the penetration and

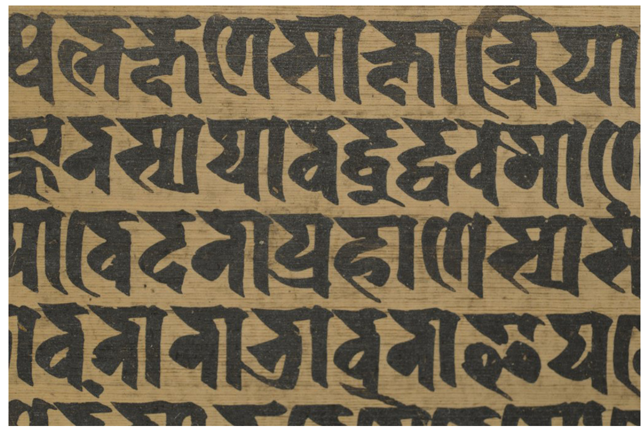

Fig. 7. Detail of calligraphy

saturation or staining by the media, held on by cohesive and chemical forces at the surface.

The text of the Buddhist manuscripts is beautifully written by skillful scribes without any separation between words in a careful and consistent hand; a single folio seems like an overwhelming ocean of letters (fig. 7). Here and there punctuation marks break the *scriptura continua* (writing without separation) with a narrow strip or band of empty space around the binding holes functioning like aeration, not necessarily breaking the sentences into meaningful units, but nonetheless giving visual breaks between many clustered letters. The paintings are usually found centered on the leaf and are placed at the end of each chapter. The paintings also mark points of rest in between the chapters so that during the reading and reciting of the text, they also serve as spiritual sign posts by which the essence of the book could be remembered and recalled (Kim 2007, 315–16).

The calligraphy within these Buddhist manuscripts is Sanskrit, a historical Indo-Aryan language, one of the liturgical languages of Hinduism and Buddhism, and one of the twenty-two official languages of India. Classical Sanskrit is the standard register as laid out in the grammar of Panini, around the fourth century. Its position in the cultures of South and Southeast Asia is akin to that of Latin and Greek in Europe and it has significantly influenced modern languages of Nepal and India. The writing is read left to right and top to bottom.[8]

PAINTING

In all examples, the images are built with thickly applied opaque paint applied directly to the palm leaf with no preparation layer or ground. Examination with infrared reflectography revealed no evidence of any underdrawings, although in some cases, examination through the binocular microscope revealed a simple, linear outline underdrawing in pale red or dilute black. Adhesion of the paint layer to the leaf is usually strong and at first glance paintings appear flat and the limited palette is deceiving. On closer inspection, quite a bit of volumetric gradation exists (fig. 8). Forms are created by the application of a broad, overall, base color and built up in stages with an economic linear outline of less thickly applied mixtures.

Illustrations from three different texts were selected for pigment identification and were categorically identified. All pigments within the three selected manuscripts were found to be consistent, as well as customary or expected for this time period. The limited use of exclusively pure pigments, as well as a combination of these pigments in mixtures, varied to some degree from manuscript to manuscript: some exploiting the possibilities of expanding the palette, gradually building up volumetric forms.[9] Blacks were identified as carbon-based, flame carbon; blue was indigo; yellow was orpiment; red was vermilion; and white was calcium carbonate. Admixtures are prevalent throughout; use indigo and orpiment for green; and white is added to many of the pigments for varying shades. Sinuous and graceful, modeled figurative forms are extremely reflective of Pala sculptures and honor their ancestry of the humanistic naturalistic painting styles of the past.[10]

CONDITION AND CONSERVATION

In general, all of the palm leaf supports were very dry and particularly vulnerable to damage around the edges. Fraying, delamination, and splitting of the structure are to be expected around the binding holes and from the edges; tears are much less common. Losses are primarily located around the perimeter of the leaves and planar distortions vary from none to moderately wavy. Separation from their protective covers and exposure to less-than-ideal climatic conditions have certainly contributed to their compromised conditions (fig. 9).

Overall, a conservative conservation strategy addressed the essential and fundamental physical stabilization needs of the leaves, making the objects safe to handle and display while respecting their age and signs of use. Utilizing natural products with good aging properties whenever possible, considerations included how the treatments would ultimately affect the structural, chemical, and mechanical characteristics and long-term stability of the object. Comparatively straightforward conservation procedures were carried out—the difference in this case being the extraordinary level of delicacy

Fig. 8. Detail of volumetric shading. Image copyright the Metropolitan Museum of Art (1986.25.5)

Fig. 9. Before treatment group of desiccated and deformed palm leaves

Fig. 10. Photomicrograph (20x) showing losses within flaking paint layer

involved, especially when treating the support problems. The object's before and after slides will not offer any great dramatic visual response and they do not necessarily present challenges that require new inventions in materials or techniques, but rather adaptations of standard practices with greater necessity of delicacy and sensitivity. These conservation challenges demand patience, and patience on a level that seemed appropriate with regard to their dharmic contents.

MEDIA PROBLEMS

Unstable media in the form of flaking and, less often, friable paint layers, existed in all in the palm leaf illustrations (fig. 10). This instability can be attributed to poor adhesion to the waxy cuticle, desiccation of the binder upon aging, and abrasion and stress caused to the paint layer as the leaves of the book are turned. Aims and considerations in consolidating unstable media are understood: to secure, stabilize, and ensure that no future losses take place without altering the visual character of the paint layers. Desired functional working properties are also important and include strength, flexibility, aging properties, effect on the original, viscosity, surface tension, penetration, and elasticity once applied.

Empirical testing on the manuscript leaves with consolidants and adhesives for either media consolidation or for use of consolidating and adhering delaminating palm leaf structures included: laboratory-grade gelatin at concentrations of 1% and 2%; Dow methyl cellulose 4C; Dow methyl cellulose 4M in 1%, 2%, and 3% concentrations for consolidation and in 5%, 6%, 7% for adhesion; and isinglass at approximately 1%, 2%, and 3% weight/volume for paint film consolidation. Gelatin and methyl celluloses in concentrations that were successful at adhesive bonding proved unacceptable, visually creating tidelines and a glossy surface sheen. Methyl cellulose concentrations at or above 5% have an unfortunate side-effect of leaving crystalline residues that appear as sparkles in reflective light.

Consolidation of the paint layer was carried out with a warm solution of 1% brush-applied isinglass while working under the magnification of a binocular microscope (fig. 11). No visual alteration of any pigments occurred (including orpiment and admixtures thereof).

Fig. 11. Consolidating the paint layer with the aid of the binocular microscope

Fig. 12. Isinglass in various forms: (clockwise from upper left) desiccated raw bladder, in solution (*bain-marie*), and dehydrated purified disks

ISINGLASS: CONSOLIDANT FOR PAINT AND PALM

Empirical testing found that the best consolidant for both the media and the palm leaf support was isinglass. It met all of the criteria discussed for consolidating media and proved effective in the self-mending of delaminating and fraying leaf structures, as well as attaching reinforcement paper in mending breaks and splits. For a complete discussion of this material, consult the article by Tatyana Petukhova and Stephen D. Bonadies, "Sturgeon Glue for Painting Consolidation in Russia" (1993). Additionally, we have been practicing a useful technique for several years of depositing freshly made isinglass into disks or wafers and allowing them to dehydrate for preservation and storage. In turn, these dehydrated disks of isinglass can easily be brought back into solution as needed, simply reducing the laborious, two-day process of cooking and extracting to once or twice a year (fig. 12).

To make isinglass, follow the basic instructions and techniques provided in Petukhova and Bonadies' article. Specifically, the following guidelines provide the approximate weight of the dried Salianski sturgeon bladder membrane and volume of water to make a large quantity of isinglass (1 L)

that will ultimately be cast into disks, preserved, and stored for future use; one can certainly adjust the weight/volume ratio according to need.[11] We normally start the procedure with thirty grams of dried sturgeon bladder, and end up with one liter, or 1000 ml, of extracted collagen liquor to be cast. In practical terms we have found it easier to divide the operation into three portions, especially for working ease of the double boiler system as well as beaker sizes. Additionally, when it is time to cast the liquid into the disks, three people help to distribute all the liquid onto the silicone-release Mylar sheets, making the whole process faster. Break the thirty grams of dried bladder into pieces no bigger than three to five grams each and place approximately ten grams of the dried bladder pieces into three separate beakers and cover with just enough deionized water to top off the bladder. Cover the beaker with a watch glass or Parafilm and let it soak overnight.

Next, pour off the water and gently gather together the swollen bladder mass into an even cluster and add 333.5 ml of deionized water; this will cover the bladder enough to surround and allow the pieces to float. Put this beaker into a larger container of water and make a *bain-marie* or double boiler. Heat the double boiler on low temperature, just enough to release the collagen extract from the fiber of the bladder. Be mindful of the heat; there should be no steam or bubbles released as this will in part denature the protein.[12] The fiber of the bladder will shrink as it releases the collagen. To keep the water from evaporating off, cover with a watch glass. After a few hours, the collagen will be extracted from the fibrous membrane of the bladder. Sieve or decant through cheesecloth (doubled or tripled) or silk. Decant into a clean beaker.

Find a safe and undisturbed area to deposit the liquid into disks; this process will require a fair amount of space in a dust-free environment with enough air to flow and circulate around them during drying. Place several medium-sized sheets of silicone-release Mylar on a flat surface where you intend to carry out dispensing of the disks (fig. 13). Disposable pipettes or eye droppers have been found to be the best tools for distributing the liquid. Deposit and form a circular puddle of the liquid approximately one and a half inches in diameter onto the silicone-release Mylar, spaced out with enough room in between so the disks don't run together. Finding solutions to protect the disks while they dry can be a little tricky to avoid disturbing the liquid as it dries. We make tents of blotters, folded in half so that they span the width of the Mylar, and taped or weighted in place onto the counters to protect the disks as they dry. Once dry the dehydrated disks can be peeled off the Mylar and kept stored in a glass jar.

Dehydrated isinglass disks can now be weighed out and dissolved to make precise concentrations of solutions in small quantities for specific purposes. Casting the disks has proven to be an extremely valuable and time-saving procedure, as the process of extracting the adhesive from the membrane is rather laborious and this step limits that production to once or twice a year. Because of the increasing difficulty and cost of obtaining Russian sturgeon bladder, the flexibility and ease of making smaller quantities are also conservatively advantageous. Isinglass can be refrigerated to postpone mold or bacterial growth and subsequently warmed back into solution in a *bain-marie*.

Fig. 13. Disposal pipettes dispensing the isinglass liquid into disk shapes to be dehydrated and stored for future use

SUPPORT PROBLEMS AND REHYDRATION

Similar to the demands of papyrus, the aged palm leaf can be very desiccated and brittle, especially around the edges that have been most exposed to oxidation processes. Rehydration of the leaves is considered one of the most important first steps in addressing support problems. Eight-hundred to one-thousand years of hydrolysis and oxidation deterioration mechanisms result in an extremely dehydrated, inflexible, and brittle material with considerable planar distortions. Humidification and rehydration of these desiccated leaves with the use of a Gore-Tex humidification package has many benefits: reducing distortions, relaxing creases, achieving original planarity, increasing suppleness to the leaf, and maintaining planarity between the support and the slightly sensitive media layer while the reintroduction of moisture content takes place. Another advantage of this slow, gentle, and effective vapor humidification procedure is the significant reduction of the all-too-present risk of causing stains and tidelines with direct applications of water or mist. Folds and flaps existed in the corners of many leaves. These folds and creases were relaxed and opened by introducing a localized fine ultrasonic mist humidification, avoiding breakage of any tissue before overall humidification.

The Gore-Tex barrier is a micro-porous membrane of polytetrafluoroethylene laminated to non-woven polyester or polyester felt (Purinton and Filter 1992). Gore-Tex

has been used for some time in the conservation profession and it has become increasingly difficult, if not impossible, to purchase. The membrane transmits water in the form of vapor while preventing the passage of liquid water, thus giving extra protection and control in introducing moisture to sensitive objects. When Gore-Tex is used in the blotter sandwich, two sheets of Gore-Tex are placed dry, one on either side of the object. The smooth membrane side of the Gore-Tex sheet is thus in contact with the object's recto and verso. Moist sheets of blotting paper rest on either side of the felted side of the Gore-Tex, and Mylar sheets are placed on top of the damp blotters to retain moisture levels. Humidification times varied between twenty to forty minutes for complete relaxation of the leaves, after which they were placed into a blotter drying pack between tissue and shuffled over several weeks while drying.[13]

STABILIZATION OF SUPPORT

After the leaves have undergone humidification and drying, they are rehydrated, more supple, flexible, and planar; structural stabilization can now be undertaken with considerably less risk of breakage. Additionally, the humidification process can often lead to improved alignment of sprung tears as well as to invigoration of the natural binding materials within the leaf itself, resulting in an adhesion and reattachment of slightly delaminated areas.

Mending methods chosen should be those least likely to cause permanent alteration of the basic fabric of the object. The choice of method is more immediately dictated by the work of art itself, location of the damage, and condition of the materials. Mending methods should be chosen for the item's intended use, future handling and storage, and compatibility in strength with the work of art so that they are strong enough to give support and physical integrity, yet weak enough to yield under stress before the work of art does. Repairs should be visually subtle yet discernible upon examination and differ from original or historic repairs that cause no harm.

Securing loose and delaminated layers of the leaf requires not only the utmost patience and delicacy but also a fluid form of adhesive that can be delicately applied. A slightly stronger solution of isinglass (approximately 3–4%) was chosen from that which was used to consolidate the media. When consolidating the palm leaf itself, the consolidant liquid needs to penetrate into the tiny crevices and pores in order to make a satisfactory adhesive bond. Increased flow is achieved by keeping the liquid warmed on a coffee cup warmer in a water bath (*bain-marie*) (fig. 14). While working under 10x magnification from the binocular microscope, careful attention was paid to the interior spongy core of the leaf as the brush-applied consolidant was observed to flow and coat the mesophyll and vascular-bundled veins through capillary action, without causing them to expand, swell, or shrink to any discernible degree.

Fig. 14. Consolidation of leaf with 2–3% isinglass while working under binocular magnification

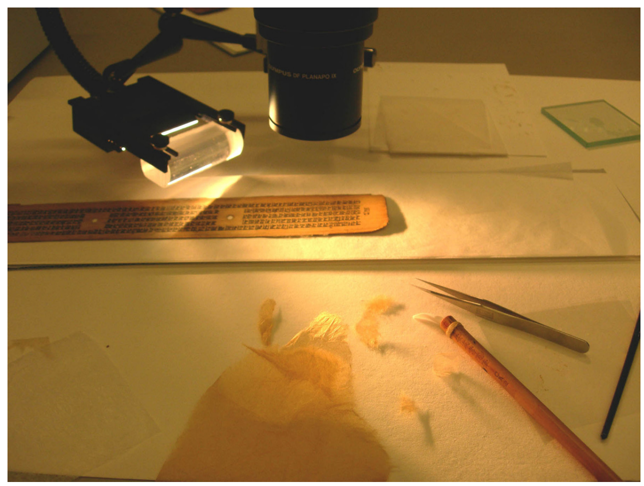

Fig. 15. Toned *tengujo* reinforcement

As in most instances while consolidating, the conservator can make empirical judgments as to the volume of consolidant that is sufficient and necessary by observing and being sensitive to the object itself.[14]

Edges of thick, porous palm leaves are often crushed and frayed, and lead to horizontal breaks along the leaflet. Usually these breaks are too weak or sprung to make a strong, self-mended joint. Therefore it is not only desirable to consolidate and strengthen these ends and breaks but also to provide additional mechanical security through reinforcement strips. A small selection of Japanese handmade *tenjugo* papers were toned with air-brushed acrylic paint in a variety of tones and employed judiciously for reinforced repairs, attached with a 2–3% isinglass (fig. 15).[15] In most cases, leaves being exhibited had a discrete recto and verso, with paintings present on one side only, thus repairs were limited to the verso side of the leaf. The tonality and the thinness of the tenjugo made them visually harmonious and they cannot be readily detected with the naked eye.

QUESTIONING INTERVENTION

Knowing the composition and properties of all the materials used in the creation of the manuscripts is essential to understanding their present physical condition and needs, as well as help to predict how the objects will respond to conservation treatment and the museum environment. Correspondingly important is to be sensitive to the object's historic and cultural context in order to acknowledge the intangible properties with which it may have been invested, as well as to recognize alterations that may have taken place through its use and function.

Ethical considerations and principles of conserving sacred objects meshed with curatorial attitudes in regards to loss compensation—all agreed to an approach not to fill losses. From monastic creation to museum display, with few exceptions, most of the leaves are no longer part of a complete manuscript and are appreciated for their visual potency and are safely mounted for two-dimensional display. Objects chosen for the exhibition did not display egregious losses, but even so, all agreed to accept the object with its losses, respect it for its age and wear, and to treat the leaves with physical stabilization as the primary concern. Considerations also included their end use and how infrequently these objects will be handled or originals requested for study. In addition, all the leaves have been captured and digitized in high-resolution image files. In those cases where the loss would promote or exacerbate a further injury, the thin, toned, Japanese paper was left to fill the void and bridge the break (Nichols 2004).

Fraying, especially around the perimeter and binding holes, was minimally addressed to prevent losses but not consolidated to the degree of aligning fractured internal structures. It was respected as the evidence of use damage caused by the binding cords around the holes.

Fig. 16. Micro-surgical tool used to remove accretions

Surface accretions of smoke, dirt, and oils are deposited and absorbed onto the surface of most of these leaves from use and handling, turning, and reading. In addition, the item showed signs of the aforementioned veneration within religious rituals: being anointed, buried, sprinkled with unguents and candles, and incense burned around them. Most surface accretions were left alone and respected as evidence of their use and history. In very few cases, where the surface accretions were particularly disturbing to the overall reading of a deity or the foreign matter was identified as insect deposits, the accretions were removed mechanically under binocular magnification with especially fine surgical tools (fig. 16). In some instances, attachments and accretions covering over images were fragments of offset leaves and these were also delicately removed in order to reveal the painting underneath.

IMAGING AND CATALOGING

The entire collection of palm leaves recently discovered, except a large Indonesian bound book, have been captured with a scan-back camera called the Betterlight on a copy stand set-up. The lights are LED lights made by Scott Geffert, our consultant from CDI (Center for Digital Imaging), for the stand. The images were captured at 600 dpi, resolution set at 100%, with a color profile of ProPhoto RGB. These images are now incorporated into the Metropolitan Museum's Media Bin, the museum's image database, which will eventually be made accessible in the public domain via the World Wide Web. Following the digital image capture, the objects were catalogued into the Museum System (TMS) for museum staff access.

MOUNTING AND DISPLAY

Art museums have a long tradition of displaying dispersed folios from manuscripts out of context in a two-dimensional format. Hinged and matted leaves and bifolios displayed in frames are more common than presenting detached leaves in a book cradle. Valued in the museum setting for their artistic expression and far removed from their original function, these objects required a systematic and thoughtful approach to hinging and mounting.

The uniquely long horizontal format of the leaves creates challenges for exhibition and two-dimensional design display. Although there would have been enough room to display three pages together within the standard-sized mat and frames chosen, the curators believed it best for the viewer's experience to limit the number to two leaves and, thus, two windows per mat. Observing reactions of the leaves after removing them from the drying presses and sensing that with any minor to moderate fluctuations in humidity that these objects will respond as sensitively as parchment, deep-mat windows were considered appropriate for exhibition and safe

housing. The mat board window was composed of a double-step, four-ply window followed by an occasional four-ply lift, for an average total of twelve plies of mat board per window.

Rather than hinge the chosen pairs of palm leaves directly to the four-ply back board of the mat package, leaves were hinged individually to a two-ply museum board (fig. 17). This approach performs several functions: it allows for optimal flexibility in exhibition (arrangements or juxtapositions can be changed in future rotations if desired) and minimizes the need for handling, cutting hinges, removal from the mat, and reattaching new hinges

Small rectangles measuring approximately 5.0 x 3.0 cm of light-weight *mino-gami* paper (Hiromi HP 04) were attached around the perimeter of each leaf. Because of the potential staining concerns with the wheat starch paste (perhaps due to its large globule size proportionate to the fully dissolved fluid form of isinglass), isinglass was chosen to attach the Japanese *mino-gami* paper hinges to the palm leaf. Following hinge attachment the areas were immediately placed under polyester webbing and blotters, under glass squares and lightly weighted until thoroughly dried. Once hinges were attached to the palm leaves, they were in turn V-hinged to a two-ply museum board (Rising Natural 2-ply) with a 2.5cm border all around. When attaching the palm leaf to the secondary, two-ply board, wheat starch paste was used in the typical manner, and in the same method described above except for the additional piece of polyester webbing placed between the hinged object and the secondary support board. Once the hinges were completely dry, the small pieces of polyester were removed with a pair of very fine tweezers. Objects on their two-ply supports were attached into their museum exhibition mats with a pressure-sensitive linen tape (Filmoplast SH). The mats also serve as permanent housings.

Fig. 17. Preparing leaves for exhibition: hinging to two-ply board

Fig. 18. *Early Buddhist Manuscripts: the Palm Leaf Tradition*. Gallery installation view. Image copyright the Metropolitan Museum of Art

EXHIBITION

Following common museum practice, these leaves, like so many other detached and dispersed manuscript folios, were mounted and presented in frames with glazing and hung on the walls of a small, rotating-display gallery devoted to works on paper in the South Asia wing of the museum (fig. 18). They were presented in the low and atmospheric lighting of 3–5 footcandles (30–50 lux). Simple, profiled wooden frames, stained with a walnut color, were outfitted with Optium glazing (a non-glare, anti-static, UV filtering, Plexiglas sheeting), all contributing to a beautifully clean presentation overall.

