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New Applications of Lascaux Acrylic Adhesive for Book and Paper Conservation

INTRODUCTION

A Need for a New Repair Tissue

The initial impetus for this research came from a simple treatment problem: how to guard the spine folds of a book in repair without adding bulk with the chosen repair tissue. Based on experience, conservators at the University of Illinois (U of I) knew that one could sacrifice either a tissue's lightness for strength and stability or vice versa, but that it was difficult to find a material that allows the luxury of both.

While not always necessary in book conservation, there are cases where having both relative thinness and strength is important to the repair. If the cover or case is intact and the treatment attemps to fit a newly guarded and consolidated text block into its original binding without altering the spine depth, having a tissue that provides enough strength for resewing without bulk is ideal. Another instance when having a thin but robust tissue is paramount is when one is applying repair tissue over areas of fragile media without obscuring the media's legibility. A third example is the desire to have a thin tissue that is easy to work with during aqueous treatment, which would allow integration into treatment steps in where the object is already wetted out. These last two cases were particularly interesting to the U of I conservators since there had been much active work on establishing an in-house protocol for treating iron gall ink documents (fig. 1). Having a water-activated mending tissue that can be applied to fragile media while the object is already wet from the process of washing and chelation offers a convenient opportunity for repair without needing to rewet the object after calcium phytate has concluded. In short, while many problems can be resolved with a selective application of materials, a handful of instances where having a very fine but strong tissue would make book and paper treatment proceed more smoothly.

Once the need for such a material was identified, it was easy enough to figure out the necessary characteristics. The desired material would maximize the properties of lightweight tissues, such as transparency and minimal bulk during lining or guarding. Simultaneously, it would need to avoid weakening the tissue overall. Finally, the desired preparation had to be one that allowed consolidation of the paper matrix such that the precoated tissue would be more workable when remoistened, allowing it to be manipulated without separating or falling apart during brushing or handling during treatment.

Fig. 1. Repair over fragile iron gall media using Lascaux-WSP tissue applied during aqueous treatment steps.

Why Tengujo?

Tengujo paper, made by the Hidaka Washi company, is thin. So thin, in fact, that some retailers refer to it as "Spider Tissue," whereas the full Japanese name, *Tosa Tengujo-shi*, translates to "wings of the mayfly" in English (Hidakawashi 2024). The thinnest version is just 0.02 mm thick—the same as the diameter of a single kozo fiber (fig. 2). Traditionally handcrafted for more than 1000 years in Japan's Kochi and Gifu prefectures, the introduction of machines in the 20th century enabled even thinner production while maintaining flexibility and high strength relative to its fineness (Whang 2020).

Tengujo 5 gsm tissue has long been a favorite material for book and paper conservation treatment. Tengujo has many excellent properties without any modification—in addition to thinness, it has long fibers, which allow for fine, stable mends across breaks without being visually intrusive. It also exhibits high chemical stability, which gives conservators

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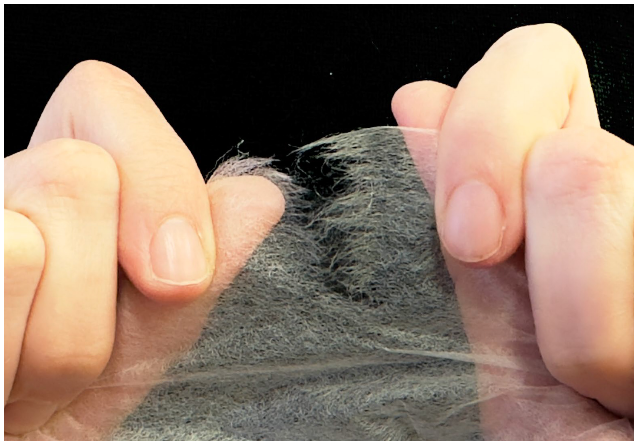


Fig. 2. Tosa Tengujo-shi, or "wings of the mayfly".

confidence when using it in treating paper-based objects (EdoFiber 2024).

Although the thinness of Tengujo paper is one of its benefits, it can be challenging to work with, especially at low weights such as 2, 3, 5, or even 7 gsm. These delicate weights must be handled with care, especially in the presence of moisture. When precoated with reversible adhesives commonly used in conservation, like wheat starch paste (WSP), methylcellulose, or gelatin, the mechanical resistance of Tengujo increases to a degree. This works well for small mending and other focused stabilization. For treatments involving greater mechanical tension, such as guarding and sewing, the crystalline molecular structures of starch and cellulose are more rigid than flexible (Davidson and Richardson 1936, 68). Consequently, Tengujo tissue can tear easily.

Why Lascaux Acrylic Adhesive?

Acrylic dispersions have been used for decades to conserve textiles (Ragauskeinė et al. 2006, 57) and paintings (Duffy 1989, 67). Although the adoption of these adhesives came a bit later in book conservation, there have been innovative uses in paper and leather treatment since the early 2000s (Kelly et al. 2020, 24). Possibly due to our wariness of other acrylic polymers in paper conservation (i.e., pressure-sensitive tape and the problem of aging plastics in cultural heritage generally), Lascaux Acrylic has not been widely adopted for paper treatment beyond its applications for heat-set tissues and leather repair. That said, the idea of using a Lascaux formulation in lightweight tissue repairs came from a familiarity with the material in other contexts—for example, the use of Lascaux 498 HV in working with degraded leather components (St. John 2000, 134), or the use of Lascaux 303 HV for making temporary mounting hinges for exhibits—a practice at the U of I since 2018. It had been observed that when Lascaux's acrylic dispersions were cast in a film over repair tissue, they significantly improved both the paper's elasticity and the overall strength of the paper's fibers. Based on that, Lascaux could potentially be used to diminish the properties of weakness and difficult handling of the Tengujo.

With Lascaux 303 HV used as a coating over Tengujo 5 gsm, an improvement in both the elasticity and strength performance was immediately observed. Tissue coated with Lascaux 303 HV sticks to a secondary paper support through its ability to remain tacky when dry. It also remains easy to remove when freshly applied. However, although useful in some applications, this residual tackiness can attract dust and accidentally adhere to surrounding documents during storage. Additionally, limited experimentation in the Conservation Lab at the U of I suggested that the tissue may become harder to remove over time—similar to the cross-linking that occurs with the adhesive on sticky notes (Siegel 1989, 67)—which would not be ideal for most conservation applications that prioritize reversibility.

Adding Paste to the Mix

The questions became this: Is it possible to retain the strength and elasticity-enhancing properties of Lascaux 303 HV without having the repair tissue compromised by the problem of long-term tackiness? Is there a way to isolate or encapsulate Lascaux 303 HV without losing the acrylic film's flexibility? The idea of adding a secondary adhesive to manage tackiness again came during myriad experiments. WSP was a natural place to start, given its familiarity, working characteristics, and long-held position as the apex adhesive in book and paper conservation. Paste would satisfy the need for this new repair tissue to be water activated and easily reversible. In a low enough concentration, it theoretically should not obscure the effects of the Lascaux adhesive. Could paste be the answer?

The next step was a lot of trial and error. Early iterations included testing with gelatin (which caused too much static cling when used) and higher paste concentrations (which reduced the desired flexibility and strength). After multiple recipes and ratios, a final formula that met the core criteria was determined—Tengujo 5 gsm coated in layers, first with a 3% concentration of Lascaux 303 HV to add strength and flexibility, then with a 2% concentration of WSP to block the tackiness, enhance the water activation, and promote reversibility (fig. 3). The result was a remoistenable precoated tissue with the thinness and transparency of uncoated Tengujo and the strength and consolidation necessary for sewing through a single layer after guarding. It was suitable for lining or double mending and did not add bulk. Because it was water activated, it could be easily integrated into aqueous steps without needing to add any additional moisture to the object after the fact.