STORAGE

The folios chosen for exhibition and display were matted in one of a limited number of standard housing and storage sizes: 19 by 14 inches, 20 by 16 inches, and 22 by 28 inches. Once the exhibition rotation came to an end, the objects were taken off view, unframed, interleaved, and stored in a standard-size Solander boxes in the Department of Asian Art's storage facility. There are numerous groups of leaves that are fragments of what was once a complete manuscript not recently or yet chosen for exhibition or display. Inspired by the simple approach to covers that the creators of the manuscripts took, we chose a similar simple solution for their storage and housing within the museum. These dispersed folios are kept together as discrete bundles and are more often than not missing their covers. Folios that have not yet been chosen for exhibition and display are kept together in accessioned numbered sets, stacked and housed between two rectangular boards of acid-free corrugated board with identification labels attached to the top board, wrapped, and tied with cotton twill cordage (fig. 19).

When working out these housing solutions it became clear why for so many reasons two simple boards, tied around the

Fig. 19. Housing and storage solutions

leaves, was ultimately the best approach of protection. Edges, especially the right and left, are particularly susceptible to fraying. Because of their brittle and vulnerable nature, if the leaves are stored in a four-walled or even a drop-spine box, the edges will be abraded, stressed, and potentially at risk for further damage. The damage potential is further increased with challenging access and handling issues. The corrugated boards are cut with a slight overhang on all sides, allowing and accommodating for even slight shifting or uneven stacking, maintaining a force of even pressure from the top and bottom and protecting the textblock leaves from undulations by restricting their movement to slight changes even within the museum environment.

CONCLUSION

The previously described case study only begins to scratch the surface of conserving the rare and important collection of early Buddhist manuscripts in the Metropolitan Museum of Art's collection. Future conservation treatments will continue to be steadfastly approached with unwavering respect for the objects, understood for their sacred and powerful nature and conserved with patience and techniques that do no harm. Stronger and healthier, the *Prajnaparamita*, the Bodhisattvas, and the Buddhas all live to tell their powerful tale, enlightening us now and for future generations.

ACKNOWLEDGMENTS

Sincere thanks are due to many people who were helpful with this project: John Guy, Marjorie Shelley, Kurt Behredt, Lyndsey Tyne, Rona Chang, Akiko Yamazaki Kleps, Martin Bansbach, Rebecca Capua, and Stephanie Porto.

NOTES

1. In his seminal essay, *Notes on the Cult of the Book in Mahayana, Indo-Iranian Journal,* Gregory Schopen discusses the emergence of a book-cult modeled after the *stupa*-cult (the *stupa* container of relics), acknowledging the physicality of the book as a physical container of the teaching that is equated with a dharma relic. Goddesses (female Bodhisattvas such as *Tara*) are symbols of the texts and the advent of the Mahayana female deities and their bearing on the book cult were being developed during this time. Buddhist female deities were created in response to the competition with other religious systems such as Brahmanism and other esoteric cult practices (Conze, *Remarks on a Pala manuscript*, 5–12).

2. Buddhist female deities were created in response to the competition with other religious systems such as Brahmanism. For a discussion of the book cult, see Schopen, *Figments and fragments,* 147–181, and Kim, *Unorthodox Practice,* chapter 5.

3. The defeat of the Pala dynasty in the mid-twelfth century by neighboring Sena rulers, and in turn the rapid collapse of the kingdom to Muslim Islamic forces, collapsed the monastic establishments responsible for the production of Buddhist manuscripts. Combination of aggressive Islamic campaigns coupled with expanding populist embrace of Hinduism broke the Buddhist hold on Eastern India, the last stronghold; monks sought refuge in Nepal and Tibet and took their portable sacred texts with them as well as other small relics and accoutrements. The eleventh-century invasions by Islamic Muslims into India is beyond the scope of this paper, but see Paul, *The Early History of Bengal,* 33, for an historical account.

4. Diodorus Siculus relates that judges in ancient times in Syracuse were accustomed to writing exile sentences on the leaves of olive trees (Pliny, *Natural History,* 69).

5. For an in-depth investigation into the types and identification of various palm leaves used in the manufacture of these types of manuscripts see Freeman, *Turning over old leaves*, 99–102.

6. Fibers were collected from a fragment of binding cord clinging to one of the binding holes of a single leaf, TMMA object 1986.25.4. The sample was dampened with water on a microscope slide to tease apart the fibers and then was examined with a stereobinocular microscope to evaluate the general composition of the fiber. Examined both dampened and after drying and staining with Graff C-stain, the sample was analyzed under 100–400 times magnification and determined to consist of bast fibers, resembling hemp (Graff, *Color atlas*).

7. In Burma and Sinhalese, scribes would employ an iron stylus or some sharp-pointed instrument to scratch the letters. The nature of the material compelled them to avoid long, straight lines because any scratch along the longitudinal fiber (running from stalk to point) would split the leaf. Perhaps this characteristic treatment of writing on this particular plant can be in part responsible for the rise or development of the distinct curvilinear shapes in the Burmese rounded hand script.

In order to make the writing or decoration clear and legible in the inscribed leaf, ink prepared from lampblack and oil was rubbed over the surface of the leaf, filling up the finely incised lines to make the writing visible and relatively permanent. Cloth pads were used to smear on

and wipe off the black flame carbon soot mixed with wood oil that fills in the inscribed characters. Fine sand or rice bran is cited to have been used to assist in wiping off the soot and resin from the top surface without disrupting the ink now sitting in the troughs of incised lines.

8. The preclassical form of Sanskrit is known as Vedic Sanskrit, with the language of the Rigveda being the oldest and most archaic stage preserved. Its oldest core dates back to as early as 1500 BCE, qualifying Rigvedic Sanskrit as one of the oldest attestations of any Indo-Iranian language, and one of the earliest attested members of the Indo-European language family (Maurer, *Sanskrit language*).

9. Micro-samples were acquired and analyzed through polarizing light microscopy (PLM) as well as through non-destructive x-ray fluorescence (XRF). XRF spectroscopy was carried out by the author using a Bruker ARTAX open-beam ED-XRF unit, at a kVof 50 and 500 μA through air, using no filter, with a live time of 100 seconds (Eastaugh et al., *Dictionary and optical microscopy of historic pigments* and *Dictionary of historic pigments*).

10. On the changing nature of the painting styles of India, see Guy, *Palm leaf and paper*, 14–17, and Bhattacharyya, *Notes on the techniques*. Filling in the color is said to make the picture blossom. The painting is compared to an opening flower as the image first burgeons forth as color is being applied to it; and in the likeness of the blossoming painting is also the moment of both shown and not shown, related to the paradoxical manifested state of things as they are in themselves.

11. The Salianski Kremer isinglass glue is made from the fresh air bladder of the sturgeon (species of origin: *Acipenseridae*). It does not include any equine, ruminant, swine, or avian species or their byproducts, and is of German origin. It is a byproduct of sturgeon farming for caviar production. The production, trade, and use of isinglass is not regulated by CITES.

12. Denaturation of proteins involves the disruption and possible destruction of both the secondary and tertiary structures. Since denaturation reactions are not strong enough to break the peptide bonds, the primary structure (sequence of amino acids) remains intact. Denaturation disrupts the normal alpha-helix and beta sheets in a protein and uncoils them into a random shape. Four types of bonding interactions between side chains are possible in tertiary structures: hydrogen bonding, salt bridges, disulfide bonds, and non-polar hydrophobic interactions. The most common observation in the denaturation process is the precipitation or coagulation of the protein. Two procedures in the preparation and use of protein-based consolidants that a conservator should be mindful of are: heat, which can disrupt hydrogen bonds and non-polar hydrophobic interactions; and alcohol, which will disrupt hydrogen bonding between amide groups in the secondary structure and side chains in the tertiary structure, increasing the risk of darkening upon aging. Therefore, use the lowest amount of heat necessary when dissolving solid proteins into solutions, and if alcohol is needed to break surface tensions, apply it before and always separately from the protein being employed in the procedure.

13. Alternative humidification techniques to this procedure could be pursued with a humidification chamber prepared in plastic photo trays with sheets of moistened capillary pads, placed underneath plastic egg crating onto which the object is placed on a piece of smooth polyester web and covered with a sheet of Plexiglas. This seems like an acceptable means of humidification, but the most suitable is the Gore-Tex package as it keeps the substrate and the media layers from flexing and distorting too much. Finding the balance in the delicate treatment of vulnerable and sensitive objects often requires creativity, especially when dealing with limited resources and materials.

14. Judgments will also be formed from perspectives of timing, when to introduce areas to localized drying and pressing, reassessment, and evaluating the number of campaigns of consolidation necessary to achieve stability without inducing stresses or harm to the original.

15. HM-1 Tengucho from Hiromi Paper Inc is *tengucho* of the finest quality, the kozo fibers cooked with wood ash making the paper optimal for conservation use.

MATERIALS

Salianski Kremer Isinglass Glue
(Product Number 63110)
Kremer Pigments
247 West 29th Street
New York, NY 10001
http://www.kremerpigments.com

Tengucho Paper
(Product number HM-1)
Hiromi Paper Inc.
2525 Michigan Ave, Unit G-9
Santa Monica, CA 90404
http://www.hiromipaper.com

Surgical tools
Roboz Surgical Instrument Company, Inc.
PO Box 10710
Gaithersburg, MD 20898–0710

Fine Science Tools (USA) Inc.
373-G Vintage Park Drive
Foster City, CA 94404–1139
finescience.com

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Archives Conservation Discussion Group 2009: Conservation and Preservation Approaches for Stabilizing Large-Scale Collections

ABSTRACT

The Archives Conservation Discussion Group 2009 focused on conservation and preservation approaches for stabilizing large-scale collections. When confronted with large-scale collections, the conservator must devise a plan that can be executed in the most efficient manner, making the best use of staff time and resources. Often large-scale treatments must be carried out in a timely fashion, such as in response to an influx of mold or when a particular collection is in high demand. Conservators may adapt a triage approach to the treatment of these materials to save on time and materials. Five conservators were invited to present four examples of large-scale efforts as applied to books, photographs, and paper-based materials. Presentations and a subsequent discussion included the use of adhesive-coated repair materials, large-scale mold remediation, the assessment and stabilization of a large collection of archival scrapbooks, and lessons learned from large-scale photograph preservation projects.

INTRODUCTION

Complementary to the Library Collections Conservation Discussion Group (LCCDG) theme, "LCCDG 2.0—New Directions," which highlighted advances in single-item treatment techniques, the Archives Conservation Discussion Group (ACDG) looked at treatment advances applied more broadly to large-scale collections. Conservators working with archival materials are familiar with collections that exist on a massive scale. When confronted with large-scale collections, the conservator must devise a plan that can be executed in the most efficient manner, making the best use of staff time and resources. Often large-scale treatments must be carried out in a timely fashion, such as in response to an influx of mold or when a particular collection is in high demand. Conservators may adapt a triage approach to the treatment of these materials to save on time and materials. The ACDG co-chairs invited a group of conservators to prepare presentations on a wide range of large-scale efforts as applied to books, photographs, and paper-based materials. The 2009 AIC Book and Paper Group ACDG met in Los Angeles on Thursday, May 21, to share and discuss these efforts, which included the use of adhesive-coated repair materials, large scale mold remediation, the assessment and stabilization of a large collection of archival scrapbooks, and lessons learned from large scale photograph preservation projects. In addition to their presentations, participants provided handouts with further information and resources and ended with a question and answer session. A summary of each presentation and the resulting discussion is provided below.

PRISCILLA ANDERSON AND SARAH REIDELL

PRE-COATED REPAIRS PART 2: PREPARATION AND APPLICATION

Adding to Priscilla Anderson's discussion of the pros and cons of pre-coated repair materials during the LCCDG session, Sarah Reidell focused on preparation and application during the ACDG session. Using her own experiences as well as information collected through a survey of conservation colleagues, Reidell presented the main categories of pre-coated materials, which include those reactivated with water, solvents, and heat. Reidell began her presentation with a discussion of incorporating the use of purchased, ready-made, heat-set materials and custom toning them with acrylics. Illustrations and instructions were provided showing several methods of toning the tissue by brush application as well as mention of the use of the airbrush demonstrated by Elissa O'Loughlin in an AIC-sponsored master inpainting class. Some conservators choose to add a cellulose ether such as Klucel G or methyl cellulose with a few drops of ethanol or other solvent to aid in

This open discussion took place on May 21, 2009, during the AIC 37th Annual Meeting, Los Angeles, CA. The moderators organized the panelists, led the discussion, and recorded notes. Readers are reminded that the moderators do not necessarily endorse all the comments recorded and that although every effort was made to record proceedings accurately, further evaluation or research is advised before putting treatment observations into practice.

dispersion. Reidell stressed that in order to prevent disruption of the thin adhesive layer on ready-made, heat-set tissues the acrylics should not be applied too thickly.

Next, Reidell explained the preparation of pre-coated tissues in the conservation lab. These materials can be prepared in advance of treatment and are fast and easy to use, which is an important factor with large, batched treatments. While Reidell focused on pre-coating papers, conservators also use alternative materials such as goldbeater's skin, fish swim bladders, parchment, Tyvek, Mylar, and Hollytex. When a conservator wishes to reactivate with water, or an ethanol-water mixture, he or she will often use a wheat starch paste and methyl cellulose mix. Other adhesives used in preparation of water-reactivated materials include a 2–5% methyl cellulose, called the "Wagner method" (Wagner 1996), sodium carboxymethyl cellulose, called the "Baker method" (Baker 2007), and isinglass. All of these adhesives require the same application technique, which involves: 1) brushing out the adhesive onto the surface of a sheet of Mylar, 2) evenly draping the repair paper at the farthest edge of the adhesive-coated Mylar, while gradually dropping the paper down, and 3) allowing capillary action to wick the paper onto the adhesive.

The second category was solvent-reactivated materials, starting with the use of Klucel G. Extremely thin repair papers—such as RK-00 and Berlin tissue pre-coated with Klucel G—make nearly transparent mends and are often used in the treatment of iron-gall ink deterioration and other water-sensitive media. Klucel G is applied through a screen to prevent the delicate repair papers from tearing. Frothing rather than traditional brush application of the adhesive mixture ensures that it is properly delivered into the interstices of the screen.

Reidell explained the preparation of pre-coated materials reactivated with solvents and/or heat. Examples included varieties of Lascaux as well as Rhoplex AC 73 and 234, used for what is known as the "Modified" Library of Congress heat-set tissue. These varieties of adhesives are often prepared on silicone-release polyester because they tend to stick to regular polyester films. Lascaux 498 HV can be used unthinned to coat thicker papers by applying the adhesive onto a silicone-release polyester sheet through a silkscreen using a squeegee. After misting the surface, the repair material is dropped onto the adhesive. The Lascaux 498 HV can also be thinned with deionized water. The final adhesive presented was BEVA 371 film (1 ml), which can be heat-sealed directly to the repair paper. There is also a BEVA 371 solution that can be sprayed from an aerosol or applied by brush. This adhesive is especially noted for its use in the repair of parchment and water-repellant oily or greasy paper artifacts. In two video clips Anderson clearly demonstrated the preparation of Klucel G and Lascaux pre-coated repair materials.

After explaining preparation in detail, Reidell described the application of these materials. Large scale treatments are made more efficient by using a scalpel to precut repairs into thin strips a few millimeters wide. Feathered edges release uncoated fibers, ruining the mend. The repair strips are reactivated directly on Mylar, a black-tile for better visibility, a Plexiglas square, or the back of the hand. A common problem encountered by conservators is the appearance of sparkling on the repair surface, which can be avoided by preparing the sheet on a matte polyester film, such as lightly sanded Mylar. Giving the material time to fully gel and reactivate and preparation in a damp-pack or small humidity chamber also assist in preventing sparkling. An additional video clip showing Anderson reactivating a paper pre-coated with Klucel G illustrated the technique used for solvent-set repairs. The repair material is allowed to gel completely before application and the mend is set in place with tweezers and dried under blotters and weights, or with the heat of the conservator's hand.

To round out her discussion, Reidell provided the following tips based on comments and common problems encountered by surveyed conservators:

- Removing the material from a polyester film carrier when it is 99% dry can prevent the carrier from sticking to the paper. Additionally, one can avoid tears by peeling the carrier away from the paper rather than the paper from the carrier. Conservators also use silicone-coated boards and baking sheets as release layers.
- Readily available at hardware stores, wide wallpaper brushes, foam brushes, and paint rollers are useful alternatives to expensive traditional paste brushes, and they are easier to maintain and may not wick up as much adhesive.
- While linings must be custom-sized, pre-coated materials used for mends can be prepared in manageable sizes. Smaller sheets lead to fewer wrinkles, are easier to handle, and help conserve expensive repair papers such as Berlin tissue, which is likely to only be used for small repairs anyhow.
- In order to make use of space when producing these materials for large scale projects, the use of a retractable clothesline for air-drying can be beneficial.
- Dropping the papers onto the surface of the adhesive reduces the typical plastic appearance of the repair material.
- Thin adhesive layers are the most successful. Many people find that their initial tests tend to be too thick so it is important to experiment with the adhesive layer.
- Sometimes a carrier may not be used at all. "Naked" cast adhesive films are sometimes used for readhering delaminations and stepped tears.
- To be thrifty with materials and save on preparation time, many conservators save the margins and offcuts of linings or traditional paste mends and reuse them as "accidental remoistenable" pre-coated repair papers.
- Different properties of solvents can be used to the conservator's advantage, such as using isopropanol, which

evaporates more slowly and allows for more time to set the repair in place. Some conservators mentioned anecdotally that acetone results in more transparent mends because it penetrates the repair substrate.

During the subsequent discussion, questions arose regarding whether there were heat sensitive dyes and if different colorants influence the way the mends react. An audience member suggested the toning of resins. Reidell also pointed out that it is possible to accidentally rinse out the adhesive when toning or reactivating very thin repair papers. An additional audience member commented on successfully using a very thin solution of Klucel G to reactivate the repair material. Comments were made regarding a mixture of Lascaux 360 with 498 HV, which may prevent blocking, and it was mentioned that toned BEVA can be molded into a loss in leather bindings as a fill material, similar to techniques used in the conservation of leather objects. In regard to batched repairs for leather bindings, time is saved by preparing toned tissue in three different colors ahead of time, completing all the repairs followed by fine-tuning the tone, and ending with a final waxing of all completed repairs. Ultimately dwell time is one of the most important factors in successful repairs.

In conclusion, Reidell emphasized that pre-coated repair papers are versatile and lend well to customization. While some time and energy is required in preparation, the ease of use leads to speed and efficiency, especially in the context of large-scale batched treatments. Anderson and Reidell provided a handout that includes a list of adhesives and their preparation guidelines and reactivation methods, which can be found in the write-up for the 2009 LCCDG session (McCann and Haun 2010).

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Sarah Reidell, Associate Conservator for Books and Paper, The New York Public Library

MELISSA STRAW

LARGE-SCALE MOLD REMEDIATION AT A SPECIAL COLLECTIONS LIBRARY

In the second presentation, Melissa Straw explained how more than three hundred thousand special collections items were affected by a mold outbreak at the University of Illinois at Urbana-Champaign (UIUC) in late 2007. The Rare Book and Manuscript Library (RBML), located within the Main Library building at the UIUC, holds approximately three hundred thousand volumes and over 7,100 linear feet of manuscript and archival material. The collection comprises audiovisual media in a wide range of formats, as well as three-dimensional items such as framed artwork and artifacts. From September 2007 to May 2008 the staff of the RBML along with the Preservation and Conservation Departments, Library Administration, and Campus Facilities and Services worked closely to devise and implement a mold remediation plan to clean all items found within the collection.

After university mycologists positively identified the mold as a species of *Aspergillus*, library conservators undertook an item-level assessment of one of the two floors. This assessment required sixty hours to complete but provided invaluable information used throughout the course of the project and confirmed that the mold was widespread throughout the storage vault. A map of the concentration of mold in the storage areas was created showing that the areas with the highest concentration of mold spores were found clustered around HVAC outflow vents. The assessment also confirmed that the mold was growing on a full range of materials from the oldest incunabulum to the most contemporary editions in the collection. To date no identifiable cause for the mold outbreak has been determined, although environmental factors and storage conditions are contributing factors.

After the scope of the outbreak was determined, University Administration and the Conservation Department prepared time frames and budgets for both in-house and outsourced cleaning of the collections. Cost and time estimates, liability issues, and impact on patron services were carefully considered and compared. University Administration decided to contract Blackmon-Mooring Steamatic Catastrophe (BMS CAT), to complete the cleaning of the collection materials, storage areas, and HVAC system during a ten-week period in early spring 2008. Cleaning of the RBML's collections and spaces was completed in May 2008 and certain University departments are still involved in projects related to this large-scale operation.

BMS CAT mobilized a small work crew to begin a week-long cleaning of the three air-handling units. Along with cleaning each unit, they cleaned all associated supply and return vents before beginning on the collection materials. The air handling units were cleaned using a HEPA vacuum on all associated surfaces. Ducts and vents were fogged using chlorine dioxide.

After the HVAC systems were cleaned, the ceilings, floors, and walls of the library were vacuumed and cleaned with Microban, an anti-microbial agent. Areas of positive and negative pressure were established in the collections areas through the use of plastic sheeting, tape, zippers, and microtrap air filtration units. Positive airflow encouraged contaminated air away from an area, and in this case it kept the mold away from areas already cleaned. Numerous zippered chambers were created in order to maintain positive and negative pressure while continuing to use cleaning equipment in different areas.

Additional personnel from BMS CAT assisted with the collection-wide cleaning of over three hundred thousand items

after the air handlers and vents were cleaned. Approximately 2% of the books showed signs of mold growth but the entire collection was treated to save on the time it would take to examine each item individually. Additionally, all the books in the storage vault were soiled even without the presence of mold due to a lack of regular housekeeping procedures.

Conservation staff, along with help from the RBML staff, created a manual detailing care and handling procedures for the various formats found within the library, as well as site-specific policies such as enclosure retention and pamphlet binders. This manual contained a photo identification guide for easy reference. In addition to this manual, BMS CAT employees completed a training session from conservation staff prior to handling the collections.

Using a top-down approach, ten teams of two individuals worked at various points within one range of compact shelving. Each team was equipped with at least one book truck, a variable speed Nilfisk vacuum, an air filtration unit, bookends, string, scissors, etc. In areas without compact shelving within the RBML, the shelves were physically removed from the support structure, vacuumed, and wiped down with Microban. Collection materials were also vacuumed and then wiped with a dry microfiber cloth and replaced onto the cart. Finally, the cleaned books were returned to the cleaned and dry shelf. Once an area was entirely clean, the range of compact shelving was draped with plastic sheeting to prevent re-contamination.

The wide range of materials housed in the RBML presented considerable challenges for both library staff and the contracted BMS CAT cleaning crews. Warping vellum, decaying leather, fragile book jackets, and the many layers within framed art required a great deal of pre-planning by UIUC staff, sensitive handling during the cleaning process by BMS CAT crews, and follow-up vacuuming by conservation staff on items deemed too fragile for others to handle safely. Areas of loose materials such as broadsheets or newspapers were treated in stacks and BMS CAT vacuumed the exterior of each stack. The RBML also has approximately 150 framed items. Each of these were sent to the conservation lab and taken apart for vacuuming. The various layers of each framed piece were photographed and vacuumed with a variable speed Nilfisk vacuum within a bio-safety cabinet. Cleaning took place ten hours a day for ten weeks. Ten conservation and RBML staff members spent at least five hours per week in the storage vault overseeing proper care and handling.

The compact shelving posed a structural challenge. There was not enough space between the ranges to have more than one aisle open at a time so one range of compact shelving held up to three crews at a time. The teams paced themselves, coordinating the completion of each section. Additionally, the compact shelving had a six-inch gap between the wall and the built-in components. Cleaning of the wall and backside of the shelving system was completed through access panels. An additional problem arose due to a lack of electrical outlets. Power supplies were brought in with help from the Library Facilities Department, ensuring that no circuit breakers were tripped due to a greater demand on the power supply.

The contracted cleaning ended in May 2008, and since then the RBML and Conservation Department have undertaken projects related to large-scale tasks. Thousands of items in the collection were already temporarily stabilized with either binder's board and/or string ties. During cleaning for mold remediation, all existing ties were discarded and the stabilization boards were vacuumed. Certain items were flagged for re-tying and the old string ties were replaced with archival quality string. Supplemental binder's board was ordered for items requiring support and stabilization as identified during mold remediation. Additionally, particular types of pamphlet binders were found to be more prone to mold bloom than others within the collection. These pamphlet binders were generally older in nature and had a green cloth spine. Over 6,000 identified pamphlet binders were discarded. Currently the string-tying is complete but the replacement of the pamphlet binders is still in progress.

During the discussion that followed, an audience member asked whether the crew wore respirators during mold remediation. Straw explained that the use of respirators is part of UIUC's policy and the crew was given the option to use them. Ultimately, the crew of BMS CAT chose not to work with respirators, although they took protective measures such as wearing gloves. An additional audience member asked whether the items treated for mold were flagged. Straw responded that they were, using foil-backed labels. In the reading room, these items may require gloves and the book supports will be cleaned more often. If they are deemed unsafe for use they are removed from the collection, therefore there are no special restrictions on use in general. It was mentioned that Harvard used to place flags in items treated for mold but they were removed as requested by the General Council because they implied that all other books are clean, which may not be the case in such large collections. There is a downloadable guideline for managing mold contamination on the Harvard Library Preservation website at: http://preserve.harvard.edu/guidelines/mold_contamination.pdf.

In conclusion, Straw stressed that the cleaning process was very efficient and could not have been achieved in the same time span by an in-house cleaning crew alone. Although the entire process was harrowing and exhausting, the cleaning was done as quickly and efficiently as possible and it was a successful collaboration for conservation and RBML staff. Straw stressed that although an outside vendor was contracted to clean the entire collection, the amount of time and energy UIUC staff put into this project remained high. Straw was happy to report that a new HVAC system will soon be installed to prevent future outbreaks. A handout

providing further details is currently available on the Book and Paper Group website at http://206.180.235.133/sg/bpg/exec/meetings/current_year/ACDG_2009_Straw.pdf.

Melissa Straw, Contract Conservator, University of Illinois at Urbana-Champaign

JENNIFER HAIN TEPER

ASSESSING AND STABILIZING ARCHIVAL SCRAPBOOKS

Jennifer Hain Teper introduced a large collection of scrapbooks and photo albums that document student life and culture from the early twentieth century to 2005, held in the University of Illinois at Urbana-Champaign's (UIUC) Archives. These memory books contain minutes, diaries, letters, notes, captions, official University documents, photographs, program booklets, art work, sheet music, news clippings, artifacts (such as homecoming pins, buttons, and freshman beanies), and animal- and plant-based substances (tobacco, cake, locks of hair). Compiled by individual students, student organizations, student administration, sports officials, student offices, and alumni, these scrapbooks provide a unique snapshot of the student culture and experience from particular periods of time. Many of the items in these scrapbooks are not found in any other collection in the Archives, therefore they offer a unique resource for a variety of researchers. Scholars consult these books to gather information about student culture for specific time periods while many current students, fascinated by the early books, write papers about the scrapbooks' individual creators. Additionally, leaders of student organizations consult their groups' scrapbooks for past history and administrative information, and genealogists use them to learn about their family history.

From 2005 to 2006, Teper and the conservation staff at the UIUC performed an assessment of the conservation needs for the entire collection of 494 items, funded by an internal NEH Challenge Grant. Completed in September 2006, this assessment and the resultant report provided the Student Life and Culture Archives and the Conservation Unit with the information to strategically approach the conservation treatment of these endangered and irreplaceable artifacts. In January 2007, the Conservation Unit began the lengthy project. Conservation staff performed simple repairs such as paper mending and the reattachment of loose artifacts for 112 scrapbooks noted in the assessment as exhibiting "poor" levels of attachment. Custom protective enclosures or the modification of existing enclosures were completed for 148 scrapbooks noted as "severely damaged," with either extremely fragile support pages or severely damaged bindings. The project was completed in May 2008. Organization and careful record-keeping is the key to running a large-scale project. Figure 1 provides a sample entry from the Conservation Unit's assessment form used for the scrapbook project.

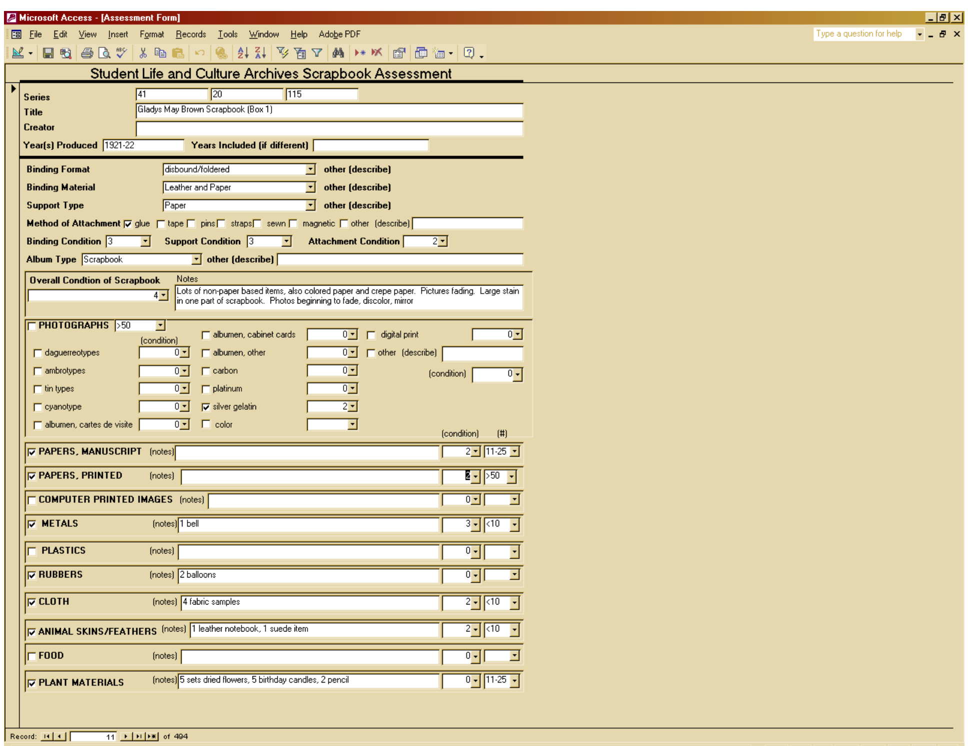

Fig. 1. Sample entry from the scrapbook assessment tool: The conservation assessment form

In the following discussion, a question arose regarding whether preservation photocopies were considered in place of treatment for the scrapbooks. Teper explained that it was definitely an option and may be considered for parts of the remaining untreated collections when deemed necessary and would be addressed in the future.

Teper shared the following time and cost observations:

- Development of guidelines: 30 hours total
- Protective enclosures for 148 items at 1 hour per scrapbook = 148 hours total
- Reattachment/repair for 112 items at 2.5 hours per scrapbook = 280 hours total
- Total estimated project time = 458 hours

A handout providing further details on this project was prepared by Teper and is currently available on the Book and Paper Group website at http://206.180.235.133/sg/bpg/exec/meetings/current_year/ACDG_2009_Teper.pdf.

Jennifer Hain Teper, Head, Department of Conservation, University of Illinois at Urbana-Champaign

BRENDA BERNIER

LESSONS LEARNED FROM LARGE-SCALE PHOTOGRAPH PRESERVATION PROJECTS AT THE NATIONAL ARCHIVES AND RECORDS ADMINISTRATION AND HARVARD UNIVERSITY LIBRARY

Brenda Bernier knows from experience what it takes to complete large-scale preservation projects. As the former senior photograph conservator at the National Archives and Records Administration (NARA), and now as the Paul M. and Harriet L. Weissman Senior Photograph Conservator at Harvard University Library, Bernier has been involved in numerous large-scale preservation projects, with collections containing tens of thousands to hundreds of thousands of photographs. Looking back at some of these projects, Bernier presented eight general lessons for planning and executing large survey, treatment, and rehousing efforts. She also offered a few specific tips for saving time, money, space, and aching backs.

Bernier began the presentation with her definition of large-scale projects. A small project includes one to one hundred items, a medium project is one hundred to ten thousand items, and a large project is ten thousand to a million items. With each of the lessons presented, Bernier gave examples and provided images from real-life projects. The scope of what she has dealt with in her career is impressive, and although the vast number of items she has been responsible for is daunting, the prospect of realistically accomplishing the preservation of such monumental collections is achievable with a little organization and creativity.

The following is a list of lessons learned:

Lesson 1 – Dedicate time each week

By reserving specific time in your schedule for the project it is easier to see progress and you are less likely to succumb to project fatigue. Bernier advised the audience not to lose sight of the project goals, and to consider the individual situation. Will the project require a large amount of supplies? Is it more cost effective to buy large quantities of materials, or if storage is inadequate are you better served by ordering supplies on an as needed basis?

Bernier provided an example of ten to twenty thousand distorted, mounted photographs belonging to the U.S. Navy Records at NARA, which were slumped and unsupported in flip-top document boxes. Before they could be properly rehoused, the photos had to be flattened. Therefore the photos were efficiently humidified in stacks of damp blotter and Tyvek. The following tips were provided:

- Tyvek is an excellent substitute for Gore-tex when humidifying objects.
- After initial humidification, a fine mist from a commercial air-pump garden sprayer was used to wet the verso of the photos. Since large batches of photographs were humidified at once, the garden sprayer was more efficient and comfortable than traditional Dahlia or pump sprayers (Bernier 2005).
- A stainless steel rolling kitchen cart with multiple shelves is useful for keeping large pre-cut blotters and other supplies handy and mobile.

Lesson 2 – Large custom orders can save time, space, and money

To deal with moving 53,000 cubic feet of film in off-site storage for NARA, Bernier ordered custom boxes. Using these ready-made boxes to pack for moving resulted in efficient use of space as well as time. Polyester sleeves on a roll were also a useful custom order for large-scale treatments. Panorama fold-lock sleeves on a roll from Atlantic Protective Pouches have been used for large-scale rehousing at both NARA and Harvard University.

Lesson 3 – Do a mock-up to estimate time

When confronted with the rehousing of nearly 95,000 lantern slides at the Fine Arts Library of Harvard University, it was helpful to do a mock-up before estimating the time and cost of the project. The first month of work produced a little less than the estimate, but the project was ultimately completed ahead of schedule.

Lesson 4 – Be comfortable

The 25,000 to 40,000 Angus McBean glass plate negatives in the Harvard Theatre Collection are stored in a room with virtually no appropriate space for completing conservation work. Considering the scope of the project, it was not feasible to move the negatives to another location to be rehoused. The expense of buying small but comfortable chairs and a table that fit easily in the narrow hallway was well worth the purchase price.

Lesson 5 – Adapt tools

Bernier gave a detailed account of using an inkjet copier to transcribe historical inscriptions onto new archival enclosures, eliminating transcription error and allowing for the preservation of original handwriting and penmanship. She provided a handout that was originally created with Andrea Youngfert, also of the Weissman Preservation Center at Harvard University, as a poster for the Society of American Archivists 2008 Research Forum. The handout can be currently viewed on the Book and Paper Group webpage at: http://aic.stanford.edu/sg/bpg/exec/meetings/current_year/bpg_prog.html. In summary, Bernier and Youngfert describe the use of an Epson Stylus CX6000 3-in-1 inkjet copier to efficiently copy 1,700 historical inscriptions from acidic original negative enclosures onto archival storage envelopes. The copy system saved on time and resources and can be used with enclosures ranging in size from 4x5 to 8x10 and even 4-flaps. Pigment-based inks can be water-soluble but tests by Bernier indicated that in the event of a flood, the ink does not bleed through the enclosure and remains legible. One other caution is that the density cannot be manually adjusted on many newer inkjet copiers, which is a drawback when copying light pencil inscriptions. Density manipulation can be achieved through the use of colored filters found at stage-lighting suppliers, although this also darkens the background resulting in a darker tone overall.

Lesson 6 – Know your materials

Non-traditional photographs can stump the conservator when it is not clear what materials were used and how they might deteriorate. Bernier encountered this with a collection of three-dimensional experimental vectographs from the Polaroid Corporate Collection at Harvard's Baker Library Historical Collections. With large collections, it is worth the time spent on researching the technical aspects of the material so the long-term preservation can be well planned from the start of the project.

Lesson 7 – Get organized

Keeping track of the preservation needs of Harvard's eight million photographs can be a tremendous challenge. Bernier discussed the creation of an Access database called COMPASS—Comprehensive Preservation Assessment. The system was designed in-house to track the preservation needs of special collections in over 50 Harvard repositories. The needs can be prioritized and linked to projects, which are also tracked in terms of tasks, materials, labor, timeframe, and costs. Even without such a sophisticated system, Bernier emphasized the importance of using simple spreadsheets or other electronic tracking to keep control of project data.

Lesson 8 – Don't panic!

Addressing the preservation needs of large collections can seem daunting, even to experienced conservators. However, it is important to remember that the lessons Bernier presented are adaptable and scalable to projects of many types and sizes. Bernier's experiences give true meaning to the phrase "large-scale." In conclusion, organization and thorough preparation well in advance are the keys to staying calm and collected while achieving large goals.

A handout providing further details on this project was prepared by Bernier and is currently available on the Book and Paper Group website at http://206.180.235.133/sg/bpg/exec/meetings/current_year/ACDG_2009_Bernier.pdf.

Brenda Bernier, Paul M. and Harriet L. Weissman Senior Photograph Conservator, Weissman Preservation Center, Harvard University Library

ACKNOWLEDGMENTS

The co-chairs of the ACDG would like to thank Priscilla Anderson, Sarah Reidell, Melissa Straw, Jennifer Hain Teper, and Brenda Bernier for sharing their experiences, challenges, successes, and ongoing work with large-scale collections. Thanks also go to the LCCDG co-chairs Laura McCann and Werner Haun for support in creating such lively discussion sessions centered on the conservation and preservation of library and archival materials during the 2009 AIC annual meeting.

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LAURA MCCANN AND WERNER HAUN
DISCUSSION GROUP CO-CHAIRS

Library Collections Conservation Discussion Group 2009: Library Collections Conservation 2.0—New Directions. New and/or Adaptive Materials, Methods and Technologies Used in the Conservation Treatment and Housing of Library Collections

ABSTRACT

The Library Collections Conservation Discussion Group (LCCDG) of the Book and Paper Group was pleased to present "Library Collections Conservation 2.0—New Directions. New and/or Adaptive Materials, Methods and Technologies Used in the Conservation Treatment and Housing of Library Collections" at the 2009 AIC Annual Meeting held in Los Angeles, California. The theme for the session was inspired by the AIC annual meeting theme, "Conservation 2.0—New Directions." The session co-chairs, Laura McCann and Werner Haun, recruited speakers to present on practical applications of new and adaptive materials and methods in the conservation of library collections. Following the presentations, the co-chairs moderated a lively discussion period. Handouts were provided for each of the presentations and are published as figures.

PRESENTATIONS

SETH IRWIN AND RANDELL HEATH
COMPARISON OF TWO SOOT REMOVAL TECHNIQUES

Seth Irwin began the presentation by explaining the impetus of his project, comparing two soot removal techniques (fig. 1). In May 2006, a fire in the Sevier County Recorders Office in Richfield, Utah, caused soot damage to approximately three hundred ledgers. Randy Silverman, Preservation Librarian, Marriot Library, University of Utah, was called upon to assist in the recovery of these ledgers and he employed an experimental cleaning technique called "dry ice dusting" for removing the soot residue from the smoke-damaged ledger books. Visual observations suggested the technique was more effective than conventional surface cleaning with a rubber sponge. From this experience, there was interest in obtaining quantifiable analysis in order to consider dry ice dusting a viable option for treating smoke-damaged books. A project was conceived to compare dry ice dusting with rubber sponge cleaning, which is the more typical method, for removing soot residue.

Randell Heath, founder and president of Coldsweep, Inc., a company specializing in dry ice applications, described the "dry ice dusting" methods his company has used for soot removal. Dry ice dusting has been used in a number of applications in the recovery of cultural heritage collections, including cleaning five hundred thousand soot damaged books in Göttingen Germany. Essentially, the dry ice dusting is a spray application of solid carbon dioxide using a high-efficiency particulate air (HEPA) filter vacuum to draw the released soot away. A stated advantage of dry ice is that it is non-abrasive to materials that are harder than dry ice.

Irwin continued the presentation by describing his study methodology and results. The research method compared the effectiveness of the dry ice dusting and the rubber sponge cleaning method by measuring residual soot remaining on cleaned book surfaces with colorimetry and surface abrasion using laser scan profilometry.

Of the two cleaning systems, Irwin found that dry ice dusting consistently excelled at preventing surface abrasion to the book covering materials and at removing the soot. In a few instances the rubber sponge did slightly better at removing residual soot, but with a significantly higher probability of causing surface abrasion—in some cases with only a minimal number of wipes. One conclusion arising from this study is the certainty that dry ice dusting, when carefully applied, is a viable option for soot removal from the exterior of bound library materials.

Seth Irwin, Master of Arts degree candidate, Queen's University, and Randell Heath, President/Founder Coldsweep Inc.

Randell Heath, President/Founder Coldsweep Inc.

This open discussion took place on May 21, 2009, during the AIC 37th Annual Meeting, Los Angeles, CA. The moderators organized the panelists, led the discussion, and recorded notes. Readers are reminded that the moderators do not necessarily endorse all the comments recorded and that although every effort was made to record proceedings accurately, further evaluation or research is advised before putting treatment observations into practice.

Seth Irwin
Candidate for Masters in Art Conservation,
Queen's University, Kingston, Ontario, Canada

Randy Silverman
Preservation Librarian
Marriot Library, University of Utah

A Comparison of Two Soot Removal Techniques:

"Dry Ice Dusting" and Rubber-based Chemical Sponges

Abstract:

Pressed by the exigency of a fire in the Sevier County Recorders Office (Richfield, Utah), in May, 2006, Randy Silverman employed an experimental cleaning technique called "Dry Ice Dusting" for removing soot residue from the surface of smoke-damaged ledger books. Visual observation suggested the technique was more effective than conventional surface wiping with rubber-based sponges but quantifiable analysis was impossible to consider at the time.

Accordingly a project was conceived to compare dry ice dusting with conventional rubber sponge cleaning for removing soot residue from the surface of smoke-damaged books. The study defined an experimental approach that standardized soot deposition on four types of bookbinding material (leather, fine and coarse cloth, and paper). The research compared the effectiveness of these two cleaning methods by measuring residual soot remaining on cleaned book surfaces with colourimetry, and surface abrasion using laser scan profilometry.