Could this new Lascaux-paste tissue be too good to be true? Possibly—especially given that no comparable data was available to evaluate the aging properties of Lascaux 303 HV in combination with WSP. Moreover, although observational tests immediately following application demonstrated that the Lascaux-WSP tissue had both high adhesion and reversibility,

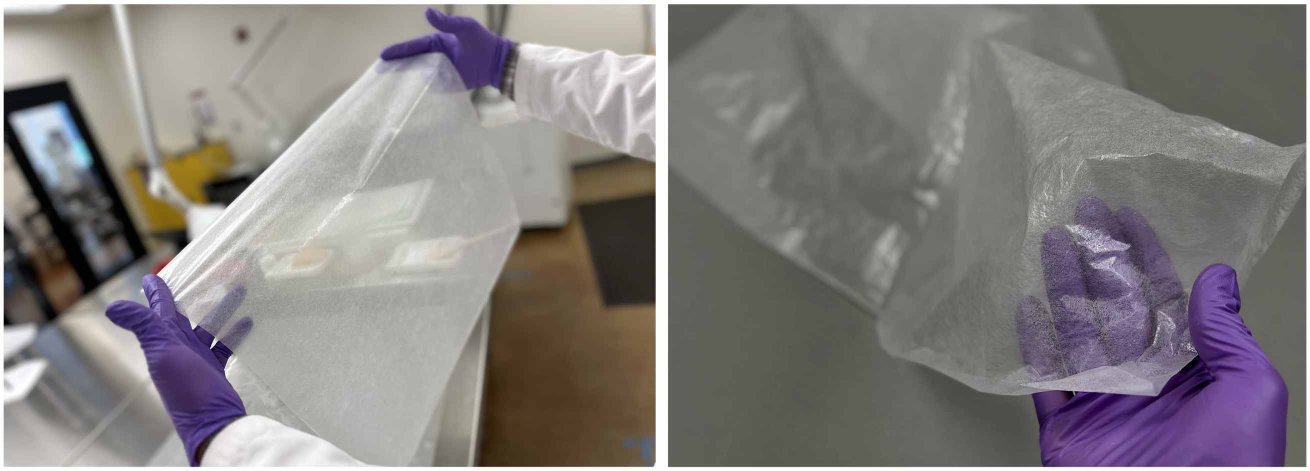


Fig. 3. "T2," the main formulation of one layer of 3% Lascaux Acrykleber 303 HV, followed by two layers of 2% cooked Zen Shofu WSP.

U of I conservators were aware that if they wanted to be confident in using this material on special collections and sharing this methodology with the field, more information on how this tissue performed over time would be needed.

DESCRIPTION OF EXPERIMENTAL SETUPS

Base Experiment: 5 gsm Tengujo Tissue Prepared With Variations of Lascaux and Paste

The first goal was to test the performance and aging characteristics of the winning formula—Hiromi Tengujo 5 gsm tissue treated with a precoating of diluted Lascaux adhesive followed by cooked and thinned Zen Shofu WSP. For the base experiment, an uncoated control sample, "TControl," plus two prepared samples, "T1" (coated with paste) and "T2" (coated with the main Lascaux-paste formula), were included. The intention of starting with these three samples was to compare the Lascaux-paste formula to the performance and aging of uncoated Tengujo tissue, and Tengujo that had been precoated with WSP.

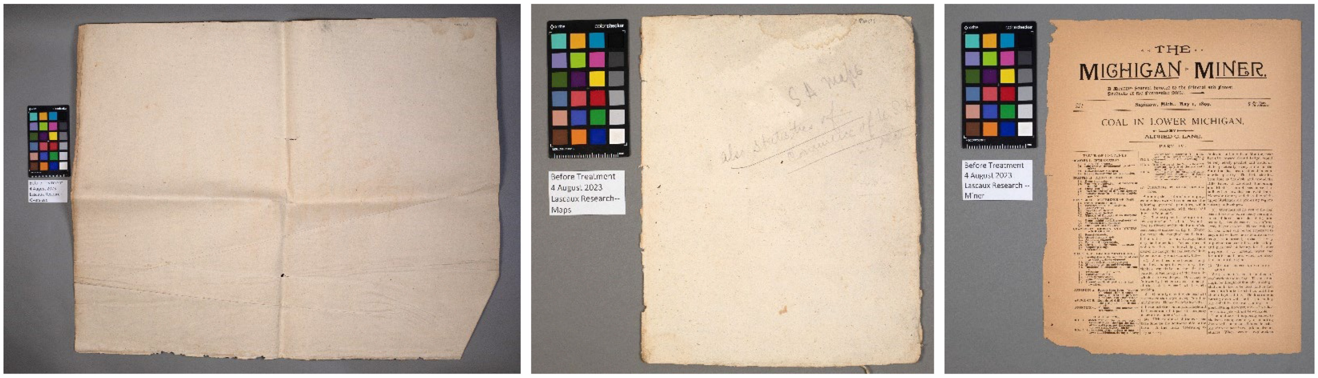
A third formulation that included pretoning with acrylic pigment was added. This is often a step during treatment meant to alter a repair tissue to be less visually disruptive to the object. For this variation, referred to as "T3," 5 gsm Tengujo was first toned with a wash of Golden Acrylic pigments before applying the same Lascaux-WSP combination as in the "T2" sample.

The final tissue preparation, referred to as "T4," swapped out Lascaux 303 HV for Lascaux 498 HV followed by coating with WSP (table 1). This was included for several reasons. First and foremost, the authors were curious to what extent WSP succeeded in encapsulating the Lascaux. Although early trials with the main formulation suggested that WSP could fully isolate the tackiness of Lascaux 303 HV, it was not clear how the adhesive characteristics of Lascaux were still operational when re-activated. Using Lascaux 498 HV, a solvent and heat-activated adhesive, was a clear way to test if the adhesive properties were still present in the Lascaux after being coated with WSP. Since the intended impact of the Lascaux was to lend the elasticity and strength of an acrylic film, the authors were not overly concerned with preserving the adhesive properties of the Lascaux but rather understanding how the adhesive properties were affected by WSP.

Second, the authors were curious whether there were any noticeable differences between using Lascaux 303 HV and 498 HV when cast over the tissue. Could the difference in application methods affect the relative strength or elasticity of the coated tissue? If not, one could attribute this feature to acrylic dispersions in general and consider 498 HV

Sample Name	Support	Preparation
TControl	Hiromi 5 gsm Tengujo	Uncoated
T1	Hiromi 5 gsm Tengujo	Coated with three layers of 2% concentration of TALAS Zen Shofu WSP applied on alternating sides
T2	Hiromi 5 gsm Tengujo	Coated with one layer of 3% Lascaux 303 HV + three layers of 2% concentration of TALAS Zen Shofu WSP on alternating sides
T3	Hiromi 5 gsm Tengujo	First toned with Golden Acrylic pigments, then coated with one layer of 3% Lascaux 303 HV + three layers of 2% concentration of TALAS Zen Shofu WSP applied on alternating sides
T4	Hiromi 5 gsm Tengujo	Coated with one layer of 3% Lascaux 498 HV + three layers of 2% concentration of TALAS Zen Shofu WSP on alternating sides

Table 1. Base Experiment Tissue Samples



"Oversized" historical end sheet paper "Maps" historical end sheet paper "Miner" historical end sheet paper

Fig. 4. Before-treatment recto images of selected historic paper stocks, "Oversized," "Maps," and "Miner" (left to right), under normal illumination.

as an alternative for this specific precoated tissue recipe. Furthermore, it would allow evaluation of the relative adhesion and reversibility of the prepared Lascaux-WSP tissue on the adhesive action of the paste alone, since it is established that Lascaux 498 HV is not water activated.

Last, including "T4" in the sample set might allow testing for different applications or benefits for a Lascaux 498 HV-WSP precoated Tengujo that the authors had not anticipated.

It is important to note that this experiment involved applying three layers of WSP, which is the maximum recommended by the authors. Although two layers are enough to achieve adhesion, using more than three will further reduce Lascaux's elasticity and strength. During the experimental phase, the most extreme formulation was used to test the limits of the adhesives in question. This detail is especially important when analyzing the results of mechanical testing.

Testing Lascaux-WSP Precoated Tengujo Over Historical Paper Samples

Once the base and variations of the tissue samples were selected and prepared, there were still further questions. Since mending tissues rarely act (or age) in isolation, the authors wondered if there was a fundamental impact on the performance and aging of the Lascaux-WSP coatings when applied to historical papers. It was quickly decided that it would be worthwhile to make a secondary sample set that consisted of the four Tengujo Lascaux-WSP formulations applied over historical papers throughout simulated treatment steps.

After a brief discussion, it was concluded that it would be useful to have several different paper stocks from which to create sample sets. Historical paper samples were selected from a collection of retained endsheet papers that were separated during repair before the formal creation of the Conservation Unit at the U of I. Three papers were selected that were different enough to yield visually distinctive results during later testing. To identify the papers individually throughout experimentation, they were each assigned names based on distinguishing characteristics.

Two of the selected historical papers (named "Maps" and "Oversized") appeared to have been handmade, with laid and chain lines visible under normal illumination, and were without printed media. The third paper selected was an anomaly in the historical endsheet collection—a print publication from 1899 called *The Michigan Miner*. "Miner" was clearly industrially produced wove groundwood pulp paper. Already discolored and embrittled, the authors were interested in how such a fragile and acidic support might impact the performance of applied mends. Each of the three papers was photographed at this "before-treatment" stage under normal illumination according to usual photo-documentation standards of practice (fig. 4).

It may have been prudent to stop with these two robust sample sets. However, the authors also wondered how the tissue might respond to aging if applied during different phases of treatment. Since each sample consisted of a full sheet, each sheet was divided into four quadrants. All quadrants were surface cleaned over the recto and verso using conservation-grade eraser crumbs combined with a soft-bristle brush. Three-quarters of the quadrants were washed following a typical procedure using successive baths of unconditioned deionized water, then allowed to dry between felts under moderate weight. Half of the quadrants received a final conditioned bath before drying, with the deionized water buffered to a pH between 8.5 and 9 using a calcium hydroxide saturated solution. The remaining quadrants were then resized after buffering, with a solution of gelatin at a concentration of less that 1%, before finally being allowed to dry between felts.

The result was the three historical papers now consisting of four pieces, each having received a different level of conservation treatment (table 2). To identify these individual historical paper samples throughout the rest of the experimentation, they were assigned two-part names, with the main paper identifier ("Oversized," "Maps," "Miner") followed by an alpha-numerical designation that indicated the treatment stage (B1–B4). With the preparation complete

Sample Name	Quadrant	Preparation
Oversized/Maps	B1 (Control, cleaned)	Historical handmade paper that has been surface cleaned
	B2 (Washed)	Historical handmade paper that has been surface cleaned and washed according to standard immersive washing practices
	B3 (Buffered)	Historical handmade paper that has been surface cleaned, washed, and buffered according to standard immersive washing practices
	B4 (Resized)	Historical handmade paper that has been surface cleaned, washed, and buffered according to standard immersive washing practices, then resized with <1% gelatine
Miner	B1 (Control, cleaned)	Historical industrial paper that has been surface cleaned
	B2 (Washed)	Historical industrial paper that has been surface cleaned and washed, according to standard immersive washing practices
	B3 (Buffered)	Historical industrial paper that has been surface cleaned, washed, and buffered according to standard immersive washing practices
	B4 (Resized)	Historical handmade paper that has been surface cleaned, washed, and buffered according to standard immersive washing practices, then resized with <1% gelatine

Table 2. Historical Paper Samples at Different Treatment Stages

for the historical paper samples, "during-treatment" photodocumentation was conducted for the four paper samples, individually capturing each of the four preparations for each sample group (fig. 5). To adhere the tissue samples to the historical paper quadrants, all four tissues were cut into strips and labeled. "T1–T4" tissue samples were applied consistently, as described in Appendix 2. This resulted in four pieces of paper with four strips of mending tissue each. For each sample, a portion of the historical paper was also left uncovered to test the paper's performance separate from the precoated tissues. Altogether, the process of preparing the historical paper samples with the Lascaux-WSP tissues took a total of 15 to 20 minutes.

Determining Accelerated Aging Test Parameters

Age testing was performed in multiple rounds to accommodate multiple sample sets. To allow for adequate working time to cycle through the progressive experimental variations, it was decided to limit the accelerated aging period to 28 to 31 days (roughly one month). To compensate for a relatively brief duration, the temperature and relative humidity parameters were set higher than the median levels suggested in the available literature so that this testing would be able to emulate the worst outcomes over time (Zou et al. 1993, 393). All samples were hung from one end through a punched hole inside a Memmert HCP50 Humidity Chamber and initially left to age for a four-week period with a relative humidity of 90% and a temperature of 90°C (fig. 6). It was quickly noticed that the first relative humidity set-point was too high, resulting in excessive condensation inside the test chamber. Subsequently, the relative humidity was adjusted to 65% for all rounds of samples.

Monitoring the aging process also included checking samples once weekly, at which point a quarter of each sample was

"Maps," recto "Maps," verso

Fig. 5. During-treatment images of "Maps" historic paper with documented phased simulated treatment.

Fig. 6. Tissue samples inserted into a Memmert HCP50 Humidity Chamber before accelerated aging has begun.

removed. This furnished a progressive set of paper coupons to document color, pH, adhesion, and reversibility changes over the four weeks.

POST-AGING QUALITATIVE TESTING: TOOLS AND PROCEDURES

Establishing Test Factors

Without having a resource on campus for mechanical properties testing, the authors began discussing what factors could be used to track differences between the aged and unaged samples. While previous research on a different project had yielded a set of qualitative criteria on which to base analysis, the context of this experimental tissue formulation presented different questions that could not be readily answered using scales designed for observed burst, foldability, and score retention. Early factors considered for qualitative testing included texture, sheen, translucency, tearing/cutting, and folding/flexing. Ultimately, however, creating a consistent test protocol for these factors that was repeatable and able to be documented proved infeasible.

The authors knew that the aged papers would likely have significant changes in both color and pH, factors for which general tools were available for evaluation. Throughout early discussion, other factors that kept surfacing were adhesion and reversibility, both immediately after first application and after accelerated aging. Finding the creation of general testing procedures for these four factors possible, the authors began drafting protocols before completing data collection on all samples.

Color Analysis

Color shift can be gauged visually—it is easy enough to discern if one thing is darker than another. That said, quantifying a change based on observation is challenging. The authors wanted to do color analysis in a way that captured the amount of change observed. Initially, familiar conservation tools were considered, such as the *Print Council of America Paper Sample Book: A Practical Guide to the Description of Paper* (Perkinson and Lunning 1996). The sample book is as advertised—a collection of historical paper samples that have been organized into scales for “Color,” “Thickness,” and “Texture.” A helpful tool in characterizing a paper object during conservation examination and documentation, the *Sample Book* presents a comparative set of standards so that anyone with a copy can understand how a particular paper might look or feel. However, it was obvious in examining the test samples after accelerated aging that the *Sample Book* would not be useful for this application, largely because only one color was classified as “brown” (fig. 7). Unfortunately, this was not a sufficient variety to accurately characterize the darkening shift across four weeks of aging.

Concurrent to this research at the U of I Library Conservation Lab, colleagues in Collection Care had long been working on responding to the growing national awareness of heavy metals in 19th century book cloth. Among other efforts, they had the idea to try using a colorimeter to see if there was a consistent diagnostic that would allow them either to more quickly identify specific hues of book cloth likely to have heavy metals present in the colorant or to eliminate hues that posed no risk. To do this, they turned to a device currently on the market, the Color Muse Colorimeter.