Conclusions:

Cleaning efficiency and abrasion using dry ice misting and rubber sponge cleaning were compared for soot removal from four types of bookbinding materials. Of the two cleaning systems, dry ice misting consistently excelled at preventing surface abrasion to the book covering materials and consistently cleaned very well. In a few instances the Gonzo® Wonder rubber sponge did slightly better at removing residual soot but with a significantly higher probability for causing surface abrasion, in some cases with a minimal number of wipes. One conclusion arising from this study is the certainty that dry ice dusting, when carefully applied, is less abrasive than traditional dry rubber sponge cleaning.

Clear characterizations of proper cleaning protocols for soot damaged books have yet to be proffered in the literature for either technique. As a result of this study it has been demonstrated that dry ice misting can be effectively used for cleaning in a non-abrasive manner if the nozzle is held approximately 18 inches from the book surface and the mist is played onto the object in a constantly moving motion. With the rubber sponge, thorough cleaning is achieved with approximately 20 passes over the soot-covered surface, with the caveat that complete cleaning is often accompanied by abrasion of friable surfaces. This was determined using the science of profilometery and colorimetry, where a link was observed between changes in colorimetric values and changes in surface topography. Finally, a standardized protocol for controlling the deposition of soot on different materials was established for conservation by relying on existing standards currently in use at the NRC Fire Research Program National Fire Laboratory in Almont, Ontario.

Fig. 1.

BRENNA CAMPBELL
THE REMOVAL OF EXCESS LEATHER DRESSING USING THE REYNOLDS HANDI-VAC: FIRST IMPRESSIONS

Brenna Campbell discussed her work with removing leather dressings from leather books at the Morgan Library and Museum (fig. 2). Dressings of various concoctions have been used to refurbish or condition leather books; however, over time these treatments have fallen out of favor because "dressed" books suffer from bloom, formation of waxy verdigris around metal furniture, unpleasant odor, and sticky residue on the leather.

Residual dressing can sometimes be reduced with solvents, but traditional methods of application, such as poulticing and swabbing, can be technically and aesthetically problematic. In order to develop new treatment options, an experimental method of introducing a controlled amount of solvent within a vacuum packet created using the Reynolds Handi-Vac system was tested. Samples of different leathers were treated with various dressings and the preliminary results evaluated. Moderate success was reported in reducing and removing the dressing from calf skins with oil stains, and it appeared to be less effective on goat skins with waxy dressings. Issues with this the system were addressed, including the difficulty in monitoring treatment progress. The presentation concluded with suggestions for other uses of the Reynolds Handi-vac, including controlled drying, packing books for moves, and disaster recovery.

Brenna Campbell, Kress Fellow in Rare Book Conservation, Thaw Conservation Center, The Morgan Library and Museum

RENATE MESMER AND ANNE HILLAM
THE USE OF RUBBER CEMENT FOR FACING LEATHER SPINES: A VIABLE OPTION?

Renate Mesmer introduced the background and development of using rubber cement as a temporary facing for degraded leather books (fig. 3). The technique was developed by Per Cullhed in 1996 to treat fire damaged books in the city library of Linköping, Sweden (Cullhed 2003). Approximately one hundred tight-back books were heavily fire damaged, rendering the spines brittle, inflexible, and prone to cracking and loss. Per Cullhed researched a facing method that would cause the least possible damage to the spines and also retain the tooling. After testing various methods, it was determined that the rubber cement technique added flexibility and visibility, as well as a weaker bond that allowed the facing to be easily removed mechanically rather than with solvents.

In the second half of the presentation Anne Hillam discussed and presented slides showing the steps involved in this technique. The process must be completed in two weeks or less to prevent the rubber cement from hardening. Some of the advantages of this technique are: it is flexibile, it uses no solvents, there is no tissue residue, and it is less damaging to the spine because of the minimal adhesive bond. Some potential disadvantages are the time constraints and the need for a wax release layer. Additionally, the presenters emphasized that they use this technique only in extreme cases where no other technique is viable, and that quantitative and qualitative testing is needed to determine its long term effects.

Renate Mesmer, Assistant Head of Conservation, Folger Shakespeare Library and Anne Hillam, Head of Conservation, Gladys Brooks Book and Paper Conservation Laboratory, New York Academy of Medicine

Anne Hillam, Head of Conservation, Gladys Brooks Book and Paper Conservation Laboratory, New York Academy of Medicine

PRISCILLA ANDERSON AND SARAH REIDELL
ADHESIVE-COATED REPAIR MATERIALS: PREPARATION AND USE

This was a joint presentation with the Archives Conservation Discussion Group (ACDG). Priscilla Anderson presented during LCCDG while Ms. Reidell gave her portion of the presentation during ACDG. Both presenters participated in the discussion period (fig. 4).

Priscilla Anderson began the presentation by defining pre-coated repairs as sheets of materials that have been coated with adhesive and then dried for later use. The adhesive is reactivated with liquids, solvents, or heat. Some of the advantages are that little or no moisture comes into contract with the original, the repairs are applied and dried quickly, and the conservator can customize the adhesive, repair sheet and color to match the needs of the original. Through surveying the profession she has found that pre-coated repairs are being used in libraries, archives, museums, regional conservation centers, and private practice to conserve both circulating library collections and special collections. The adhesive-coated repair materials are applied in batch treatments and in single-item treatments. The stated advantages for single item treatments are generally good reversibility and portability. In addition, the method is appropriate for easily friable and water-soluble or -activated media. It is also suitable when tide lines are likely, where cockling or dimensional instability is a concern, and for mold-damaged or brittle items. For batch treatments, the advantages are ease and speed of use. The disadvantages are that adhesion may be difficult, there is less feathering than traditional mends, and there may be concerns about aging and reversibility, and solvent-sensitive media/coloring.

The Reynolds® Handi-Vac™: First Impressions

What it is

The Reynolds® Handi-Vac™ was designed as an inexpensive alternative to home food sealing systems.

How it works

The item to be sealed is placed inside a zip-closure polyethylene bag, the bag is sealed, and the air is sucked out. Under optimal conditions, the seal can last months. In practice, it must be checked periodically.

Conservation Applications

- Removal of stains and residues from leather and paper
- Packing of wet books to delay mold growth
- Controlled drying
- Stabilization of damaged books for transport or storage
- On-site work
- Substitute for traditional vacuum packer

Advantages

- Inexpensive (~$10)
- Portable
- Battery powered (6 AA)
- Bags can be re-used a few times
- Sealing system doesn't use heat
- Can seal items of many shapes

Disadvantages

- Seal can fail over time, especially on re-used bags
- Bags must be bought from Reynolds®
- Large size bag can only accommodate books 9-10" high

Tips

- For short term use (solvent treatment, flattening), bags can be re-used
- For long term storage, a new bag should be used each time
- Pieces of thin board can be sandwiched on either side of book for added protection, or between boards and textblock
- Dry blotter can be used to dry wet materials
- Blotter dampened with water or a solvent can be used for humidification or stain reduction

Cautions

- Quality control on bags is spotty—must check seal periodically
- System is not designed to handle flowing liquid—blotters or other absorbent material must be used to contain any liquid
- The Handi-vac™ filter chamber is soluble in acetone

For More Information

Handi-Vac™ Website: http://www.reynoldspkg.com/reynoldskitchens/handi_vac/en/home.asp

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Fig. 2.

The Use of Rubber Cement for Facing Leather Spines: A Viable Option?

This technique was developed by Per Cullhed in 1996 to treat fire damaged books in the city library of Linköping, Sweden. Approximately 100 tight back books were heavily fire damaged, rendering the spines brittle, inflexible, prone to cracking and loss. A facing method was needed that would cause the least possible damage to the spines (Many had retained their tooling). Various techniques were tested, including Japanese paper, heat-set and Archibond tissue, all requiring solvents to activate the adhesive layer. Rubber cement was added as an alternative method. After testing, it was determined that the rubber cement technique added flexibility, visibility and a weaker bond that allowed the facing to be removed mechanically.

This technique has been used both at the Folger Shakespeare Library and at the New York Academy of Medicine for the removal of tight back spines that would otherwise be too difficult to lift. It has proved to be a successful alternative to more traditional facing methods. As more members of the conservation community try this technique, we would love to get feedback from your results. Scientific testing results would be especially welcome.

Cullhed, Per. ***Facing Leather: A Description of a Facing Method for Fire Damaged Tight Back Leather Bindings.*** Paper Restaurierung Vol 4 (2003) No. 4.

Sequence:

1- Apply a layer of Micro-crystaline wax on the spine. This both establishes a barrier between the leather and the rubber cement and as a release layer allowing for easy removal of the adhesive.
2- Using a brush, apply a layer of rubber cement.
3- Place plastic wrap on top of the rubber cement and press with your fingers to ensure full adhesion. Pay particular attention to the sides of raised cords. (When facing a spine with raised cords you need to apply individual panels of plastic wrap instead of one single piece).
4- Remove spine piece (or label) with knife, as you would when using more traditional facing methods.
5- Once faced spine is removed, use either a spatula or a Dremmel to remove flesh layer in order to achieve the desired thickness. The plastic wrap provides support and flexibility sufficient to allow the creation of a very thin spine piece.
6- Reattach spine (or label) to new spine and let dry completely.
7- Gently remove plastic wrap, working slowly from one end to the other. Some rubber cement will lift up during this process. When the plastic wrap has been removed, peel or roll off any remaining rubber cement – it should come away easily. It is important to carry out this process within a three or four day period so that the rubber cement does not completely dry; it needs to remain somewhat gummy in order to facilitate easy removal.

Anne Hillam
Head of Conservation
Gladys Brooks Book and Paper Conservation Laboratory
New York Academy of Medicine

Renate Mesmer
Assistant Head of Conservation
Werner Gundersheimer Conservation Laboratory
Folger Shakespeare Library

Fig. 3.

Adhesive Pre-Coated Repair Materials Book and Paper Group, LCCDG and ACDG
Priscilla Anderson and Sarah Reidell May 21, 2009

Thanks to our generous conservation colleagues who contributed their opinions and experience. Special thanks to Jan Paris, whose curiosity and encouragement got us started.

ADVANTAGES	Successfully treated originals
Low- or no-moisture: suitable for water-soluble and water-reactive media; avoid distortion + tide lines; avoid reactivating mold damage	Iron-gall ink; copper-based media (Islamic mss, verdigris); brittle paper; art on paper with water-soluble media; colored papers; historic textiles; mold damaged or weak papers; Chinese papers (rubbings, books); transparentized papers; copy press books; thin papers
Customizable: your choice of repair sheet; pre-coating makes it possible to use a very thin mending paper (can't be pasted out, too hard to handle in wet-floppy state); can get very transparent mends	Double-sided manuscripts and art on paper
Speed: quick application and drying time	Circulating collections, batch mending projects; stabilization for large-scale digital projects; photographs mounted on boards
Portable: few tools needed, very little mess	Traveling exhibits and loans, on-site treatment
Reversibility: easy short-term removal lends itself to temporary applications	Leather spines; bridge mends and facings
Custom toning: before coating, after coating, even after mending	Leather and parchment bindings
Compatible: repair is aesthetically or materially compatible with original	Parchment texts and bindings; original pressure sensitive tape; short-fibered paper, clay-coated paper

DISADVANTAGES	How to mitigate:
Strength or flexibility of the repair (some are too strong/inflexible, some are not strong enough)	Experiment with adhesive type, dilution, preparation method, and with repair sheet thickness; if having trouble sticking, gel up the adhesive layer in damp pack or chamber
Undesirable plastic or sparkly look	Gel up the adhesive layer completely during application; experiment with plastic sheet that you are preparing on (i.e., matte polyester drafting film rather than shiny polyester
Time & complication of pre-coating the repair material	Weigh it against time saved in application
Staining/tidelines are possible from in situ liquid application	Test application method on small area of original; avoid in situ application with originals likely to stain
No feathered edges	Pin tear; use a thinner repair sheet that will blend in
Inconsistent application of adhesive can make portions of the pre-coated sheet useless	Experiment with screening (window or silk-screens), different brushes; reconsider for other application use
Original has solvent sensitivity	Stick with cellulose ethers and wheat starch paste because long-term reversibility of acrylics isn't proven
Repair more dimensionally stable than original	In storage conditions with dramatic RH fluctuations, stick with thinner repairs made with cellulose ethers, which discourage biological attack
Adhesive aging characteristics and long-term reversibility questionable	See forthcoming CCI research

Fig. 4.

Priscilla Anderson, Collections Conservator, Baker Library Historical Collections, Harvard Business School, and Sarah Reidell, Associate Conservator for Books and Paper, New York Public Library

Sarah Reidell, Associate Conservator for Books and Paper, New York Public Library

DISCUSSION SESSION

Immediately following the final presentation, the co-chairs opened the discussion period for comments and questions/answers. Questions and comments from the audience were directed to all the speakers and are summarized by presentation.

COMPARISON OF TWO SOOT REMOVAL TECHNIQUES

There was a great interest in dry ice dusting applications in the preservation of library collections. One participant asked if dry ice dusting can be used for cleaning mold. Heath described residential application of mold remediation using dry ice dusting, but he has not applied the method in treating mold-infected library collections to date. Additional questions addressed the practicality of dry ice dusting, specifically the time and set-up requirements, cost-effectiveness, and the possible side effects caused by condensation. The speakers reported that: an average of five minutes was required to effectively clean soot from a volume using dry-ice dusting (20–30 psi), the set-up can be in-situ or the books can be sent to the vendor, and there was no observed evidence of condensation on volumes after dry-ice dusting. Significant reduction of odor was reported after dry ice dusting, which was also observed in the testing with the rubber sponge. Cost effectiveness was considered on a case-by-case basis dependent upon the scale of the project.

THE REMOVAL OF EXCESS LEATHER DRESSING USING THE REYNOLDS HANDI-VAC: FIRST IMPRESSIONS

A question regarding the type and age of the leathers used in the testing of the leather dressing removal (modern leathers) led to a discussion about modern versus historical leathers. It was suggested that the results of the leather dressing removals may be more successful on historical leather than modern leathers as the quality of modern leather is relatively poor and further testing with historic leather would be worthwhile.

There was interest from the audience in the Reynolds Handi-Vac and its various applications in library collections conservation. Campbell described other projects using the device or similar devices. These including the controlled drying of the recently discovered "Bog book," and using it for moving large collections and a range of anoxic treatments.

THE USE OF RUBBER CEMENT FOR FACING LEATHER SPINES: A VIABLE OPTION?

Questions directed to Anne Hillam and Renate Mesmer about the use of rubber cement as a facing material focused on technique and materials. The speakers have only used the technique on calf bindings to date and have not observed any darkening of the treated spines. A discussion took place on preparation activities necessary before using the facing, consolidation, and the application of the microcrystalline wax release layer. Mesmer reported that she used the consolidant Klucel G in either an ethanol or acetone solution. Hillam described her method for applying the very thin layer of microcrystalline wax using a cotton pad. Methods for removing the rubber cement were described by the speakers as using fingertips and rubber crepe erasers. The importance of fully drying the wax release layer before application of the rubber cement was emphasized. A participant inquired about alternatives to microcrystalline wax. One suggestion of cyclododecane was rejected, because of concerns of brittleness. Speakers encouraged further research into the method, especially research employing natural aging and instrumental analysis.

ADHESIVE-COATED REPAIR MATERIALS: PREPARATION AND USE

Priscilla Anderson and Sarah Reidell encouraged participants to attend the Archives Conservation Discussion Group for further information about the preparation of adhesive-coated repair materials. A question was raised about failed mends and mends that were very shiny. The speakers emphasized the need to completely swell the adhesive before application as a repair material. Shininess is caused by non-adhered adhesive. A discussion took place on the methods used to cut the repair material. Scalpels, Olfa knives, awls, pin tools, and the Crayola Cutter were reported as tools used for shaping repairs.

ACKNOWLEDGMENTS

The co-chairs of LCCDG wish to express their gratitude to speakers Priscilla Anderson, Brenna Campbell, Randell Heath, Anne Hillam, Seth Irwin, Renate Mesmer, and Sarah Reidell for their presentations and handouts. Their willingness to share their experience with new and adaptive materials and methods is greatly appreciated. The co-chairs also thank Angela Andres for recording the session.

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Clearing the Image: A Quantitative Analysis of Historical Documents Using Hyperspectral Measurements

ABSTRACT

Quantitative hyperspectral imaging is an optical measurement technique that has a great potential for historical document analysis. With the technique described here, optical images in seventy different wavelength bands are recorded. These images are subsequently spectrally calibrated by comparing them to recordings of a white calibration target with a known spectral reflectance curve. Post-processing algorithms for feature extraction and classification applied to the calibrated spectral image data can be used to investigate document aging phenomena and enhance the visibility of hidden features such as underdrawings.

In a first example a supervised feature extraction and classification algorithm is used to map the distribution of iron-gall ink corrosion within a nineteenth-century handwritten letter. The example shows that with the described procedure it is very possible to discern printing ink, uncorroded iron-gall ink, and corroded iron-gall ink.

The second example describes an unsupervised feature extraction and classification method, which was used to visualize underdrawings on a seventeenth-century historical map. The resulting false-color image shows a drastic increase in contrast between the underdrawings and the surrounding ink lines and pigments. Both examples demonstrate the great potential of the QHSI technique in combination with well-chosen, post-processing algorithms for investigation of historical documents.

INTRODUCTION

Major public archives and libraries worldwide have two competing tasks. In the first place, documents of cultural value should be made accessible to researchers and the general public of the present generation. Secondly, the institutes are responsible for keeping these documents in optimum condition for future generations. For the first task, new quantitative techniques are required that help to extract as much information as possible from already-degraded and fragile documents in a completely non-destructive way. For the second task, quantitative techniques are required to support an objective evaluation of the document condition and environmental influences therein. Hyperspectral Imaging (HSI) is a technique with which objects are imaged in tens or even hundreds of wavelength bands. Typically, HSI covers not only the visible, but also the near-ultraviolet and near-infrared, spectral range. This technique has already proven its worth in various fields such as agricultural research, space exploration, environmental investigations, and defense (Stein et al. 2001; Kruse et al. 2003; Lawrence et al. 2003). Often, these HSI systems are mounted on airborne or space-borne vehicles. On a microscopic scale, HSI technology is increasingly becoming a valued research tool especially in biomedical research (Schultz et al. 2001, Qin and Lu 2006).

However, it is also possible to use HSI for investigation of objects of cultural heritage. In the last few years this technique has been introduced for research in the area of document conservation and document analysis (Melessanaki et al. 2001; Mansfield et al. 2002; Casini et al. 2002; Attas et al. 2003; Klein et al. 2006; Kubik 2007; Padoan et al. 2008; Klein et al. 2008).

In this contribution we discuss the application of advanced numerical hyperspectral image processing techniques applied to the analysis of historical documents. Powerful computer algorithms for spectral feature extraction and classification allow one to detect even very subtle correlations in the hyperspectral data that cannot be detected with conventional visual comparison. Such algorithms can provide reproducible, quantitative results that enable a comparison of different objects or of the same object measured at different times, so that it becomes possible to establish databases and to measure the influences of treatments or aging. In the following, two particular applications will be discussed: 1) Evaluation of iron-gall ink corrosion and 2) detection of underdrawings.

Poster presented at the AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

MEASUREMENT PRINCIPLE

The SEPIA quantitative hyperspectral imager used in these experiments is based on two wavelength TUnable LIght Projectors (TULIPs) that illuminate the document under an angle of 45°, and a monochrome digital camera that records the document from above (fig. 1). The light sources subsequently illuminate the document with a series of seventy well-defined optical wavelengths in the ultraviolet, visible, and near-infrared wavelength range (365–1100 nm). At each wavelength, a 4 megapixel grayscale image of a document area of 125 mm x 125 mm is recorded, corresponding to a resolution of 60 μm x 60 μm per pixel (ca. 400 dpi).

In order to translate the pixel values of each image into quantitative measurement values of the local spectral reflectance of the document, the recorded images have to be compared to recorded images of a reference target for each wavelength band. In this case recordings of a white reference target (Spectralon target, supplied by Labsphere, Inc.) with known reflectance are used for this calibration step. After this calibration, the value of each image pixel represents a precise measurement of the fraction of light reflected from the corresponding tiny document area at this particular wavelength and can be regarded as a local quantitative reflectance measurement. This hyperspectral imaging technique is therefore referred to as Quantitative Hyperspectral Imaging (QHSI) (Klein et al. 2008). The entire set of these (calibrated) spectral images is called the *hyperspectral data cube*. It contains for each pixel the entire spectral reflectance curve (fig. 2).

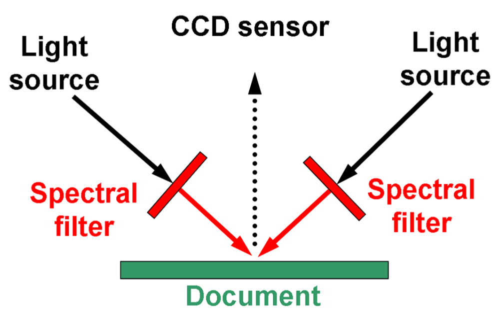


Fig. 1. Schematic overview of the QHSI instrument setup

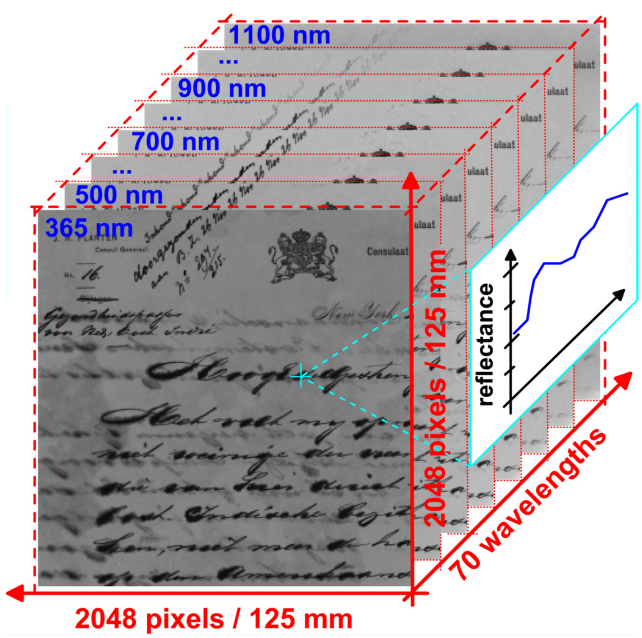


Fig. 2. Schematic representation of the hyperspectral data cube

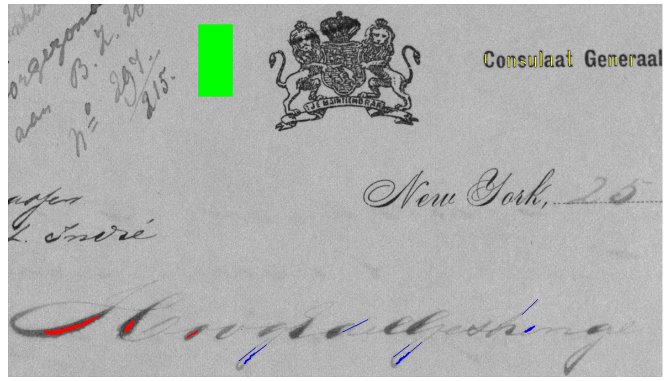
The spectral information in the hyperspectral data cube can then be used to distinguish different writing materials, such as inks and pigments, to measure the progress of aging processes, to enhance the legibility of degraded texts, and to determine deterioration effects on a document following an exposition or treatment.

MAPPING OF IRON-GALL INK CORROSION BY SUPERVISED CLASSIFICATION

In the field of conservation of documents, the behavior of ink over time is often studied (Public Record Office 1999; Havermans et al. 2003). Many of these studies focus on the deterioration of metal gall ink, due to its use in the production of handwriting inks for over one-hundred years and its well-known instability.

One important task for assessing the condition of a document is to distinguish ink areas that exhibit different degrees of corrosion. Figure 3 shows a section of an old letter (dated 1885) that contains both printed and handwritten text on a paper substrate. The handwritten text consists of iron-gall ink, which is in some places heavily corroded. A supervised classification procedure using QHSI measurements was used to assess the degree and distribution of the ink corrosion within this document.

The first step in the classification procedure is the definition of the different classes that are to be discerned (different spatial regions that can be identified on the document). In this case the classes *substrate* (paper substrate without ink), *corroded* (corroded iron-gall ink), *uncorroded* (uncorroded iron-gall ink), and *print* (print ink) have been defined. For each class a so-called Region-Of-Interest (ROI) is defined within the image, which represents a set of image pixels that can be clearly ascribed to one specific class. In our example, we defined the four ROIs as shown in figure 4. Using a so-called Spectral Distance Similarity (SDS) feature extraction algorithm (Homayouni and Roux 2004; Klein et al. 2008) all relevant spectral information for each pixel, which in this case is distributed over the seventy different wavelength bands, is compressed to four values per pixel (four, since four classes have been defined).



LEFT TO RIGHT

Fig. 3. A real-color image of a part of an old letter (dated 1885) containing printed and handwritten text on a paper substrate

Fig. 4. Overview of the defined regions-of-interest for the classes substrate, corroded, uncorroded, and print

Since for every pixel an absolute reflectance curve is available within the hyperspectral data cube, an average spectral curve can be calculated for each ROI (fig. 5). The SDS algorithm is subsequently used to measure for each pixel the similarity of its spectral content with each of the four ROI spectral curves. The result is a set of four so-called SDS *feature images* shown in figure 6. The pixel values in each image are a measure for the similarity of the pixel spectral curve with the average spectral curve of the corresponding ROI. A high pixel value (bright pixel) corresponds to a high similarity, a low pixel value (dark pixel) to a low similarity value.

Based on its four SDS values visualized in the feature images, each pixel has to be assigned to one of the four predefined classes. In order to do this, one has to define the so-called *decision boundaries*, which are mathematical conditions that are applied to the set of four SDS values of each pixel to determine to which class the pixel belongs. Instead of using the SDS values of the pixels in their spatial context, as described by the feature images, the mathematical conditions can be defined more easily if one regards each pixel as a certain point in four-dimensional (4D) mathematical space. The coordinates of the point on each of the four axes in this so-called *feature space* are given by the SDS values of each image pixel. While it is of course not possible to plot (and not even to truly imagine) a distribution of points in a 4D-space, one can select any two of the four coordinates to generate a so-called *scatter plot*. A scatter plot is a diagram, in which each pixel is plotted at a point whose x and y coordinate are given by the SDS values of the pixel in two selected feature images. In a mathematical sense, a 2D scatter plot is thus a particular projection of the pixel distribution in the 4D feature space.

Figure 7 shows the scatter plot of all pixels of the hyperspectral data cube for the two feature space axes that correspond to the corroded and uncorroded ink ROIs, respectively. Pixels that belong to one of the three ROIs that had been defined in the corroded, uncorroded and print ink area are

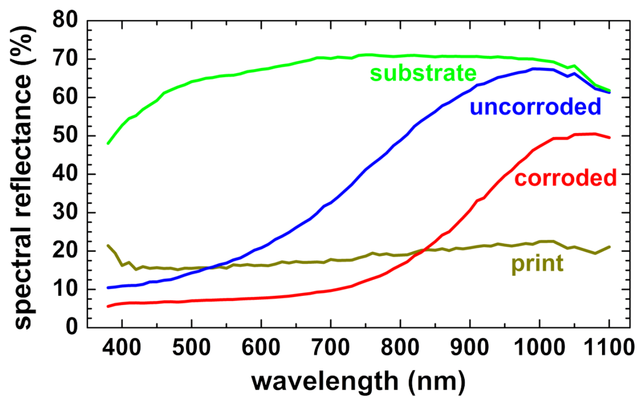


Fig. 5. Average spectral curves for each of the defined regions-of-interest

Fig. 6. Feature images calculated with the SDS algoritm: sub-strate (top left); print (top right); uncorroded ink (bottom left); and corroded ink (bottom right)

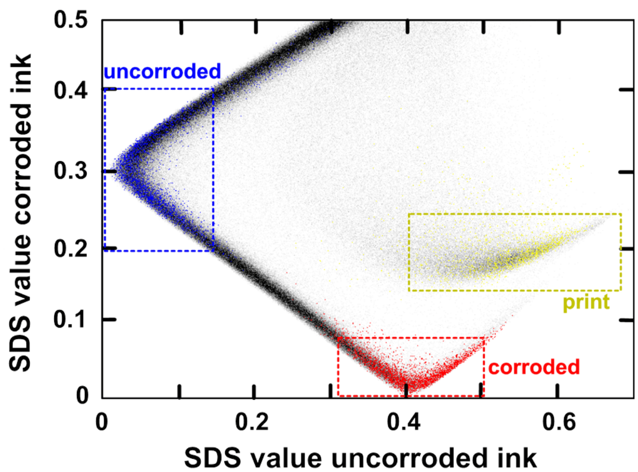


Fig. 7. A graph showing the distribution of two of the four SDS values for each pixel, together with the decision boundaries for the classification. Every pixel that falls outside the uncorroded, the corroded, or the print box is assigned to the class substrate.

Fig. 8. A color-coded graph showing the result of the classification process

Fig. 9. A real-color image of the map of New York (dated 1665), drawn by J. Vingboons. Part of the inventory of the Nationaal Archief

represented in the corresponding colors. Guided by the distributions of the colored ROI pixels in the scatter plot, three non-overlapping boxes are marked manually. Each box defines the decision boundaries for one of the ink classes. If due to its SDS values a pixel lies within such box, it is assigned to the corresponding ink class. If the pixel lies outside all three boxes, it is assigned to the substrate (background) class. The result of this classification process is depicted in figure 8. This image shows that ink corrosion is present in areas where the iron-gall ink is applied thickest.

DETECTION OF UNDERDRAWINGS BY UNSUPERVISED CLASSIFICATION

Besides the detection and mapping of deterioration effects, hyperspectral imaging can also be used for the detection of underdrawings (Kubik 2007). This example describes the results of using the described QHSI technique and post-processing algorithms to detect underdrawings in a historical map with a view of New York, drawn around 1665 by J. Vingboons in the Netherlands (fig. 9). This map is in possession of the Nationaal Archief (the National Archives of the Netherlands) in The Hague.

The historical map contains underdrawings that are visible with the naked eye in some places, but which cannot be discerned clearly in other areas. QHSI recordings of a small part of the map were made (fig. 10) in an effort to enhance the contrast between the underdrawings and the ink and pigments that are present. The resulting hyperspectral data cube was subjected to an unsupervised classification procedure.

In this classification procedure, the first step is to automatically extract the most important spectral information from the large number of images using a Principal Component Analysis (PCA) algorithm (Jolliffe 2002). This algorithm condenses the information from all spectral bands into only a few images. In this case the first five components were found to contain all relevant spectral data, thus resulting in a 5-dimensional feature space (the information is condensed to five values per pixel).

Subsequently, four classes were defined: substrate, ink, underdrawing, and road pigment (a brownish pigment used for coloring the road in front of the houses, see figure 10). The probabilities for each pixel to be a member of one of these classes were calculated using a Quadratic Maximum Likelihood (QML) classification algorithm (Hsieh and Landgrebe 1998). The result is visualized in a color-coded image that clearly shows the difference between underdrawings and ink drawings (fig. 11).

The false-color image shows that the contrast between the underdrawings (yellow) and the substrate with ink and pigments has increased drastically and that the underdrawings can now easily be discerned within the entire imaged part of the document.

LEFT TO RIGHT

Fig. 10. A real-color image of a recorded section of the map of New York where some of the underdrawings can be seen with the naked eye

Fig. 11. A false-color image of a recorded section of the map of New York, showing a strong contrast between the underdrawings (yellow), the substrate (blue), the ink (red), and the brownish pigment used in the road area (brown)

SUMMARY AND CONCLUSION

In summary, this article discusses the use of the quantitative hyperspectral imaging technique for the analysis of historical documents. Through advanced image processing algorithms a classification of the pixels in the recorded area is achieved. This can be used to map the distribution of different types of ink and of corroded and uncorroded areas for investigating degradation effects. As a second application, the use of the technique to enhance the visibility of hidden features such as underdrawings is demonstrated.

In conclusion, quantitative hyperspectral imaging is a very capable non-destructive analysis technique that can help to balance the needs of retrieving more contents information and preventing the decay of valuable historical documents. Current research at the Nationaal Archief and Art Innovation concentrates on further developing the hyperspectral imaging technique for investigation of aging and deterioration processes of historic documents.

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Letterpress Copying Books: Conservation and Preservation Implications

INTRODUCTION

Letterpress copying books are an early form of document copying process common from the turn of the nineteenth century and used through the 1950s. They are present in most archival collections in the Western world and contain a vast amount of information relating to the history of our culture. Many are quickly deteriorating to the point that records are being lost, and they often contain the only extant version of a document as the originals were routinely discarded or lost.

The historic copy press process involved the transfer of ink on a freshly written document to a moistened sheet of copying paper through the use of direct contact and pressure. Because the soluble copying ink was transferred directly, it left a mirror image print to be read from the verso of the thin paper. The process required inks and papers to have certain properties to be effective. It was necessary for the ink to remain wet for an extended period in order to achieve multiple high quality copies without causing unacceptable damage to the original. The paper had to be thin enough to read through the verso of the sheet, but also had to be strong enough to withstand the strains of being handled wet and pressed repeatedly. These parameters led to experimentation and the development of many different formulations of ink and papers (Cleveland 2000, Rhodes 1999). The addition of gums and sugars, for example, encouraged the ink to move from the original to the copying paper, while the lack of size and the translucence of the paper allowed the writing to be read from the verso of the leaf.

These unique materials display specific and unusual degradation characteristics, and their preservation needs are likewise unique. This study investigated the common conditions, conservation best practices, and storage issues related to copying books in archival collections.

METHODOLOGY

Methodology included a questionnaire submitted to professional list-serves, a survey of the copying books at the Center for American History at the University of Texas at Austin, and an exploratory conservation treatment, which included lining, mending, humidification and flattening, and aqueous techniques.

Two copying books were selected from the collection of the Center for American History for examination and treatment. The books exhibited a wide variety of problems and were chosen to represent the range of possible conditions of letterpress copying books. The Ballinger copying book from 1856 exhibited widespread letter drop-out from iron gall ink corrosion and severe distortion and tight creases from improper

LEFT TO RIGHT

Fig. 1. Ballinger Letterpress Copying Book. Before treatment. Ballinger Papers 1856, The Center for American History, University of Texas at Austin

Fig. 2. Fulton Letterpress Copying Book. Before treatment. Fulton Papers 1890–91, The Center for American History, University of Texas at Austin

Poster presented at the AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

Fig. 3–5. Ballinger Letterpress Copying Book. Existing housing condition (left); after treatment, new case (center); and after treatment, new custom enclosures (right). Ballinger Papers 1856, The Center for American History, University of Texas at Austin

storage of the textblock in a document case without the support of a binding, but the paper remained relatively flexible (fig. 1). The Fulton copying book dated 1890–91 exhibited severe embrittlement, darkening, foxing and reverse foxing, but the binding was in relatively good condition (fig. 2). Both books exhibited widespread fading, offsetting, and feathering inks. One section from each book was removed in order to treat the individual leaves. Surface cleaning was performed as needed with a Hake brush, soot sponges, and a microspatula. Local humidification was performed with a small sable brush, cotton swabs and de-ionized water to release tight creases and folds, as needed to allow legibility. Selected leaves were humidified overall in a humidity chamber and flattened between wool felts. A few leaves were washed in successive five-minute baths of re-calcified de-ionized water. Lining repairs were performed using lens tissue applied wet with 4:1 wheat starch paste, as well as with a variety of prepared tissues using the following adhesives: 2% Klucel G, Lascaux 498 HV, and 2:1 wheat starch paste / methyl cellulose. The Klucel G and Lascaux tissues were re-activated using ethanol, isopropanol, acetone, and heat. The re-moistenable tissue was re-activated with humidity. Several methods of application were performed for each type of lining. After treatment the bindings were stabilized, housed in custom tuxedo boxes, and returned to their existing manuscript boxes (figs. 3–5).

FINDINGS

The results of the questionnaire illustrated a great need for research into best practices for treatment, storage, and duplication of these materials. Of those that responded, 74% reported that records in their collections are currently losing information. There was great variety in current practices, highlighting a lack of standards, and many archivists are unclear about appropriate preservation plans for copying book collections. Through a relatively high response rate and many detailed comments, the respondents communicated a feeling of both urgency and enthusiasm for research in this area.

Overall humidification, aqueous treatments, and linings applied wet caused unacceptable damage to the manuscripts and should be avoided as possible when treating copying books. Fragile areas were torn, shattered, and lost when transferring the wet paper from one support to another and when removing the support after the manuscript was dry. Common supporting materials: Hollytex, Tyvek, Mylar, and silicone release paper; were all found to cause damage. Silicone-release Mylar was the only support that was not found to cause damage. Recent research supports the decision to avoid moisture when treating copying books that contain iron gall ink. Reissland (2000) found that paper deteriorated by ink corrosion is less hydrophilic than surrounding undamaged paper, causing stress on the inked areas during aqueous treatment, and Eusman and Mensch (2000) found that damaging water-soluble components of the ink can spread throughout the sheet causing further deterioration.

The copying paper, although sticky to the touch, resisted adhesion to the solvent-set lining tissues. Klucel G was inadequately adhered when re-activated with ethanol, isopropanol, and acetone. Lascaux 498 HV was more effective than Klucel G, but required special procedures to gain sufficient tack. It was found that the solvent evaporated before the lining was well adhered to the manuscript, so containing the solvent in a silicone release Mylar sandwich was attempted and found to be very effective. The Lascaux 498 HV-coated tengujo tissue was misted with ethanol and boned down with a Teflon folder while inside the silicone-release Mylar sandwich. The manuscript was left under weight until completely dry (fig. 6–10).

The copying paper also resisted adhesion to heat-set tissue. The Lascaux heat-set tissue adhered more adequately than the Klucel G when used with high heat and left under heavy weight until completely cool. Although this method does not provide the strongest lining possible, in some situations its benefits may outweigh its disadvantages. It is fast, easy, allows in-situ repair, and does not require moisture. Many copying books are in excellent condition except for a few leaves, which contain highly corrosive ink or were otherwise damaged. Because copying books often contain many different inks, the level of deterioration is often inconsistent within a single volume. For these books, performing in-situ

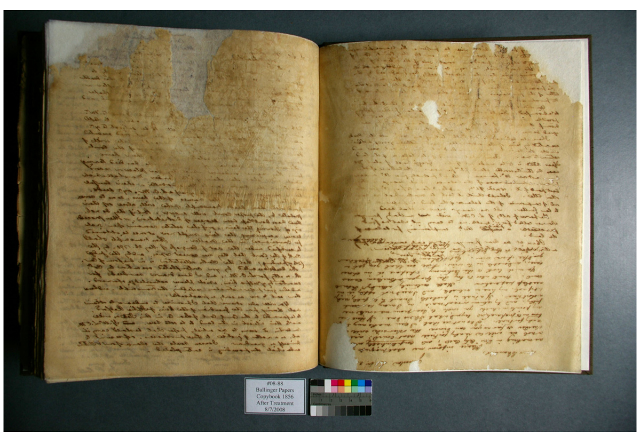
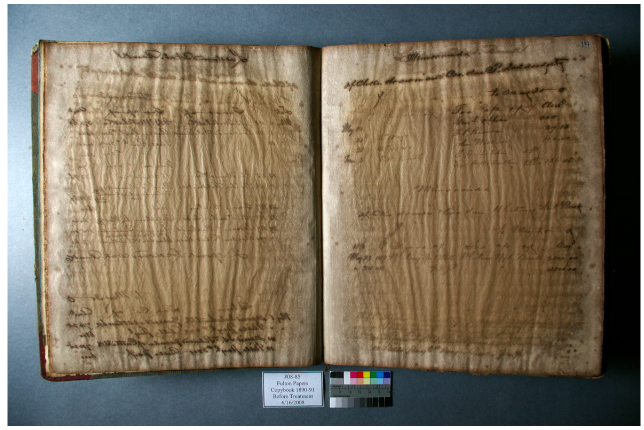


Fig. 6–8. Ballinger Letterpress Copying Book. During treatment (top); before treatment (center); and after treatment (bottom). Ballinger Papers 1856, The Center for American History, University of Texas at Austin

lining or mending with a very thin heat-set Lascaux tissue is preferable to dis-binding.

Although dis-binding and lining each leaf is a very invasive and time-consuming treatment, it may sometimes be the only viable option. Access is often restricted for severely embrittled copying books because they cannot be handled safely, and in some cases cannot be safely digitized. For these artifacts,



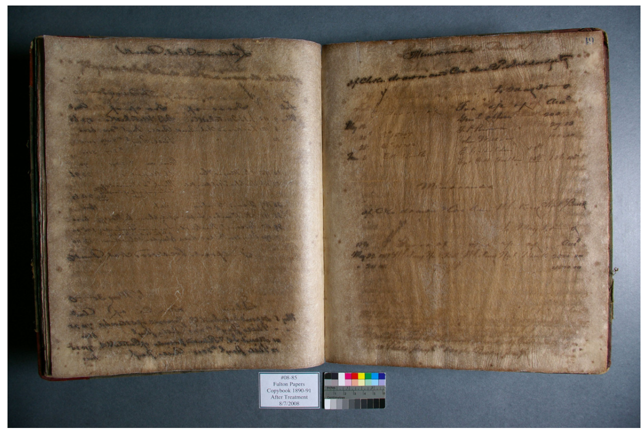


Fig. 9–10. Fulton Letterpress Copying Book. Before treatment (top) and after treatment (bottom). Fulton Papers 1890–91, The Center for American History, University of Texas at Austin

restoring access to the information may be a higher priority than retaining the character of the bindings. Unfortunately, the original tissue is so thin, that adding even extremely thin linings will significantly increase the thickness of the text-block, putting pressure on the joints of the binding.

CONCLUSION

The composition of the copying papers and inks creates special challenges for conservation treatment. The inclusion of sugars, glycerin, and other humectants causes the papers to become quite sticky and difficult to handle when wet, yet the papers do not readily adhere to solvent-set or heat-set lining tissues. The most effective treatment method found was lining with a Lascaux 498 HV-coated tengujo tissue misted with ethanol and boned down with a Teflon folder while inside a silicone-release Mylar sandwich. Because the materials are particularly sensitive to moisture, the use of enclosures and environmentally controlled storage conditions are particularly important preservation measures.

FURTHER RESEARCH

Further research will be conducted in an effort to characterize the materials on a chemical level, explore new methods for conservation treatment, and investigate imaging options. The study will be conducted at the Smithsonian Institution Archives with the support of the Smithsonian's Museum Conservation Institute and Office of Fellowships.