Color Muse is a tool that is promoted for use in interior design. Its main selling point is that one can take a reading from any color encountered in daily life and use it to get a customized paint color to match. While that aspect of its operation did not apply to this research, it does offer a diagnostic tool that tracks shifts in value by percentage. The device is straightforward to use (see Appendix 3), pairs with an app downloaded to a smartphone or tablet, and can give suggestions of color matches based on CIE $L^*a^*b^*$ color space metrics. Although not the intended use of this tool, the authors thought it would be a way to furnish quantitative results for the color shift observed after aging.

Once a testing protocol was established, evaluation was performed on the historical paper samples (on both recto and verso) and the test tissues. In recording the data, particular attention was paid to the L^* value, which concerns the shift from lightness to darkness. “After-treatment” photography

Fig. 7. *Print Council of America Paper Sample Book* (Perkinson and Lunning 1996); a useful didactic for description but has only one "brown" sample for comparative use.

was also performed under normal illumination of all samples to document the color shifts and have a comparative guide available for each historical paper.

There were a few complications—the aperture of the Color Muse's sensor, which allows for data collection, is quite small, only about 8 mm. As a result, it was difficult to get readings without significant variation. Data collection had to be repeated three times in multiple locations for each sample and then averaged to create a representative value. The source of variation seemed to come from the fact that the discoloration of the aged samples was non-homogeneous and mottled, especially for the historical handmade papers. Similarly, when analyzing the color shift in the Tengujo tissues, it was a challenge to get accurate readings simply because the tissues were naturally quite open and translucent. To accommodate this, testing required that the each sample was folded over itself up to four times to make a surface dense enough to render a measurement.

pH Analysis

Testing the pH of a paper or solution is a standard aspect of conservation treatment. This is because the reaction mechanism that causes cellulose degradation—acid hydrolysis—presents a need to mediate the deleterious presence of acid by introducing alkaline buffering components (Joel et al. 1972, 119). Thus tracking pH fluctuation throughout the accelerating aging process was considered a valuable metric for evaluation. Previous research projects included testing in this category by using a cold-water pH extraction method with great success. Unfortunately, since only a limited amount of each sample was available, there was insufficient bulk to accurately perform cold-water extraction, especially without sacrificing the aged paper before other qualitative testing was completed. Likewise, for the Tengujo tissue formulations, initial assessments of the sample material available were deemed insufficient due to the fineness of the tissue.

The authors collected surface readings with a flat-head pH probe. Although surface pH can also be taken with pH strips, this methodology was not ideal. While arguably more consistent, the test strips do not allow for measuring smaller variations. Testing before and after aging was included across all samples. In addition, pH testing was conducted on all of the prepared tissues themselves, but because of the thinness of the Tengujo, it was again necessary to fold the tissue samples over themselves to get an accurate reading.

Once the pH testing methodology was selected, data collection was still laborious. The pH probe needed to be fastidiously calibrated to get reliable data before every measurement. It was also necessary to adjust the dwell time between 1 and 3 minutes to accommodate the absorption rate of different papers. This was particularly noticeable when comparing the historical handmade paper samples (which absorbed quite quickly) to the lignin-rich machine-made paper (which was generally hydrophobic). The process also required multiple passes to recheck and confirm measurements. The instructions for using a pH probe with recommended modifications are included in Appendix 4.

Adhesion

As mentioned previously, the chief concern in using an acrylic adhesive on paper was the extent to which aged Lascaux-coated tissues cross-linked or became intractable when applied to historical paper. Given how often conservators are left trying to remove or reverse the effects of tape, glue, and other types of acrylic adhesives from previous repair attempts, it is practical to be wary when using acrylic compounds to repair paper-based special collections objects. Previous peel and lap/shear strength testing for Lascaux and BEVA adhesives has been conducted in the context of canvas lining (Katz 1985, 60). However, similar mechanical testing on Lascaux adhesive for use with paper has not been conducted.

The idea behind this criterion is to gauge how aged tissues might stick fast to a secondary paper support and therefore be able to draw conclusions on the ability of Lascaux-WSP tissue to retain its working characteristics over time. In lieu of mechanical testing, the authors chose to keep it simple by creating a protocol for the samples that was flexible but empirical. The samples were evaluated on a sliding scale using observational language to characterize each formulation's performance. The scale was rated from 1 to 5, and a rubric of clear definitions for each numerical value was available for consultation throughout the evaluation process. A worksheet was created to record the rating and additional observations about the tissue's performance (see Appendices 5 and 6 for adhesion evaluation instructions and a worksheet). Aged and unaged samples of each tissue sample set were applied in a consistent manner over commercial white Somerset 100% Cotton paper. The authors opted for a good-quality unaged stock to focus adhesion testing on the aged Lascaux-WSP tissues' performance rather than the interaction between the tissues and the historical papers. The aged and unaged tissues were applied to the Somerset paper as described in Appendix 2. After being allowed to dry, the adhesion was tested by pulling, scraping, and otherwise manipulating the dry tissue to see how its attachment to the secondary support remained intact or separated.

Reversibility

For the purposes of this article, adhesion and reversibility are, in many ways, two sides of the same coin. The authors were interested in judging how fully removable the precoated mending tissues were immediately after application versus after accelerated aging and whether the aging process on Lascaux's acrylic medium impacts how reversible the tissues remains over time. Like adhesion, a sliding scale with a clearly defined rubric for evaluation was developed, using much of the same observational language contextualized to the process of removing the tissues from a secondary support rather than applying it. However, one significant difference from the adhesion testing was that rather than preparing new samples using a commercial paper stock, instead testing was performed on the tissues applied to historical papers that had been treated and aged earlier in the experimental process (see Appendices 7 and 8). This was an important distinction since, in this case, the focus was on the interaction between the tissues and the historical papers to which there were applied. The hope was that this evaluation would clearly demonstrate a change in reversibility within the tissues over the aging process. When the evaluation of reversibility began, a 1:1 solution of deionized water (DiH_2O) and ethanol (EtOH) was used to re-activate and reverse the adhesive bond between the tissue and the paper. This ratio was adopted from personal experience of trying to reverse the adhesion of Lascaux 303 HV without significant deformation to the paper. Based on previous testing, this formula was generally successful, but the solution was later changed to a 4:1 DiH_2O to EtOH ratio, which improved the ease of removal after aging.

POST-AGING QUALITATIVE TESTING: RESULTS

Color Analysis: Results

Handmade Papers "Oversized" and "Maps"

Color analysis of both the Lascaux-WSP tissues and the prepared historic samples was as expected. Predictably, the discoloration worsened over the course of aging and was most significant for the historical paper samples that were aged for the full four weeks. Table 3 and Table 4 show the two handmade historical papers with the four tissue formulations ("T1–T4") applied to each quadrant of phased treatment preparations ("B2–B4") and aged one month. While the color shift was more significant in "Oversized" than in "Maps," numerically, each sample is mostly consistent in how much color change occurred, showing +/– 2% from one treatment phase to the next, except for "T3," where the variation is slightly higher.