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ACKNOWLEDGEMENTS

Stephanie Malmros, Center for American History, University of Texas at Austin; Karen Pavelka, Kilgarlin Center, University of Texas at Austin; Chela Metzger, Kilgarlin Center, University of Texas at Austin; and The American Association of University Women.

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The Removal of Leather Dressing from Paper

ABSTRACT

This paper represents the preliminary findings of an ongoing study of techniques for the removal of leather dressing from paper. Four types of paper were selected for study, using three dressing formulae, and four aging protocols. Five immersion techniques for reduction and/or removal of leather dressing stains were compared, and their effectiveness measured using qualitative examination. The results were analyzed by type of dressing, age of stain, and kind of paper.

INTRODUCTION

Traditionally, the preservation of leather bound books included the regular application of "leather dressing," a mixture of fats, waxes, solvents, and other ingredients designed to keep the leather supple and resistant to moisture, insects, microbial attack, and environmental pollutants. This process of furbishing and conditioning the leather was typically undertaken in concert with wet or dry surface cleaning of the binding.

In recent years, however, conservators have become increasingly aware of the detrimental effects of leather dressing. While the routine dressing of leather bound books has fallen out of favor, many previously treated volumes are now suffering from waxy surface residue (bloom), formation of corrosion products around metal furniture, and perhaps most problematically, staining of the textblock. This staining occurs when the leather dressing penetrates through the leather and spine linings and is absorbed into the textblock, causing discoloration and damage to the paper. A second mechanism for transmission is via direct contact with dressed turn-ins, leading to staining of the outermost pages of the textblock. Over time, discoloration caused by oxidation of the oil impairs legibility, and gradual embrittlement of the stained paper puts the entire volume at risk.

Stains resulting from leather dressing are comprised of a mixture of ingredients. Some of these ingredients may be soluble in water, while others may only respond to organic solvents. Still other elements, such as waxes, may be insoluble in water and unaffected by treatments commonly used to reduce oil stains. These stains may also contain components of the leather covering that have been solubilized and carried along with the dressing into the textblock.

Treatment of these complex stains poses ethical, technical, and logistical problems. Staining typically affects the entire textblock, as oily elements of the dressing are wicked along the sewing supports and into the sewing thread. Because the gutter is usually the area most heavily affected, the book must often be disbound for treatment. Immersion treatments are generally selected for efficiency, but lack the level of control offered by a localized technique. While solvents are frequently used to reduce oil stains, they have the potential to cause problems such as softening, desiccation, and bleeding in oil-based printing inks. Given these concerns, it is important to select a technique that is effective enough to justify a highly invasive treatment without causing damage to the inks.

The goal of this study is to evaluate common immersion treatments for oily stains to determine which are most effective at reducing leather dressing stains without harming printing inks.

PAPER SELECTION

Four papers from the seventeenth to twentieth centuries were chosen for testing (fig. 1). Three of the papers were taken from German printed books, selected in order to test the effect of the various treatments on printing ink. The fourth paper tested was Whatman #1 filter paper.

The outer margins of each sampled book page were trimmed off to eliminate any possible differences in the character of the paper caused by prolonged exposure to air. The first and last pages of each book were rejected for the same reason. Areas with staining, tears, previous repairs, or any other detectable deviation from the normal character of the

Poster presented at the AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

Paper	Origin	Description	Observations	Optical microscopy of fibers	Thickness	pH	Biuret test for protein	Iodine / potassium iodide test for starch	Aluminon test for alum	Phloroglucinol test for ligin
1	*Luther Bible*—matches collation of 1670 and 1693 editions, pp. 55/56 and 941/942	17th century handmade, laid paper from a German Bible	Rattles like gelatin sized paper, yellowish color, opaque, fluoresces yellow under UV	Looks like cotton with a few shives or woody inclusions	.11 mm	5.0	Slight positive	Negative	Slight positive	Slight positive - localized in woody inclusions
2	*Sammlung ber besten Reisebeschreibungen* (Brunn, 1786), pp. 27-98	18th century handmade, laid paper	Slightly textured, little body, many visible inclusions, fairly opaque	Looks like cotton with shives or woody inclusions and blue and red fibers	.09mm	5.3	Negative	Negative	Negative	Slight positive - localized in woody inclusions
3	*Stunden der Undacht fur Befürderung wahren Christenthums und häuslicher Gottesverehrung* (Frankfurt, 1848), pp. 27–52	19th century machine made wove paper from a German book	Smooth, thin, regular, shorter fibers, opaque	Looks like cotton	.06 mm	4.3	Negative	Positive	Positive	Slight positive - localized in woody inclusions
4	N/A	Whatman #1 filter paper, known to be cotton	Fairly soft, somewhat textured	Cotton	.16mm	4.8	Negative	Negative	Negative	Negative

Fig. 1. Sample Papers

historic paper were removed from the sample population. Each historic sample included an area without printing, to facilitate measurements of color change and translucency, and an area with printed text. Some samples of the seventeenth-century paper also had areas of woodcut printing.

DRESSINGS TESTED

Three dressings were selected for testing based on their frequent mention in literature describing the application of leather dressing, and the range of ingredients and working properties they represent. They were numbered from earliest to latest publication and distribution, and perhaps not coincidentally, from least to most viscous—later formulae typically contained a far higher proportion of wax, possibly in response to an increased awareness of the potential damage caused by excessive application of oily, readily flowing dressings.

While the formulae of leather dressings varied widely, they generally contained some combination of liquid oils, solid fats, waxes, and various additives such as soaps, metal salts, and starches (fig. 2). Although animal fats such as neat's-foot oil and lanolin were most common, fish, vegetable, and mineral oils were also used. Solvents were sometimes added to thin dressing mixtures and to enhance penetration.

Formula #1
Lanolin (anhydrous)..........................7 oz
Cedarwood Oil..................................1 oz
Beeswax...1/2 oz
Hexanes..11 oz
(Plenderleith 1946)

Formula #2
Neat's-foot Oil...................................60%
Lanolin (anhydrous)..........................40%
(Rogers and Beebe 1956)

Formula #3 [1]
30 parts Neat's-foot Oil
20 parts Anhydrous Lanolin
10 parts Carnauba Wax

The dressings were applied with a brush to one side of each paper sample, and allowed to air dry for one week on a sheet of Mylar. It is important to note that this method of application does not replicate the wicking action most commonly implicated in this type of staining. Nevertheless, brush application was selected because it ensured that all components of each formula (including those that do not

Lubricants	Polishes/surface sealants	Solvents	Other Additives
Lanolin	Beeswax	Hexanes	Cedar oil
Mutton fat	Carnauba wax	Turpentine	Glycerin
Tallow	Paraffin	Trichloroethane	Castile soap
Butter	Japan wax	Diethyl ether	Salt
Egg yolk	Acrylic wax	Alcohol	Borax
Oil of egg	Microcrystaline wax	Milk	Imidazole
Neat's foot oil	Acrylic resins	Water	Sodium stearate
Sperm oil	Egg white		Lye
Cod oil	Blood albumen		Potassium lactate
Castor oil	Starch		Saddle soap
Linseed oil	Rosin		Shoe polish
Coconut oil	White glue		
Olive oil			
Mineral oil			
Vaseline			

Fig. 2. Some ingredients mentioned in recipes for leather dressings

flow readily at room temperature) were applied to each sample. One group of samples was left undressed as a control.

AGING PROTOCOL

The samples were broken into four groups for aging.

- *Unaged:* The unaged samples were treated after air-drying for 3 weeks.
- *Oven-aged:* The oven-aged samples were aged at 75°C and 65% RH for two weeks before treatment.
- *Naturally light-aged:* The naturally light-aged samples were hung in a south-facing window in the Thaw Conservation Center.
- *Naturally dark-aged:* The naturally dark-aged samples were placed on a sheet of Mylar and stored in a drawer in the Thaw Conservation Center.

Only the unaged and oven-aged samples were treated for this paper. Aging continues for the naturally aged samples.

TREATMENT PROTOCOL

In order to more closely simulate the conditions of treating an entire stained textblock, only immersion techniques were evaluated. The treatments were selected based on approaches used by book and paper conservators who had treated leather dressing stains. Three solvent-based treatments and two aqueous techniques were selected for testing. One group of samples was left untreated as a control.

Hexanes (15 minutes)

Hexanes were selected for their low polarity, and because they are a component of Formula #1 leather dressing. Each sample was washed in a single fifteen-minute bath, blotted lightly, and allowed to air dry on blotter.

Isopropanol (15 minutes)

Alcohols are among the solvents most frequently used by paper conservators. Isopropanol was selected over ethanol because it is available at a very high purity for substantially lower cost than absolute ethanol. Each sample was washed in a single fifteen-minute bath, blotted lightly, and allowed to air dry on blotter.

Acetone (15 minutes)

Acetone is a moderately polar solvent frequently used in laboratory settings as a degreaser. Each sample was washed in a single, fifteen-minute bath, blotted lightly, and allowed to air dry on blotter.

Lipase bath (300 units of activity/mL, 30°C for a total of 1 hour, plus 30-minute rinse)

Lipase is a water-soluble enzyme that catalyzes hydrolysis of water-insoluble lipids, breaking triglycerides into fatty acids and glycerol (Blüher et al 1997). The solution was buffered to a pH of approximately 7.5 using Trizma® Pre-set

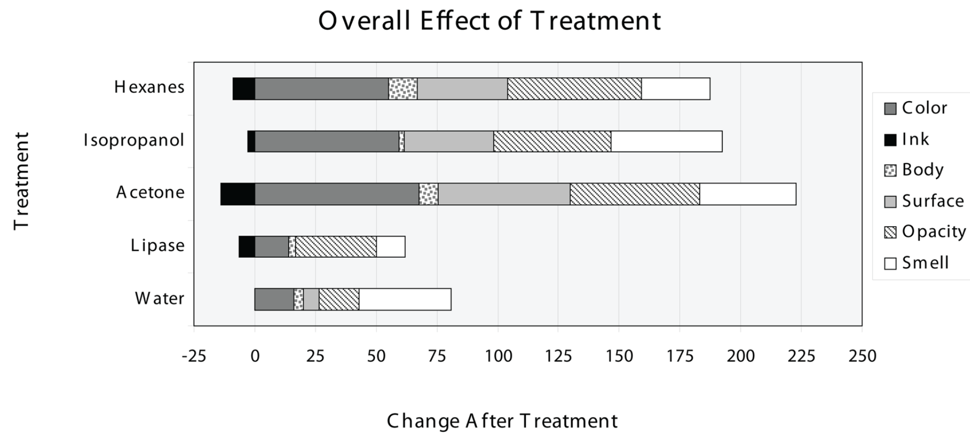

Fig. 3. The cumulative change in score on six different parameters of all treated samples. Each parameter was qualitatively evaluated before and after treatment, and a numerical score was assigned. The change in these scores after treatment is shown here. A positive score represents an improvement. A negative score represents undesirable change or damage to the paper or ink. All treatments except a water bath caused some change or damage to inks

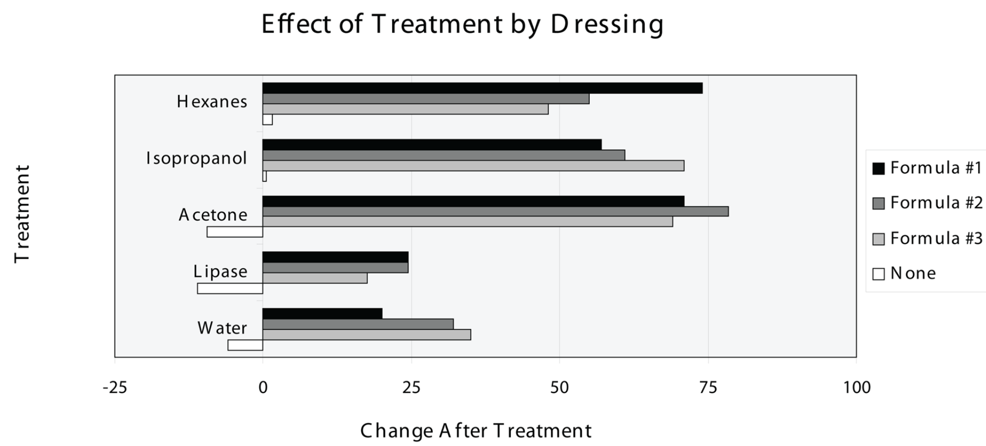

Fig. 4. The effectiveness of each treatment, broken down by type of leather dressing. A negative score represents undesirable change or damage to the paper or ink. The hexanes bath was notably more effective in treating samples dressed with Formula #1, which contains hexanes as a solvent. This increased effectiveness was most pronounced in the unaged samples

Crystals[2]. Each sample was washed in two successive thirty-minute baths, followed by a thirty-minute rinse. After treatment, the samples were blotted lightly and allowed to air dry on blotter.

Water (30°C for a total of 1 hour, in 3 baths)

A water bath was selected to compare the effectiveness of a lipase bath with that of warm water. The pH of each bath was adjusted to approximately 8 using calcium carbonate. The samples were washed in three successive baths. After treatment, the samples were blotted lightly and allowed to air dry on blotter.

ASSESSMENT

Each sample was evaluated before and after treatment, and assigned a score[3] for each of six parameters: color, strength of ink, body, surface feel, opacity, and smell. The change in score after treatment was calculated for each sample. A positive number indicated an improvement, and a negative number indicated an undesirable change or damage. In the case of the control samples that were not stained with leather dressing, any detectible change to the paper or ink resulting from treatment (such as softening, loss of sizing, or color change) was considered undesirable.

RESULTS

The data were analyzed to determine which treatments were most effective at reducing the undesirable characteristics of leather dressing stains. The findings were broken down by several variables to identify secondary trends, such as a treatment being particularly effective on one type of paper or dressing. The findings are summarized in figures 3–6.

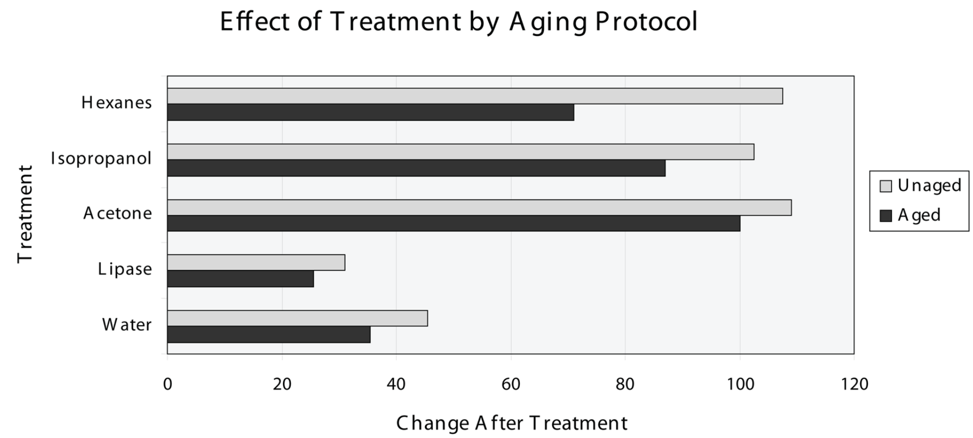

Fig. 5. The effectiveness of each treatment, broken down by whether or not the sample was aged. The unaged samples showed more improvement with all treatment protocols. The more polar solvents were most effective in treating the aged samples, where cross-linking was likely to have occurred

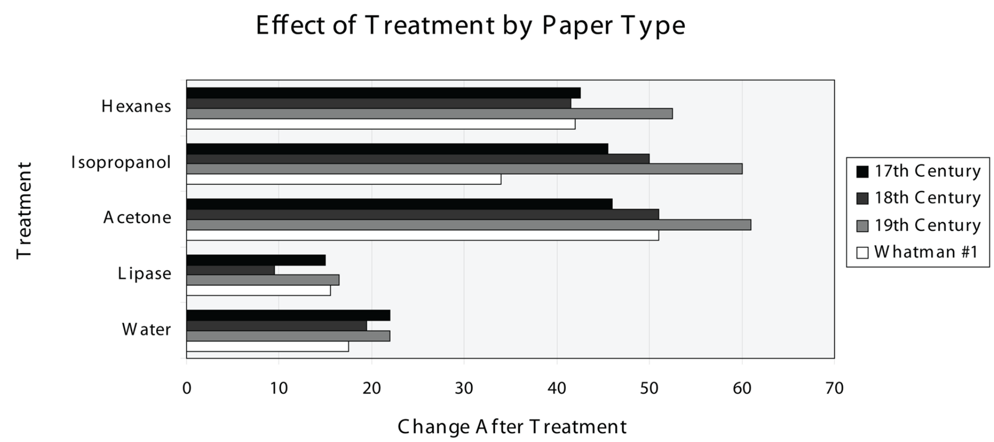

Fig. 6. The effectiveness of each treatment, broken down by the type of paper. The nineteenth-century paper showed the most improvement with all treatments, possibly because its smoothly sized surface made it less absorbent

Overall, acetone was found to be the most effective at reducing the discoloration and oily surface feel of the stained samples (fig. 3). Unfortunately, acetone was also most likely to damage the printing inks, including softening of the ink leading to offset, haloing, and the formation of a white bloom on the surface of the ink. This haze was most noticeable in the samples dressed with Formula #3, which contained the highest proportion of wax. Treatment may have resulted in selective removal of the oily components of the dressing, leaving the wax behind as a white film.

Isopropanol and hexanes were both moderately effective at reducing staining, with less risk of damage to the printing ink and paper. Hexanes were particularly effective at removing Formula #1, which contains hexanes. Isopropanol worked well overall, and was the least likely of all the treatments other than water to damage the printing inks.

The aqueous treatments were significantly less effective, particularly at improving the color, opacity, and surface feel of the samples. Aqueous samples were most likely to dramatically change the character of the paper, probably due to the removal of sizing in the bath. Warm water was effective at reducing the smell of the dressed samples, and was the only treatment that caused no damage to the printing ink. Lipase was slightly more effective than water at improving opacity, but was least effective at reducing discoloration, smell, and oily surface feel. The samples treated with lipase tended to have a yellow cast and a blotchy appearance after treatment.

Variables such as the dressing type (fig. 4), age of stain (fig. 5), and type of paper (fig. 6) were all shown to have some effect on the outcome of treatment. A different treatment was most effective on each leather dressing formula, but acetone and isopropanol were reasonably effective at reducing all three. Knowing the type of dressing used might give the conservator a slight advantage in selecting a treatment protocol, but the same advantage could be gained through spot testing.

No one dressing was markedly harder or easier to remove, although the waxy component of Formula #3 did not seem to be removed by any treatment. All treatments

were more effective on unaged samples, but the difference was most pronounced in the hexanes bath. The cross linking that occurs with aging may have made the oils less soluble in non-polar solvents.

While different types of paper showed trends in terms of the overall effectiveness of treatment, a given treatment was not notably more effective on a specific type of paper within the sample set. The nineteenth-century paper showed the most improvement with all treatments, possibly because it was less absorbent than the other papers tested. Conversely, the highly absorbent Whatman filter paper generally showed the least improvement. The ink on the nineteenth-century paper was the most vulnerable to damage.

CONCLUSIONS AND RECOMMENDATIONS

None of the treatments completely removed the stains; however solvent treatments generally produced good results. Thorough testing of the ink and paper is necessary to determine the most appropriate balance of stain reduction and protection of the original character of the object. Spot testing for the formation of waxy bloom should also be carried out before immersion. In general, isopropanol provided the best compromise between effective treatment and minimal damage.

It is important to note that solvent baths, while efficient and effective, require large volumes of solvent. Access to a fume hood or alternate source of ventilation is essential, as is personal protective gear. Because of the cost and environmental impact of organic solvents, care should be taken to minimize the amount of solvent wasted.

While lipase was not found to be effective in this study, it is possible that longer immersion, higher concentration, or other refinements in technique could increase its effectiveness. Poulticing with lipase in an agarose gel has been shown to be effective in reducing oil stains in works of art on paper (Stockman n.d.).

Successive solvent baths, or solvent baths followed by aqueous treatment were not tested as part of this study but could potentially provide greater improvement.

While this study investigated a range of dressing formulae, paper and ink compositions, and treatment approaches, every object and every treatment is different. Treatments that were not found to be effective may prove ideal for certain applications, and treatments that were relatively safe for the papers and inks used in this study may cause damage to other artifacts. In some cases, forgoing treatment may be the best option.

FUTURE WORK

The next phase of this investigation will examine the effectiveness of each treatment on naturally aged samples. The questions of which components of leather dressing are wicked into the paper, and whether they carry any components of the leather itself along with them, will also be explored.

ACKNOWLEDGEMENTS

The Samuel H. Kress Foundation; the staff of the Thaw Conservation Center, The Morgan Library & Museum; FAIC; Margaret Holben Ellis, Eugene Thaw Professor of Paper Conservation, and Hannelore Roemich, Acting Chairman and Associate Professor of Conservation Science, The Conservation Center, The Institute of Fine Arts, New York University; Yana van Dyke, Associate Paper Conservator, The Sherman Fairchild Center for Works on Paper and Photograph Conservation, The Metropolitan Museum of Art; Denise Stockman, Assistant Conservator for Paper, Barbara Goldsmith Preservation Division, New York Public Library.