The exception seems to be in the areas of the "Oversized" historical paper samples without mending. Although there is a clear and expected difference in performance between samples that received limited versus full treatment, the biggest data gap exists in comparing the percentage of color shift between recto and verso of the non-mended area (see table 3). Although true for all of the samples without mending regardless of treatment phase, it is especially noticeable in the B2 samples where the percentage of color shift in the verso compared to recto was a

"OVERSIZED" HANDMADE PAPER
Total Percentage of Color Shift After Accelerated Aging
Temperature was maintained at 90°C, RH at 65% for a period of one month

Before Aging Average
L* = 78.66

Before Aging	MENDED WITH T1 (WSP only) Recto		MENDED WITH T2 (LA303HV + WSP) Recto		MENDED WITH T3 (Toned, LA303HV + WSP) Recto		MENDED WITH T4 (WSP+LA498HV) Recto		WITHOUT MENDING Recto		WITHOUT MENDING Verso	
B2 L*=78.57	L*=52.62	–33.03%	L*=53.18	–32.32%	L*=51.70	–34.20%	L*=55.14	–29.82%	L*=55.28	–29.64%	L*=48.92	–37.74%
B3 L*=79.14	L*=54.25	–31.45%	L*=54.72	–30.86%	L*=50.38	–36.34%	L*=55.32	–30.10%	L*=50.08	–36.72%	L*=52.35	–33.85%
B4 L*=78.28	L*=53.95	–31.08%	L*=53.96	–31.07%	L*=57.78	–33.85%	L*=53.54	–31.60%	L*=50.84	–35.05%	L*=55.17	–29.52%

Table 3. Color Shift in "Oversized" Historical Paper After Accelerated Aging

full 8 points. This is a significant difference when compared to the +/–2% change of the rest of the sample set. Trying to make sense of these results, the authors hypothesized that a more dramatic color shift on verso that on recto might have been caused by a combination of inherent vice in the specific paper stock and/or the relative position of the endsheet paper to other materials or components of the original binding context. In particular, acidic boards or leather in contact with endsheets can lead to acid migration into the paper substrate over time, which may have remained latent or less obvious but for the extremities of the accelerated aging process (fig. 8).

It was also observed for many of the handmade paper samples that the presence of the precoated tissue acted as a barrier to a more extreme color shift. The exception was the "T3" Tengujo, the formulation that was first toned with Golden Acrylic pigments, then Lascaux-WSP. It showed higher darkening when applied over historical paper compared to the other tissue preparations. Despite having data that clearly quantified the darkening, it was still difficult to distinguish where the shift was happening. Was the discoloration in the tissue? In the pigment? In the paper? A combination of all? Or is it even just an artifact of seeing the normal color shift of the historical paper support through the toned translucent layer of the "T3" tissue? Ultimately, it could not be conclusively determined based on the collected data and was noted as a possible future path of inquiry.

Industrial Groundwood Pulp Paper "Miner"

The color shift observed in the third historical paper sample, "Miner," was less extreme—both numerically and visually—when compared to the handmade paper samples. Whereas "Maps" and "Oversized" both shifted 30% and above, "Miner's" percentage of color change was in the mid-high 20%. The lesser shift and overall data stability despite "Miner" having arguably higher factors of inherent vice is surprising at first glance. Lignin-rich papers have their own internal accelerated rate of embrittlement and discoloration due to the amount of naturally occurring acid present in the paper

"MAPS" HANDMADE PAPER
Total Percentage of Color Shift After Accelerated Aging
Temperature was maintained at 90°C, RH at 65% for a period of one month

Before Aging Average
L*=89.36

Before Aging	MENDED WITH T1 (WSP only) Recto		MENDED WITH T2 (LA303HV + WSP) Recto		MENDED WITH T3 (Toned, LA303HV + WSP) Recto		MENDED WITH T4 (WSP+LA498HV) Recto		WITHOUT MENDING Recto		WITHOUT MENDING Verso	
B2 L*=90.65	L*=60.60	–33.15%	L*=60.79	–32.94%	L*=59.36	–34.52%	L*=62.89	–30.62%	L*=62.13	–31.46%	L*=59.72	–34.12%
B3 L*=89.82	L*=60.92	–32.18%	L*=63.12	–29.73%	L*=60.82	–32.29%	L*=61.04	–32.04%	L*=59.59	–33.66%	L*=63.44	–29.37%
B4 L*=88.98	L*=62.42	–29.85%	L*=62.86	–29.35%	L*=59.17	–33.50%	L*=61.06	–31.38%	L*=60.93	–31.52%	L*=65.39	–26.51%

Table 4. Color Shift in "Maps" Historical Paper After Accelerated Aging

Oversized B2-B4 during treatment, recto

Oversized B1-B4 after aging, recto

Oversized B2-B4 during treatment, verso

Oversized B1-B4 after aging, verso

Fig. 8. During-treatment and after-treatment images of "Oversized" historic paper with precoated Lascaux-WSP tissue samples, with recto (above) and verso (below) showing uneven darkening over four weeks of aging.

matrix. This often results in acid hydrolysis autocatalysis, the reaction mechanism that accounts for industrial papers' rapid degradation rate. The authors surmise that the less extreme color shift in the "Miner" sample is simply because the paper had already aged poorly all on its own, having reached the exponential peak of darkening earlier in the object's history and resulting in a comparatively smaller range of color shift overall during accelerated aging (table 5).

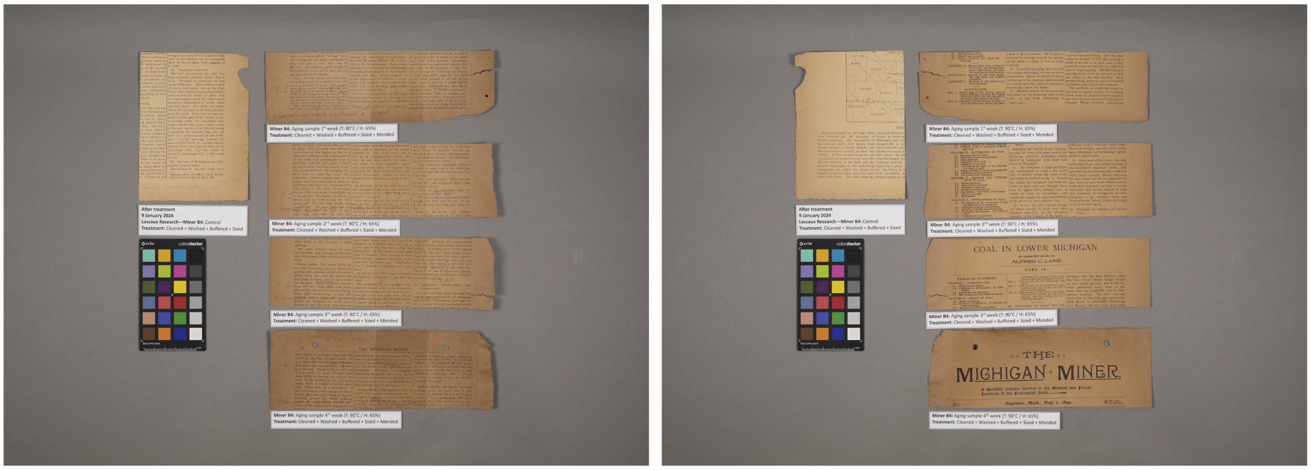
Looking again at the "T3" tissue sample applied over "Miner," there is a more dramatic color shift in week 4. The authors suppose that the acrylic paint emulsion and/or Lascaux adhesive break down, so the color suspension is possibly

"MINER" INDUSTRIAL PAPER
Total Percentage of Color Shift After Accelerated Aging
Temperature was maintained at 90°C, RH at 65% for a period of one month

Before Aging Average
L*=72.83

Before Aging	MENDED WITH T1 (WSP only) Recto		MENDED WITH T2 (LA303HV + WSP) Recto		MENDED WITH T3 (Toned, LA303HV + WSP) Recto		MENDED WITH T4 (WSP+LA498HV) Recto		WITHOUT MENDING Recto		WITHOUT MENDING Verso	
B2												
L*=73.84	L*=55.35	–25.04%	L*=55.45	–24.91%	L*=54.00	–26.87%	L*=54.58	–26.08%	L*=55.28	–25.14%	L*=55.62	–24.67%
B3												
L*=71.64	L*=54.51	–23.91%	L*=55.40	–22.67%	L*=53.68	–25.07%	L*=54.03	–24.58%	L*=54.45	–23.99%	L*=56.00	–21.83%
B4												
L*=73.03	L*=53.78	–26.36%	L*=55.28	–24.31%	L*=54.00	–26.06%	L*=53.54	–26.69%	L*=51.56	–29.40%	L*=55.11	–24.54%

Table 5. Color Shift in "Miner" Historical Paper After Accelerated Aging



Miner B4 over four weeks of aging with Lascaux-WSP tissue, recto

Miner B4 over four weeks of aging with Lascaux-WSP tissue, verso

Fig. 9. "Miner B4" industrial paper with precoated Lascaux-WSP tissue samples, recto (left) and verso (right), showing significant discoloration in the area where "T3" was applied.

transferring to the paper support itself. After the reversibility testing, when the "T3" tissue was removed from the "Miner" paper sample, it was observed that the darkening was, in fact, in the historical sample and not a visual effect of the toned tissue being layered over the historical paper (fig. 9).

This effect is just visible in "Miner" only in the fourth week, and under the most extreme conditions of accelerated aging, although exactly why is difficult to say. It may be related to the major differences between "Miner" and the other historical handmade paper samples. "Miner" has both the presence of lignin and other contemporary industrial additives or coatings that might have been popular at the time.

One could posit that adhesive film and/or acrylic pigment have migrated into the paper during the aging process, leading to a more significant color shift overall. To better understand where the darkening was happening, the authors followed up the planned qualitative analysis by taking one of the "Miner" samples aged four weeks, splitting it in two, and washing one half to see if the stronger darkening in the paper support where the "T3" tissue had been applied could be diminished or reversed (fig. 10). After washing and allowing it to dry, it was clear that the preferential color shift remained as visible in the washed half as it was in the unwashed half. Although the percentage color shift numbers themselves remain stable,

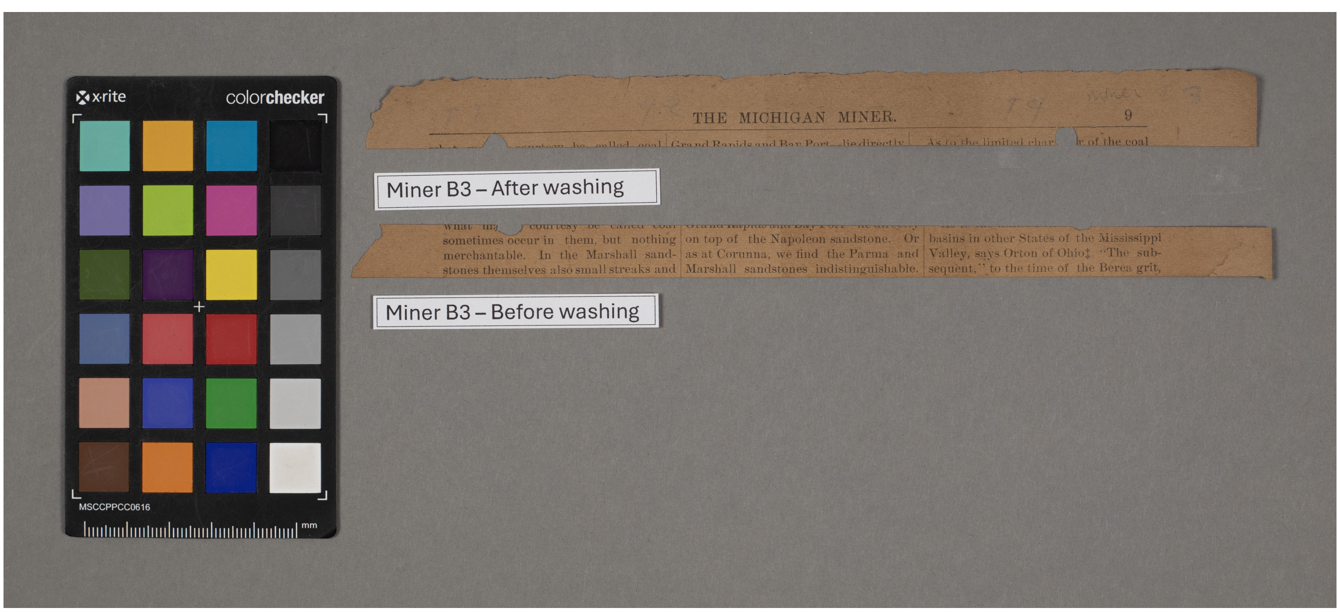
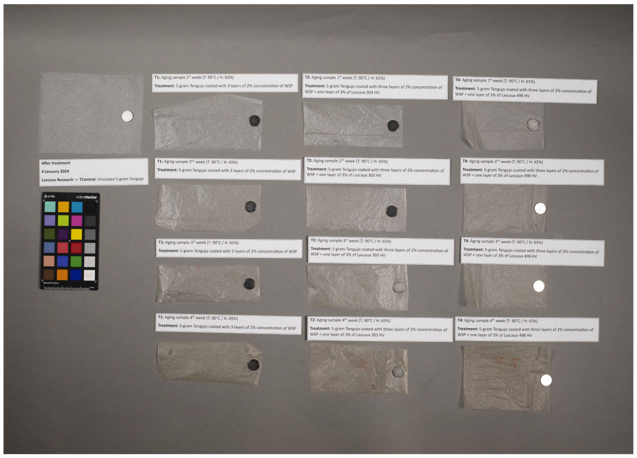


Fig. 10. "Miner B3" industrial paper divided and then washed to see if the discoloration caused by contact with "T3" could be removed or reversed. The discoloration appears unchanged.



Tengujo samples over four weeks of aging, including T1, T2, T4 and a control sample

Tengujo T3 samples over four weeks of aging

Fig. 11. Four formulations of precoated Tengujo, plus the control, showing the progression of physical changes as a result of accelerated aging over four weeks.

the more significant darkening of the industrial paper in the presence of toned Lascaux-WSP tissue suggests that use of the "T3" formulation could potentially pose risks for use in certain papers. It is imperative to do more experiments using acrylic color in paper conservation to establish the risk of using this material in archival materials.

Tengujo 5 gsm Precoated With Lascaux and Paste

As mentioned previously, the openness of the paper matrix in the Tengujo tissue makes the impact of the observed color shift much less significant compared to the perceived changes in the historical papers (fig. 11). The tissue showed a lower percentage of darkening overall, only 14% to 16% inclusive of formulations "T1–T4" (table 6). It is evident that both acrylic and paste influence paper degradation. The uncoated control sample of Tengujo was quite stable, with a total change of –3% by the end of four weeks and no change at all from the first to the second week and the second to third week of aging. For the rest of the samples, any coating applied to the paper—regardless of whether they are WSP only or Lascaux-WSP—makes a notable difference in the overall tissue aging. In handling, the week 4 samples of the tissues (compared to the base control sample) universally shift brown rather than yellow, and in handling, they feel weaker and more fragile when manipulated by hand. The "T3" sample (toned) again is harder to evaluate based on color analysis alone, but observationally looks and feels changed from its unaged control sample.

While the precoated tissues underperformed compared to the uncoated Tengujo, seeing a weakening of the coated tissue is still preferable to potential cross-linking or the development of intractable adhesion, as we often see in pressure-sensitive tape or other acrylic-based materials, or the migration of components into the secondary support, as discussed earlier in the "Miner" + "T3" combination. This aspect is more thoroughly discussed in later sections of this article.

pH Analysis

Handmade Papers "Oversized" and "Maps"

Like color analysis, pH analysis met predicted expectations. Before aging, the pH of all sample set improved throughout phased treatment, with an increase in the initial pH after the document was treated (tables 7, 8). Once inserted into the humidity chamber, the pH values sharply drop after the first week, followed by a more stable decrease in subsequent weeks. Based on the data collected, it could be deduced that when comparing an untreated and treated sample, most of the decrease in pH had to do with the inherent vice of the historical paper stock, presumably for similar reasons as discussed previously. The change of the unmended recto and verso in "Oversized" is much smaller in pH than observed in color shift. This suggests that the pH of the aged sample is potentially more evenly distributed throughout the paper matrix than the discoloration, which appears to be operating on the surface of either side.