NOTES

1. Clarkson, Christopher. Conservator of Library & Archive Materials. Personal correspondence. 22 February 2009.
2. Sigma-Aldrich # T8068. The exact pH of the buffer is dependent upon the temperature of the solution. The pH ranges from 7.7 at a temperature of 25°C to 7.4 at 37°C. Testing of the treatment solution with pH strips showed the pH to be approximately 7.5 at 30°C.
3. Each qualitative parameter was scored based on a pre-determined scale. Color and surface feel were evaluated using thirteen-point scales based on the paper samples in *The Print Council of America's Paper Sample Book* (Lunning and Perkinson 1996). The scales ranged from "very bright white" to "brown" (4) for color, and "very smooth" to "rough" (2) for surface feel. Because many samples had substantial leather dressing residue on their surfaces, the surface feel scale was extended to include scores for slight, moderate, or extreme oiliness, waxiness, and/or stickiness. Ink quality was rated on a six-point scale from "very strong" to "very weak." Embrittlement, smell, and opacity were rated based on six-point scales ranging from "supple" to "very brittle," "no odor" to "very strong odor," and "opaque" to "very translucent," respectively.

The scores shown in the charts reflect the change after treatment. For example, if a sample was "brown" (4) before treatment and "brown" (1) after treatment, the score would be 3. A positive score reflects an improvement, whereas a negative score reflects damage or undesirable change.

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A Cut Above: The Crayola Cutter as Conservation Tool

INTRODUCTION

The Crayola Cutter, although marketed as a children's toy, has great potential as a conservation tool. It consists of a stylus with a pulsating needle at the tip and a hard foam mat for cutting. Using the Crayola Cutter to draw a perforated line makes it easy to cut out complex shapes from a sheet of paper. It can achieve results ranging from a soft, feathered edge for a hinge to a precisely crafted fill or inlay, depending on the type of paper and the speed of the drawing motion.

The Crayola Cutter was compared to more traditional tools for creating fills, inlays, and hinges (e.g., scalpel, needle, water and brush). A variety of shapes and sizes were cut from various thicknesses of both western and Japanese papers. Factors considered in the comparison included the ability of the tool to achieve a desired result and the time necessary to do so. The Crayola Cutter was then used in three case studies: making an inlay for a drawing, creating a fill for a complex loss in an etching, and making hinges.

The following is a summary of the findings of these tests and demonstrates how the Crayola Cutter is an effective new tool for paper conservators.

Fig. 1. The Crayola Cutter

Poster presented at the AIC 37th Annual Meeting, May 20–23, 2009, Los Angeles, California.

COMPARISON WITH CURRENT TECHNIQUES:

How does the Crayola Cutter measure up to other current practices? The Crayola Cutter was tested against three common tools currently employed by conservators: scalpel, brush and water, and needle. All methods were evaluated for their speed, ease of use, effectiveness on various paper thicknesses, and ability to feather edges. The results are organized in table 1.

CASE STUDIES

1. Inlay

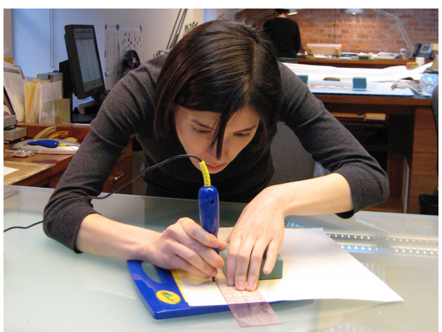
The time it takes to create an inlay is significantly decreased by using the Crayola Cutter instead of another tool to cut and feather an inlay paper. First, the outline of a drawing is traced onto the inlay paper. Next, the Crayola Cutter is used to follow the traced line to create a perforated outline. The perforated line can easily be pulled apart to release the inlay. Moving the stylus at a fast, consistent speed creates wider perforations and more feathering. See figures 2–5.

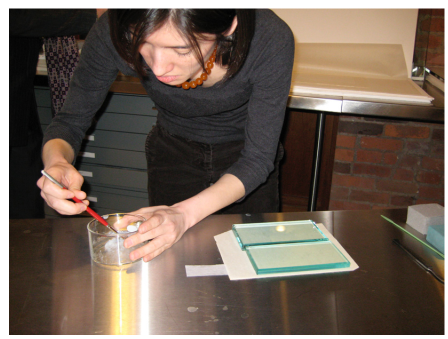
2. Fills

The Crayola Cutter can be used to create a variety of fills for losses ranging from simple to complex in works on paper. In this case, the precision of the Crayola Cutter allowed a fill to be cut quickly and easily for a small, complex loss. See figures 6–11.

Technique	Advantages	Disadvantages	Notes
Crayola™ Cutter	•Can cut complex shapes accurately •Fast •Good for most paper types •Creates good feathered edges on thin papers	•Cannot use on light table •Minor feathering on many papers	•Wetting perforation allows for more feathering
Needle	•Can cut complex shapes by perforating •Fast when scoring thin papers •Can create feathered edges when scoring thin papers •Can use on light table	•Very slow when perforating •Does not create an even feathered edge	•Can be difficult to create fine edges when perforating •Wetting perforation allows for better feathering
Brush and Water	•Can create feathered edges on all paper types •Fast when making straight lines •Can use on a light table	•Difficult and slow to cut complex shapes accurately	•Best on thin papers
Scalpel	•Can cut complex shapes accurately •Fast when cutting straight lines •Can use on light table •Can be used on all paper types	•Slow if attempting complex shapes •No feathering	

Table 1.





ABOVE, LEFT TO RIGHT

Fig. 2. Following the traced line with the Crayola Cutter to create a perforated outline of the drawing

Fig. 3. Pulling the perforated line apart to release the inlay

Fig. 4. Pasting up the edge of the drawing with wheat starch paste before attaching it to the inlay

LEFT

Fig. 5. The drawing adhered to the inlay

Fig. 6. An etching with an irregularly shaped loss in the upper right quadrant

Fig. 7. A detail of the loss, recto. A template, made by tracing the loss onto polyester film, was used to make an outline of the fill to be cut from a thin western laid paper

Fig. 8. Following the pencil line with the Crayola Cutter to create a perforated outline

Fig. 9. The fill after it has been pulled away from the rest of its sheet

Fig. 10. The fill adhered to the print with wheat starch paste

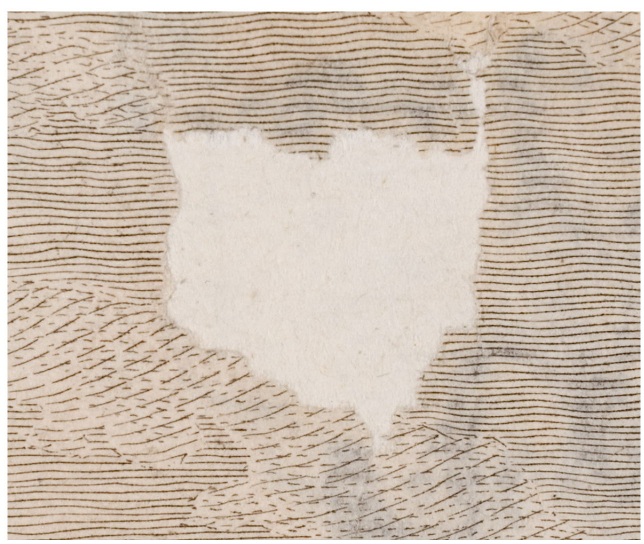


Fig. 11. A detail of the fill, recto. The edges of the fill were pared down and feathered further using water and a needle

Fig. 12. Using the Crayola™ Cutter to create a series of perforated lines on a medium-weight Japanese paper to be used as detachable hinges

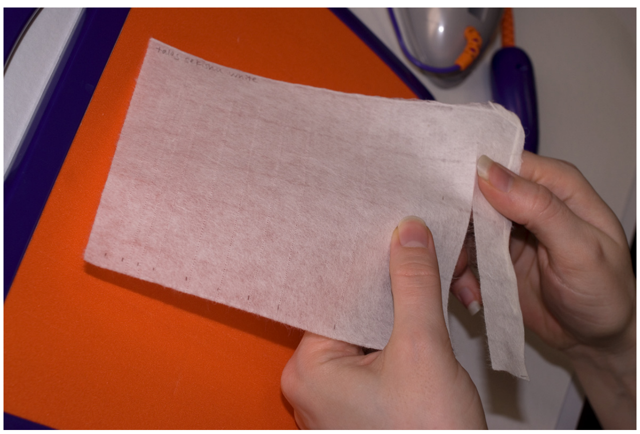


Fig. 13. Pulling one of the perforated lines apart

3. Hinges

Hinges made from a variety of papers can be created quickly using the Crayola Cutter. Both feathered and non-feathered edges are achievable depending on the speed at which the stylus is drawn across the sheet. See figures 12–13.

TIPS

1. If a larger cutting surface is required, a sheet of Volara foam over a hard surface works well as a substitute. The Crayola Cutter does not perform as well directly on hard surfaces.
2. The space between perforations depends on the speed of the drawing motion; wider perforations are achieved by moving the stylus faster.
3. At least two models of the Crayola Cutter exist. There are minor operating differences; the needle in the "new and improved" (purple and orange) version pulsates more quickly than in the original version (blue and yellow).
4. Depending on the project, using the Crayola Cutter in conjunction with other methods may be beneficial. For example, once a shape is cut, a wet brush helps to pull the shape apart from the paper and a needle may be used to increase feathering as desired.

ACKNOWLEDGEMENTS

Margaret Holben Ellis; New York University; The Conservation Center, Institute of Fine Arts, New York University; The Morgan Library & Museum; and The Foundation of the American Institute for Conservation

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Effect of Aqueous Treatments on Nineteenth-Century Iron-Gall-Ink Documents: Calcium Phytate Treatment—Optimization of Existing Protocols

ABSTRACT

Research results presented in this study were obtained in a project carried out at the Gatineau Preservation Centre, Library and Archives Canada (LAC) in partnership with the Canadian Conservation Institute (CCI) in Ottawa, Canada. This project uses nine original iron gall ink documents and compares various treatments with the combination of calcium phytate and calcium bicarbonate. The aim of this study was to identify visual changes, determine immediate side effects, and assess the effectiveness of delaying ink corrosion. Best results, which include an effective delay of ink corrosion and an alkaline reserve deposit, were achieved by immersing the naturally aged samples with a combined calcium phytate/calcium bicarbonate treatment. The significance of the findings is discussed.

INTRODUCTION

Thirteen years after the introduction of the aqueous calcium phytate treatment for ink-corroded materials proposed by the Instituut Collectie Nederland (ICN), this method is applied increasingly in paper conservation laboratories all over the world. For years paper and book conservators have been considering the recently developed treatments using calcium or magnesium phytate solutions with or without ethanol in addition to an alkaline reserve. Part 1 of this research includes: aqueous treatments; testing for solubility of corroded inks; measuring water absorption of the papers; measuring surface pH of inked areas; measuring cold water extraction pH; microchemical testing for detention of iron (II) ions with a bathophenanthroline paper indicator developed by Dr. Han Neevel (Reissland and de Groot 1999; Neevel and Reissland 2001; Eusman 2002); using a calibrated color chart developed by the Canadian Conservation Institute (CCI) (Tse et al. 2006); and evaluating the side effects (under magnification, daylight, and ultraviolet fluorescence illuminations) using instrumental analyses and evaluation by test panels of conservators. Part 2 investigates the changes that occur in selected treated samples following artificial aging by exposure to heat, light, and humidity (Tse et al. 2006; Tse et al. 2010). These tests were carried out to address concerns regarding the sensitivity of media treated with phytate (Neevel and Reissland 1997), and the possibility of fungal growth when exposed to a high humidity environment (Homolka 2001).

Independent submission. Paper presented at 2008 AIC meeting.

EXPERIMENTAL

This research focused on the comparative effectiveness of currently used and newly developed aqueous treatments.[1]

Samples

Nine original Canadian documents dating from the mid-nineteenth century (c. 1841–1875) were washed with eighteen separate aqueous treatments. The naturally aged ledgers were provided by CCI from their sample collection. The samples had different ink compositions and paper substrates and were selected for the study even though they were in relatively good condition overall, at the initial stages of ink corrosion (table 1). These documents were assessed to be in ICN condition rating 1 (Reissland and Hofenk de Graaff 2000).

The ledgers were cut into strips of various dimensions and one strip of each document was left untreated as a control sample. Some sets of samples did not receive all the proposed treatments due to the amount of sample material available.

Treatments Protocols

The individual sequences of wash combinations used are summarized in table 2.1 and 2.2. Samples were treated separately. Consequently, the solutions were individually prepared and performed without drying the samples in between. To ensure a standardized washing procedure, the nine sample sets and each aqueous treatment option were treated consistently (table 2.2 and fig. 1). Samples in this study were not

Samples	Substrates	Media
1	1856 Cream wove paper; cotton rag; machine made; and with ruled lines	Dark brown ink ICN rating: 1 CCI Fe^{2+}: 50+ ;Fe^{3+}:50+
2	1849 Grayish cream wove paper; cotton rag; machine made; and with ruled lines	Light brown ink ICN rating: 1 CCI Fe^{2+}: 10; Fe^{3+}:50++
3	1864 Blue laid paper; cotton rag; hand made; chain and laid lines; and w/ ruled lines	Light brown ink ICN rating: 1 CCI Fe^{2+}: ~25; $Fe3^{+}$: ~25
4	1865 Blue ledger; laid cotton rag paper; hand made and with chain and laid lines	Light brown ink ICN rating: 1 CCI Fe^{2+}: ~25; $Fe3^{+}$: ~25
5	1841 Cream wove paper; cotton rag; machine made; and with ruled lines	Light brown ink ICN rating: 1 CCI Fe^{2+}: ~25; $Fe3^{+}$: ~25
6	1846 Cream wove paper; cotton rag; machine made; and with no ruling lines	Medium brown ink ICN rating: 1 CCI Fe^{2+}: 50; Fe^{3+}:50++
7	1846 Cream wove paper; cotton rag; machine made; and with no lines	Medium brown ink ICN rating: 1 CCI Fe2+: ~25; Fe3+: ~25
8	1875 Cream wove paper; cotton rag; machine made; and with thin blue lines	Light brown ink ICN rating: 1 CCI Fe2+: ~25; Fe3+: ~25
9	1846 Green wove paper; cotton rag; machine made and with no lines	Light brown ink ICN rating: 1 CCI Fe2+: ~10; Fe3+:~10

Table 1. Description of treated sample sets

	Treatment Sequences & Duration
1	Untreated control
2	Pure reverse osmosis (RO) water: pH 6.4; 20 min
3	Alkaline wash: pH 8.5 $Ca(OH)_2$; 20 min
4	RO + 0.011M $Ca(HCO_3)_2$: pH 6; 20 min each
5	RO + 0.086M $Mg(HCO_3)_2$: pH 7.3; 20 min each
6	RO + Alkaline wash: pH 8.5 $Ca(OH)_2$; 20 min each
7	Alkaline water simmer: pH 8.3 $Ca(OH)_2$; 40°C; 15min
8	Alkaline water simmer: pH 8.3 $Ca(OH)_2$; 90°C; 15min
9	Ethanol (EtOH) immersion, 15 min + alkaline water simmer: pH 8.3 $Ca(OH)_2$; 40°C; 15 min each + sprayed EtOH
10	EtOH immersion, 15 min + alkaline water simmer: pH 8.3 $Ca(OH)_2$; 90°C; 15 min each + sprayed EtOH
11	100% EtOH immersion; 20 min (used 95% Ethyl alcohol, denatured)
12	3:1 EtOH:RO; 20 min
13	1:1 EtOH:RO; 20 min
14	1:3 EtOH:RO; 20 min
15	Pre-wet with EtOH spray; calcium phytate: pH 5 (Ca-phy) + 0.011M $Ca(HCO_3)_2$: pH 6; 20 min each
16	Ca-phy: pH5 + 0.011M $Ca(HCO_3)_2$: pH6; 20min each
17	Ca-phy: pH 5 diluted 1:1:1 with RO: pH 5.5 and EtOH + 0.011M $Ca(HCO_3)_2$: pH 6; 20 min each
18	Pre-wet with EtOH spray; Ca-phy (20 min) + water rinse: pH 5.5 (3x 10 min) + $Ca(HCO_3)_2$ (20 min)
19	Ca-phy:pH 5 (20 min) + water rinse: pH5.5 (3x 10 min) + 0.011M $Ca(HCO_3)_2$ (20 min)

Table 2.1. Aqueous treatments used

resized with gelatin after washing as recommended by Gessa Kolbe for treated ink-corroded archival materials (2004).

Testing methods

Samples were tested extensively before treatment for media solubility in distilled water and in mixtures of ethanol and water (3:1, 1:1, and 1:3). Preliminary testing was carried out under magnification at 10X and 16X using a Zeiss microscope, Stemi SVII and with a thin sable brush (#000) dipped in the reagent, applied to heavily inked areas for thirty seconds and the surface observed for bleeding with a blotter. The water absorption of the nine samples was estimated by measuring the time the substrates absorbed a distilled water drop and then identifying the presence of sizing agents. Bathophenanthroline iron (II) ions test was performed on each document tested to assess the presence or recurrence of Fe^{2+} or Fe^{3+} ions before and after aqueous treatments (fig. 1). The non-bleeding indicator papers were examined and compared using the calibrated color chart developed at the CCI (table 1: media) and described the colors as 1: detectable; 10: weakly positive; 25: positive; and 50+: strongly positive. In the presence of Fe^{2+}, the test strips turned a deep magenta, as there were observed upon application to untreated and treated inked areas. The study was carried out in May through July of 2002.

Surface pH measurements of the overall population (162 samples) were peformed with a Beckman 60 pH Meter equipped with Beckman refillable combination (flat bulb, epoxy, AgCl, 12 x 155 mm electrode) in December 2004 (figs. 2–3,5). pH Measurements with cold water extraction were determined by using a glass microelectrode (Microelectrode Inc.) and recorded in June 2006 (figs. 4, 6).

Non-destructive instrumental analyses were carried out by Dr. Joseph Weber and the author at the Winterthur Museum

Table 2. **Different Washing Treatments Used**

No.	Treatments	Pre-wetting by spraying	Immersion 1	Immersion 2	Immersion 3	Pre-drying on suction table
1.	No treatment (control sample)	-	-	-	-	-
2.	Pure RO water pH6.4	-	**RO** (20 min, 20°C)	-	-	-
3.	Alkaline water pH8.5	-	**RO+Ca(OH)$_2$** (20 min, 20°C)	-	-	-
4.	RO + Ca bicarbonate pH6	-	**RO** (20 min, 20°C)	**.01M Ca(HCO$_3$)$_2$** (20 min, 20°C)	-	-
5.	RO + Mg bicarbonate pH7.3	-	**RO** (20 min, 20°C)	**.064M Mg(HCO$_3$)$_2$** (20 min, 20°C)	-	-
6.	RO + alkaline water pH8.5	-	**RO** (20 min, 20°C)	**RO+Ca(OH)$_2$** (20 min, 20°C)	-	-
7.	Hot alkaline water pH8.3	-	**RO+Ca(OH)$_2$** (15 min, 40°C)	-	-	-
8.	Hot alkaline water pH8.3	-	**RO+Ca(OH)$_2$** (15 min, 90°C)	-	-	-
9.	Alcohol + Hot alk. w. pH8.3	-	**100% EtOH** (15 min, 20°C)	**RO+Ca(OH)$_2$** (15 min, 40°C)	-	**100% EtOH**
10.	Alcohol + Hot alk. w. pH8.3	-	**100% EtOH** (15 min, 20°C)	**RO+Ca(OH)$_2$** (15 min, 90°C)	-	**100% EtOH**
11.	Alcohol	-	**100% EtOH** (20 min, 20°C)	-	-	-
12.	Alcohol/water mixture	-	**3:1 EtOH/RO** (20 min, 20°C)	-	-	-
13.	Alcohol/water mixture	-	**1:1 EtOH/RO** (20 min, 20°C)	-	-	-
14.	Alcohol/water mixture	-	**1:3 EtOH/RO** (20 min, 20°C)	-	-	-
15.	Phytate + deacidification	**100% EtOH**	**Ca Phytate pH5** (20 min, 20°C)	**.01M Ca(HCO$_3$)$_2$ pH6** (20 min, 20°C)	-	-
16.	Phytate + deacidification	-	**Ca Phy pH5** (20 min, 20°C)	**.01M Ca(HCO$_3$)$_2$ pH6** (20 min, 20°C)	-	-
17.	Phytate + deacidification	-	**1:1:1 Ca Ph/RO/EtOH** (20 min, 20°C)	**.01M Ca(HCO$_3$)$_2$ pH6** (20 min, 20°C)	-	-
18.	Phytate + deacidification	**100% EtOH**	**Ca Phy pH5** (20 min, 20°C)	**RO pH5.5 X3** (10 min each rinse, 20°C)	**.01M Ca(HCO$_3$)$_2$ pH6** (20 min, 20°C)	-
19.	Phytate + deacidification	-	**Ca Phy pH5** (20 min, 20°C)	**RO pH5.5 X3** (10 min each rinse, 20°C)	**.01M Ca(HCO$_3$)$_2$ pH6** (20 min, 20°C)	-