Another difference between the two handmade papers is that while the change in pH in the "Maps" sample set is quite consistent across the different tissue formulations and from one treatment phase to the next, the "Oversized" pH data is much more irregular. The authors attribute this to the mottled and non-homogeneous character of the paper that became visible after aging, which could also be an artifact of acid migration, as discussed in the previous section.

Industrial Groundwood Pulp Paper "Miner"

A few unusual trends were noted—specifically, a decrease in pH after aging directly related to buffering and sizing. This

PRECOATED TENGUJO TISSUE FORMULATIONS Total Percentage of Color Shift After Accelerated Aging Temperature was maintained at 90°C, RH at 65% for a period of one month				
Coated Tengujo Formulation/Week	Initial Color L*	Weekly Color Value Shift	Total % Decrease Week to Week	Average % Compared to the Previous Week
TControl (Uncoated)	96.59			
TControl (Uncoated)/Week 1		94.75	–1.90%	–1.90%
TControl (Uncoated)/Week 2		94.62	–2.04%	–0.14%
TControl (Uncoated)/Week 3		94.38	–2.29%	–0.25%
TControl (Uncoated)/Week 4		93.57	–3.13%	–0.84%
T1 (WSP Only)	94.07			
T1/Week 1		90.96	–3.31%	–3.31%
T1/Week 2		87.27	–7.23%	–3.92%
T1/Week 3		82.54	–12.26%	–5.03%
T1/Week 4		81.35	–13.52%	–1.26%
T2 (LA303HV + WSP)	94.12			
T2/Week 1		91.20	–3.10%	–3.10%
T2/Week 2		86.78	–7.80%	–4.70%
T2/Week 3		82.18	–12.69%	–4.89%
T2/Week 4		79.12	–15.94%	–3.25%
T3 (Toned, LA303HV + WSP)	71.30			
T3/Week 1		70.90	–0.56%	–0.56%
T3/Week 2		70.61	–0.97%	–0.41%
T3/Week 3		69.49	–2.54%	–1.57%
T3/Week 4		67.00	–6.03%	–3.49%
T4 (LA498HV + WSP)	94.17			
T4/Week 1		93.34	–0.88%	–0.88%
T4/Week 2		89.48	–4.98%	–4.10%
T4/Week 3		85.07	–9.66%	–4.68%
T4/Week 4		81.00	–13.99%	–4.33%

Table 6. Color Shift in Precoated Tengujo After Accelerated Aging

"OVERSIZED" HANDMADE PAPER Total Average Percentage of Decrease of pH After Accelerated Aging Temperature was maintained at 90°C, RH at 65% for a period of one month													
		MENDED WITH T1 Recto Avg. Decrease: 34%		MENDED WITH T2 Recto Avg. Decrease: 34%		MENDED WITH T3 Recto Avg. Decrease: 34%		MENDED WITH T4 Recto Avg. Decrease: 35%		W/O MENDING Recto Avg. Decrease: 34%		W/O MENDING Verso Avg. Decrease: 34%	
	Initial pH	Final pH	% D.	Final pH	% D.	Final pH	% D.	Final pH	% D.	Final pH	% D.	Final pH	% D.
B1	3.70	–	–	–	–	–	–	–	–	–	–	–	–
B2	6.00	4.14	–31.00%	4.04	–32.67%	4.20	–30.00%	3.97	–33.83%	3.99	–33.50%	3.97	–33.83%
B3	6.16	3.97	–35.55%	4.15	–32.63%	3.99	–35.23%	4.09	–33.60%	4.04	–34.42%	4.04	–34.42%
B4	6.18	3.93	–36.41%	3.86	–37.54%	3.88	–37.22%	3.92	–36.57%	4.04	–34.63%	4.03	–34.79%

Table 7. Change in pH in "Oversized" Historical Paper After Accelerated Aging

“MAPS” HANDMADE PAPER Total Average Percentage of Decrease of pH After Accelerated Aging Temperature was maintained at 90°C, RH at 65% for a period of one month													
		MENDED WITH T1 Recto Avg. Decrease: 34%		MENDED WITH T2 Recto Avg. Decrease: 34%		MENDED WITH T3 Recto Avg. Decrease: 34%		MENDED WITH T4 Recto Avg. Decrease: 35%		W/O MENDING Recto Avg. Decrease: 34%		W/O MENDING Verso Avg. Decrease: 34%	
	Initial pH	Final pH	% D.	Final pH	% D.	Final pH	% D.	Final pH	% D.	Final pH	% D.	Final pH	% D.
B1	4.75	–	–	–	–	–	–	–	–	–	–	–	–
B2	5.72	4.35	–23.95%	4.32	–24.48%	4.34	–24.13%	4.37	–23.60%	4.32	–24.48%	4.35	–23.95%
B3	6.15	4.36	–29.11%	4.35	–29.27%	4.34	–29.43%	4.34	–29.43%	4.34	–29.43%	4.36	–29.11%
B4	5.82	4.26	–26.80%	4.28	–26.46%	4.23	–27.32%	4.24	–27.15%	4.24	–27.15%	4.23	–27.32%

Table 8. Change in pH in “Maps” Historical Paper After Accelerated Aging

decrease was present in samples for all of the historical papers but was of particular note in the industrial “Miner” paper stock (table 9). Given that “Miner” had the lowest pH to start, it is reasonable to hypothesize that washing the samples had a disproportionately positive effect on the pH. At the same time, the less than 1% gelatin solution used during sizing caused the pH to dip slightly. Despite buffering and sizing consistently causing a dip in pH, the pH data from the “Miner” sample set shows a stable change in percentage decrease overall, with the peak in acidity being minor and consistent regardless of the tissue formulation being applied. While the effect of buffering with calcium carbonate followed by gelatin sizing on industrial—or indeed any—paper is not the focus of this research, it might be an interesting topic for future investigation. Since the most dramatic change is observed in lignin-rich wood pulp paper, one could postulate that it may be related to other industrial additives. However, it is hard to draw firm conclusions since this may depend highly on individual paper manufacturers’ era, location, and practices.

Tengujo 5 gsm Precoated With Lascaux and Paste

For the Tengujo tissue samples, the overall change between initial and final pH measurements has a narrower range. As with the historical paper samples, the change in pH for the precoated tissues decreased steadily over the four weeks of aging. Regarding the “T2” tissue, the initial pH of the Tengujo was 6.2, which increased to 6.45 immediately after coating and decreased to 5.08 after aging. The total decrease is 23%. Although this clearly indicates that precoating with Lascaux-WSP does have an impact on how much the pH decreased, it is important to note that the “T1” formulation—coated only with WSP—is just a few percentage points behind the tissues with the Lascaux-WSP combination. The uncoated control sample’s pH value decreased from 6.2 to 5.01 over the four weeks, registering a 19% decrease (table 10). Even though the pH percentage decreases at first glance, it is higher in the case of Lascaux-WSP-coated formulations of Tengujo and the change overall is not radically different from Tengujo aging on its own.