Table 2.2. Individual sequences of wash combinations used in the study

Treatments	Sample Set 1	Sample Set 2	Sample Set 3	Sample Set 4	Sample Set 5	Sample Set 6	Sample Set 7	Sample Set 8	Sample Set 9
1. Control Fe (II) ions									
Control Fe (III) ions									
2. RO water									
3. Alkaline water									
4. RO+Ca (HCO3)2									
5. RO+Mg (HCO3)2									
6. RO+Alkaline w.									
7. Alkaline w. 40°C									
8. Alkaline w. 90°C									
9. EtOH+Alk.w. 40°C	N/A							N/A	
10. EtOH+Alk.w. 90°C	N/A							N/A	
11. 100% EtOH									
12. 3:1 EtOH/RO w.									
13. 1:1 EtOH/RO w.									
14. 1:3 EtOH/RO w.									
15. EtOH + Ca Phy + Ca (HCO3)2									
16. Ca P.+Ca (HCO3)2									
17. 1:1:1 Ca P./RO/EtOH + Ca (HCO3)2									
18. EtOH+Ca P.+RO3X + Ca (HCO3)2									
19. Ca Phy + RO 3X + Ca (HCO3)2	N/A		N/A		N/A	N/A		N/A	

Fig. 1. Bathophenanthroline Test: Corrosive Ferrous (Fe^{2+}) and Ferric (Fe^{3+}) Ions

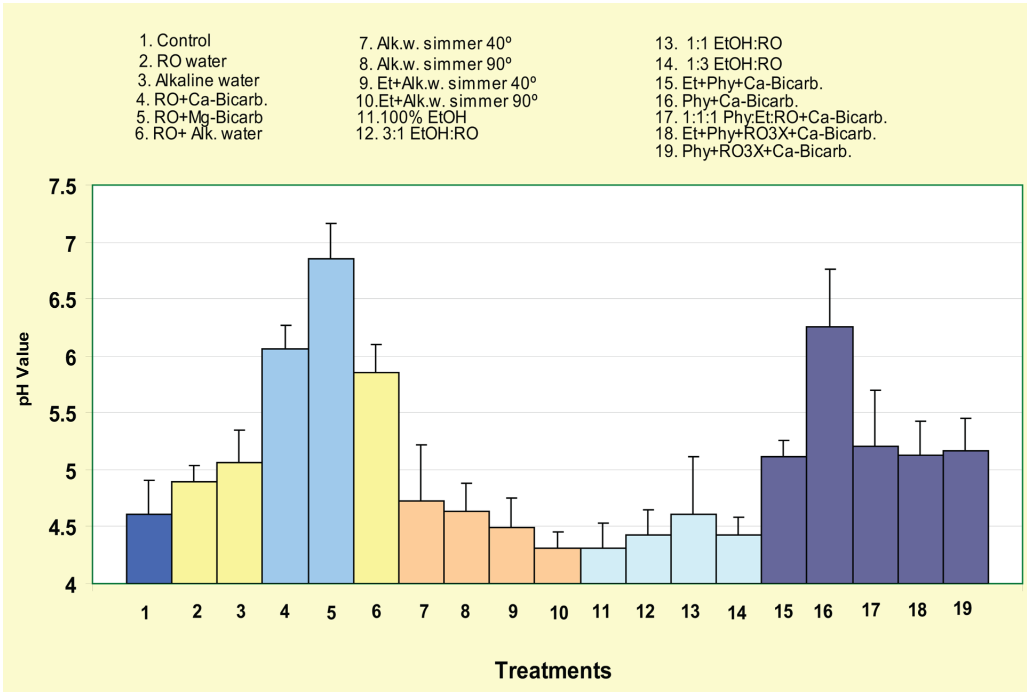

Fig. 2. Error bars represent the population standard deviation from the pH averages of each of the 18 aqueous treatments used and the untreated control samples recorded with surface pH measurements.
Part 1 - pH of 9 Iron Gall Ink Paper Samples Standard Deviations (recorded with a surface electrode)

to identify the inorganic components of inks and papers used in this study. Energy dispersive x-ray fluorescence spectroscopy (ED-XRF) with an ArtTAX ìXRF spectrometer. X-ray fluorescence consistently established the presence of iron as the only major element. The presence of gelatin size in the samples was determined by Season Tse at the Canadian Conservation Institute using Fourier transform infrared spectroscopy-attenuated total reflection (FTIR-ATR) with a Travel IR ATR-IR spectrometer (SensIR Technologies, Smiths Detection) (table 3).

Evaluation of Side Effects

Visual assessment was conducted of the entire population of samples under magnification, daylight, and UV-fluorescence to identify color changes, dissolution of ink compounds, crystal formations, and immediate side effects after washing. Visual side effects of media and substrates were recorded in daylight and UV-fluorescence photography using a digital camera, (Olympus E-10, 4,0 megapixel).

SUMMARY OF RESULTS

Aqueous Washing at Room Temperature

Most papers washed with aqueous treatments at 20° C looked lighter than the untreated control samples. All aqueous treatments removed the thin, blue, ruling lines present

Samples	Substrates	Media
1. 6.28.1856	FTIR-ATR: gelatin µXRF-paper: Fe, Ca, K, Cl, S	µXRF-ink: Fe, K
2. 12.13.1849	FTIR-ATR: no gelatin µXRF-paper: Fe, Zn, Cu, Ca, Mn, K	µXRF-ink: Fe, K, Ca
3. 7.6.1864	FTIR-ATR: gelatin µXRF-paper: Ca, S, Fe, K, Cu	µXRF-ink: Fe, Ca, S
4. 12.31.1865	FTIR-ATR: gelatin µXRF-paper: Ca, Fe, Mn, Cu, Zn, S, Cl	µXRF-ink: Fe, Zn, Cu, K, S
5. 6.8.1841	FTIR-ATR: no gelatin µXRF-paper: Fe, Ca, K, Mn, Cl	µXRF-ink: Fe, K, Cu, Ca, Zn
6. 10.5.1846	FTIR-ATR: gelatin µXRF-paper: K, Ca, Cu, Fe, Co, S	µXRF-ink: Fe, K, Ca
7. 10.6.1846	FTIR-ATR: gelatin µXRF-paper: K, Co, Cu, Fe, Ca, Ni, S	µXRF-ink: Fe, Co, K, Cu, Zn
8. 3.22.1875	FTIR-ATR: no gelatin µXRF-paper: Fe, Ca, K, Cl, Ti	µXRF-ink: Fe, K, Ca
9. 4.5.1846	FTIR-ATR: no gelatin µXRF-paper: Fe, Ca, K, Cl, Ti, S	µXRF-ink: Fe, Cu, Mn, Ca, Cl, K

Table 3. Instrumental Analyses: FTIR and XRF

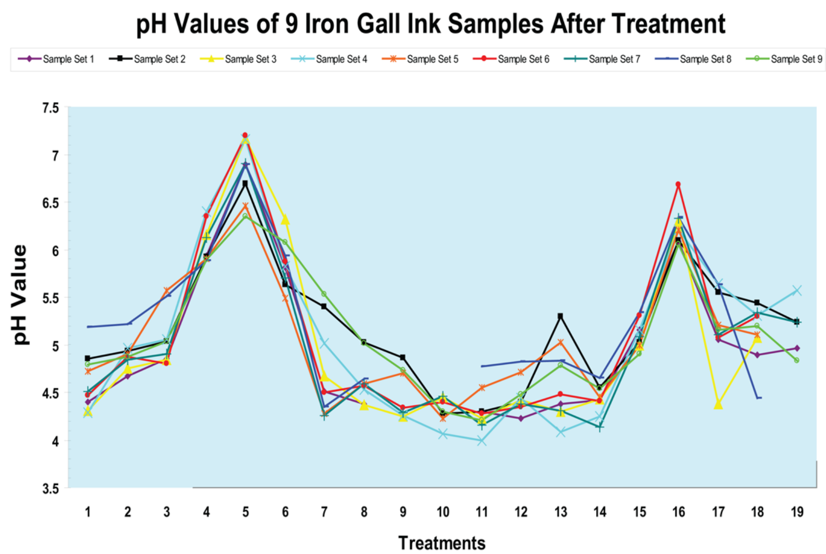

Fig. 3. pH Measurements—Surface Electrode Recorded in Dec. 2004

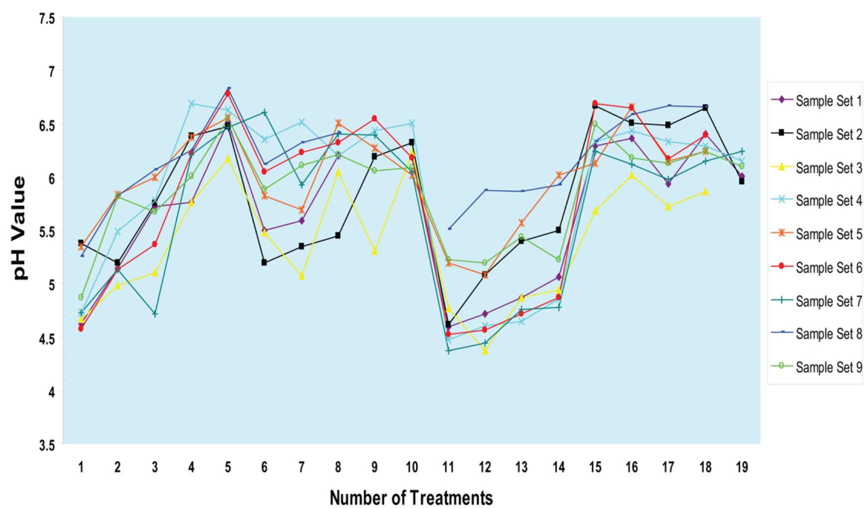

Fig. 4. pH Measurements—Cold Water Extraction Recorded in June 2006

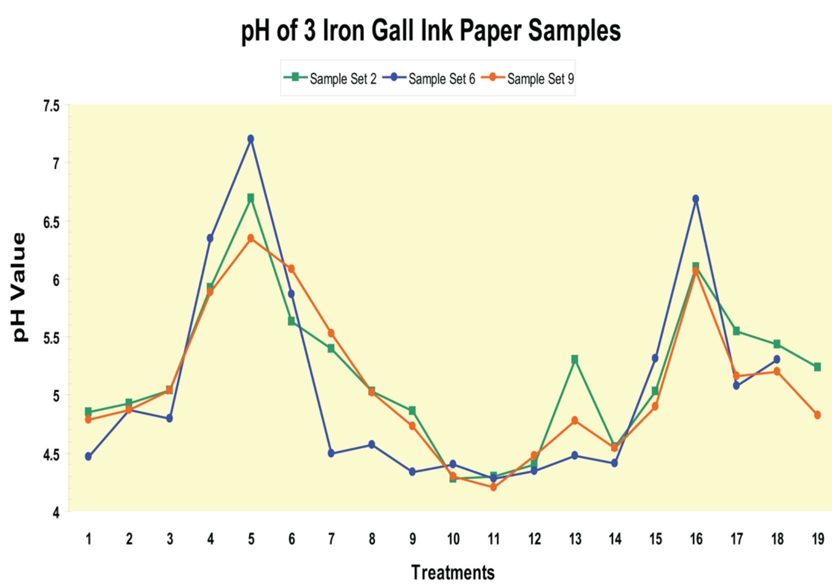

Fig. 5. pH Measurements—Surface Electrode Recorded in Dec. 2004

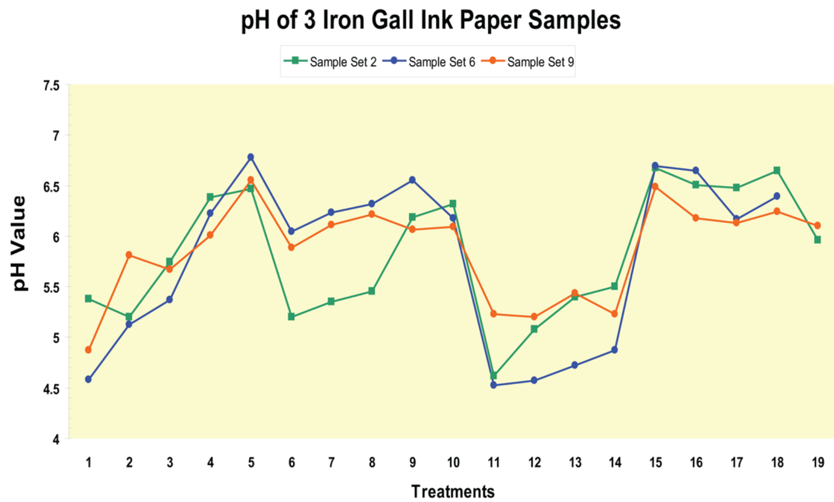

Fig. 6. pH Measurements—Cold Water Extraction Recorded in June 2006

on most samples. The more water that was used in the treatments, the more these lines were removed. From all the aqueous treatments at 20° C used (#2, #3, and #6), the one that removed most Fe^{2+} and resulted in the least bleeding of inks was RO+alkaline water (#6). The pH values of aqueous treatments at 20° C were higher than the results obtained with all aqueous washing at increased temperature treatments, with and without alcohol (#7, #8, #9, and #10) and baths of 100% alcohol and mixtures of water+EtOH solutions, e.g., 3:1; 1:1; and 1:3 (#11, #12, #13, and #14).

Aqueous Deacidification

In many samples deacidified with calcium or magnesium bicarbonates solutions (#4 and #5) the papers looked slightly darker than RO+alkaline water (#6). Some samples showed a slight bleeding of the inks with both calcium and magnesium bicarbonate deacidification solutions. Both calcium and magnesium bicarbonate removed most of Fe^{2+} and few samples showed slight deposits across the papers after treatments. The traces of Fe^{2+} were quite similar, but samples treated with calcium bicarbonate appeared to have better results overall than those treated with magnesium bicarbonate. All the samples treated with magnesium bicarbonate achieved the highest pH values after treatments.

Aqueous Washing at Increased Temperature

By elevating the water temperature to 90° C (#8 and #10), the efficiency of washing the papers increased. Daniels and Kosek have had similar results (2002); more soluble Fe^{2+} ions and water-soluble components seemed to be removed. Overall, the papers appeared much cleaner and lighter in color. The increase in flexibility and brightness in the substrates is likely caused by the removal of sizing and fillers. Generally, all the samples subjected to simmering water treatments (#8 and #10) looked the brightest, inks became duller, and the colors of both inked lines and papers were affected. When examined under UV-fluorescence illumination (figs. 3–4), all the samples treated with simmering water washing have a very distinctive appearance; this is probably due to the removal of sizing (gelatin), calcium sulfate (gypsum) filler, and other additives. The pH values (measured with a surface electrode) after simmering were quite low, ranging from pH 4.04 to pH 5.53. Cold water extraction results trended toward an increase in pH with simmering (Tse 2008) (figs. 3–6).The samples treated in this study with simmering water washing were not deacidified.

Alcohol and Water: Alcohol Mixtures

Because the dissolution of the ink compounds was different in the nine sample sets, the various combinations of alcohol and mixtures of water and alcohol were studied to assess the ability to protect the media. Ethanol seemed to prevent damage, such as bleeding, ink blurring, and color change. Alcohol is known to lower the surface tension of water, promoting fast and even penetration and improving wetability of both the inked lines and paper supports. The use of alcohol limits the migration of the inks and the brown haloing effect that is apparent when using only aqueous treatments. It appears from this study that the more EtOH used in the solutions, the less effective the removal of Fe^{2+} discoloration of paper, and other water-soluble materials. The addition of EtOH to various aqueous treatments decreased the water content of the solutions and limited the solubility of water-soluble degradation products. From all the combinations tested, the 1:1 EtOH:RO solution (#13) produced the least bleeding in comparison with other aqueous treatments (#2, #3, and #6) but had an undesired effect of reducing the extraction of Fe^{2+} ions after treatment. Possibly, this could cause a risk of continued degradation of the paper in the future.

Ca-Phytate + Ca-Bicarbonate Solution

From the five calcium phytate treatments used, the best visual results and the least side effects were achieved with calcium phytate/calcium bicarbonate (#16). This treatment seems also to be the most effective in delaying ink corrosion as well as depositing an alkaline reserve in the papers. This was evident from the Fe^{2+} tests and pH values obtained from all the samples treated with the chelating agent followed by deacidification. Most of the Fe^{2+} ions were removed during aqueous phytate treatments and overall there were no traces across the papers after washing. Additionally, no white calcium phytate precipitates were found on the surface of the papers and inks following the treatments. These deposits can be caused by the low solubility of calcium phytate and favorable results were obtained with pH 5. The pH values were overall slightly higher than the results obtained with deacidification with calcium bicarbonate (#4). When using calcium phytate plus calcium bicarbonate (Ca-Ph+Ca bicarb.), no visual changes in the ink colors and in paper appearances were observed. After bathing, the discoloration present in the papers seemed to be reduced much more than with the treatment option employing the addition of ethanol as a wetting agent (#15).

CONCLUSIONS

The combined treatment using calcium phytate and calcium bicarbonate proved to be the most effective, causing the least side effects and thus providing the most lasting protection (Tse et al. 2006; Tse et al. 2009). Phytate does not destroy iron (III) gallotannates and deactivates iron, but no other transition metals such as Cu, Zn, Mn, Al, Mg, CO, and Ni (Neevel 2008).

Visual examination showed that there were not many differences in yellowing of papers and browning of inks when calcium or magnesium bicarbonate solutions were used. Research has shown that a single deacidification treatment with calcium or magnesium bicarbonate solutions is not sufficient to prevent ink corrosion because the Fe (II)-catalyzed oxidation is only blocked temporarily, since Fe (II) ions are oxidized in an alkaline environment to Fe (III) ions (Reissland 1999). A combined treatment with aqueous calcium phytate and calcium bicarbonate provides a better protection than aqueous calcium bicarbonate alone (Neevel 2008).

The aqueous washing treatments with increased temperatures at 40º C and 90º C caused substantial color changes in both the inks and papers, and especially the treatments that were pre-dried with a suction table (#9 and #10) that removed the sizes and dyes or pigments from colored papers. Visually the results obtained with simmering water washing were the least desirable treatment options.

The results from this study concluded that the more alcohol in the solution, the less effective the removal of Fe (II) ions and other water-soluble degradation components. Both Reissland (1999) and Eusman (2006) have had similar results. Conservators should consider to what extent it is necessary to preserve the original characteristics of the papers and inks, such as dyes or pigments in colored papers, ruling lines, and variation in the colors of the inks caused by the presence of additives or impurities, e.g., natural dyes, logwood, indigo, Brazilwood, and aniline dyes.

All aqueous treatments removed most of the iron (II) ions, and there were no deposits across the papers tested with indicator paper to identify iron (II) ions present as salts in the paper. The more water that was involved with the treatments, the more discoloration was removed from the papers, but occasionally the inks bled or appeared slightly dull after treatments. The test for iron (II) ions, Bathophenanthroline, has proven to be a useful tool for conservators to monitor the presence of free iron ions on the media and paper and within the washing solutions.

Resizing with gelatin should be considered after simmering water treatments and most stabilization treatments of ink-corroded archival materials (Kolbe 2004).

ACKNOWLEDGEMENTS

The author gratefully acknowledges M. Trojan-Bedynski, Senior Paper Conservator, Library and Archives Canada; S. Tse, Senior Conservation Scientist/Chemist, Canadian Conservation Institute; and J.L. Biggs, Senior Paper Conservator, Library of Congress, for their guidance and collaboration during the project. Also, J.M. Kosek, Acting Head of Paper Conservation at the British Museum; B. Reissland, Paper Conservator/Conservation Scientist and Dr. H. Neevel, Conservation Scientist, Instituut Collectie Nederland; Dr. V. Daniels, Senior Conservation Scientist, Royal College of Art; I. Brückle, Head of Conservation, Kupferstichkabinett Staatliche Museen zu Berlin; S. Watkins, Senior Paper Conservator, Harry Ransom Center, University of Texas; E. Eusman, Assistant to the Director for Preservation, Library of Congress; and L.A. Blaser, Associate Manager, National Park Service, for their very generous comments about this research project. The author is also most grateful to the entire faculty of the University of Delaware/Winterthur Art Conservation Program for all their teaching efforts, especially to R.C. Wolbers, Associate Professor, who acted as the advisor for this research; D. Hess Norris, Acting Chairperson and Director the University of Delaware/Winterthur Art Conservation Program; L. Olcott Price, Senior Conservator of Library Collections; J. Krill, Head of Paper Conservation; and J. Carlson, Dr. J. Mass, and Dr. J. Weber at the Analytical Laboratory. The financial support received from the University of Delaware/Winterthur Museum to carry out this investigation and from the Foundation of the American Institute of Conservation

of Historic and Artistic Works (FAIC) to present this work at the Second Iron Gall Ink Meeting/(MIP) Metals in Paper Conference 2006 and the AIC Annual Meeting 2008 in Denver, CO, is gratefully acknowledged.

MATERIALS

Bathophenanthroline iron (II) ion test
PEL - Preservation Equipment Ltd.
Norfolk, UK
www.preservationequipment.com

NOTES

1. For more on currently used and newly developed aqueous treatments see Biggs 1997; Biggs et al. 2006; Choi and Zinsmeister 2006; Connelly et al. 2006; Eusman and Mensch 2001; Fleischer et al. 2006; Heller et al. 1993; Hofmann 2007; Kolar and Strlič 2001; Kolar and Strlič 2004; Kolar et al. 2003; Kolar et al. 2005; Kolar and Strlič 2005; Kolar et al. 2007; Neevel 1995; Neevel 1995a; Neevel 2001; Neevel and Reissland 1997; Neevel and Reissland 2001; Orlandini 2006; Orlandini et al. 2008; Reissland 1999; Reissland 2000; Reissland and de Groot 1999; Reissland and Hofenk de Graaff 2000; Reissland et al. 2007; Sistach 2007; Trojan-Bedynski et al. 2003; Tse et al. 2005; and Tse et al. 2009.

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