Adhesion

“T1” and “T2” formulations both adhered to the paper support uniformly and without visible delamination, regardless of the length of aging. Subtle planar deformation was noted in the area where the mending was applied, but there was no puckering, cupping, or warping; the deformation was only

“MINER” INDUSTRIAL PAPER Total Average Percentage of Decrease of pH After Accelerated Aging Temperature was maintained at 90°C, RH at 65% for a period of one month													
		MENDED WITH T1 Recto Avg. Decrease: 23%		MENDED WITH T2 Recto Avg. Decrease: 25%		MENDED WITH T3 Recto Avg. Decrease: 24%		MENDED WITH T4 Recto Avg. Decrease: 24%		W/O MENDING Recto Avg. Decrease: 23%		W/O MENDING Verso Avg. Decrease: 24%	
	Initial pH	Final pH	% D.	Final pH	% D.	Final pH	% D.	Final pH	% D.	Final pH	% D.	Final pH	% D.
B1	3.4	–	–	–	–	–	–	–	–	–	–	–	–
B2	6.00	4.98	–17.00%	4.78	–20.33%	4.80	–20.00%	4.83	–19.50%	4.91	–18.17%	4.90	–18.33%
B3	6.58	4.75	–27.81%	4.75	–27.81%	4.85	–26.29%	4.84	–26.44%	4.84	–26.44%	4.83	–26.60%
B4	6.50	4.87	–25.08%	4.83	–25.69%	4.82	–25.85%	4.83	–25.69%	4.82	–25.85%	4.80	–26.15%

Table 9. Change in pH in “Miner” Historical Paper After Accelerated Aging

PRECOATED TENGUJO TISSUE FORMULATIONS Total Percentage of pH Change After Accelerated Aging Temperature was maintained at 90°C, RH at 65% for a period of one month				
Tengujo Formulation/Week	Initial pH	Weekly pH Decrease	Total % Decrease Week to Week	Average % Compared to the Previous Week
TControl (Uncoated)	6.20			
TControl/Week 1		5.45	–12.10%	–12.10%
TControl/Week 2		5.36	–13.55%	–1.45%
TControl/Week 3		5.20	–16.13%	–2.58%
TControl/Week 4		5.01	–19.19%	–3.06%
T1 (WSP Only)	6.40			
T1/Week 1		5.93	–7.34%	–7.34%
T1/Week 2		5.70	–10.94%	–3.59%
T1/Week 3		5.53	–13.59%	–2.66%
T1/Week 4		5.25	–17.97%	–4.38%
T2 (LA303HV + WSP)	6.56			
T2/Week 1		6.00	–8.54%	–8.54%
T2/Week 2		5.52	–15.85%	–7.32%
T2/Week 3		5.21	–20.58%	–4.73%
T2/Week 4		5.08	–22.56%	–1.98%
T3 (Toned, LA303HV + WSP)	6.56			
T3/Week 1		5.93	–9.60%	–9.60%
T3/Week 2		5.73	–12.65%	–3.05%
T3/Week 3		5.49	–16.31%	–3.66%
T3/Week 4		5.28	–19.51%	–3.20%
T4 (LA498HV + WSP)	6.52			
T4/Week 1		5.71	–12.42%	–12.42%
T4/Week 2		5.41	–17.02%	–4.60%
T4/Week 3		5.21	–20.09%	–3.07%
T4/Week 4		5.09	–21.93%	–1.84%

Table 10. Change in pH in Precoated Tengujo After Accelerated Aging

visible under raking light. This is likely due to a combination of brief drying time and an absorptive secondary paper support. The mechanical resistance of the tissue—that is, the ability of the tissue to remain in a consolidated sheet when pulled or manipulated—diminished over the four-week period of testing. Regardless, this did not have any visible effect on the ability of the tissue formulations to adhere to the secondary support. The adhesives in "T1" and "T2" appeared to retain their adhesive capacity, as demonstrated through physical attempts to pull the tissue away from the paper surface, and lift or abrade it away using a microspatula.

The adhesion of the "T1" tissue (WSP only) was significantly stronger than the tissues precoated with the Lascaux-WSP combination. Whereas for "T2" the tissue broke at the line of adhesion when pulled or abraded, "T1" would also cause delamination or skinning to the surface of the secondary support. "T2" additionally showed some diminishment of the adhesive capacity in the final week of aging, going from a rating of 5 (strong adhesion) to 4 (moderate observed resistance).

The other two formulations, "T3" (toned, Lascaux 303 HV + WSP) and "T4" (Lascaux 498 HV + WSP), did not perform nearly as well as the previously discussed tissues. Both tissues had much lower initial adhesive properties, disappearing almost completely as early as the first week of aging (fig. 12). Likewise, the mechanical resistance to manipulation was lower and diminished as the aging process progressed. Additionally, when examined under magnification, it was easily observed how the acrylic film suspended in the interstices between the Tengujo fibers had largely diminished or disappeared after four weeks of aging (figs. 13, 14).

This bifurcation in the data is curious. In considering why "T3" and "T4" might behave similarly in their inability to adhere after aging, the authors hypothesize that there must be some physical and chemical similarities between the acrylic

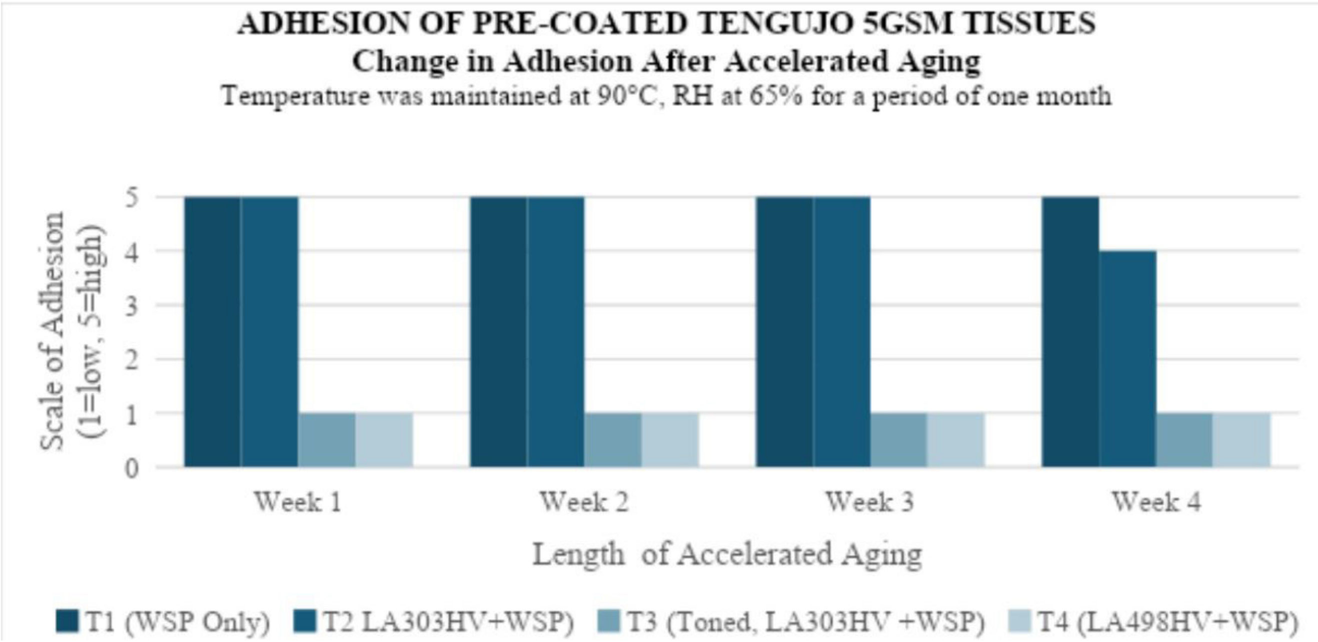


Fig. 12. The adhesion performance of "T1" and "T2" compared to "T3" and "T4" is significant and distinct.

paint emulsion and Lascaux 498 HV—something about how the acrylic film is cast over this tissue in both Golden Acrylics and Lascaux 498 HV must be similar, resulting in the adhesive properties failing quite early in the aging process for both formulations.

Another thing to note, however, is that this adhesion failure is only observed in the Tengujo tissue samples that were first aged and then applied to the new Somerset 100% Cotton stock. The historical paper samples with the "T3" and "T4" formulations applied before aging showed no signs of weakening and delamination before reversibility was attempted by introducing the 1:1 DiH_2O and EtOH solution.

Reversibility

Reversibility testing began using a 1:1 solution of DiH_2O and EtOH applied to the "Oversized" sample set. It quickly became clear, especially for the samples with more aging, that a solution with a higher proportion of water was more effective in

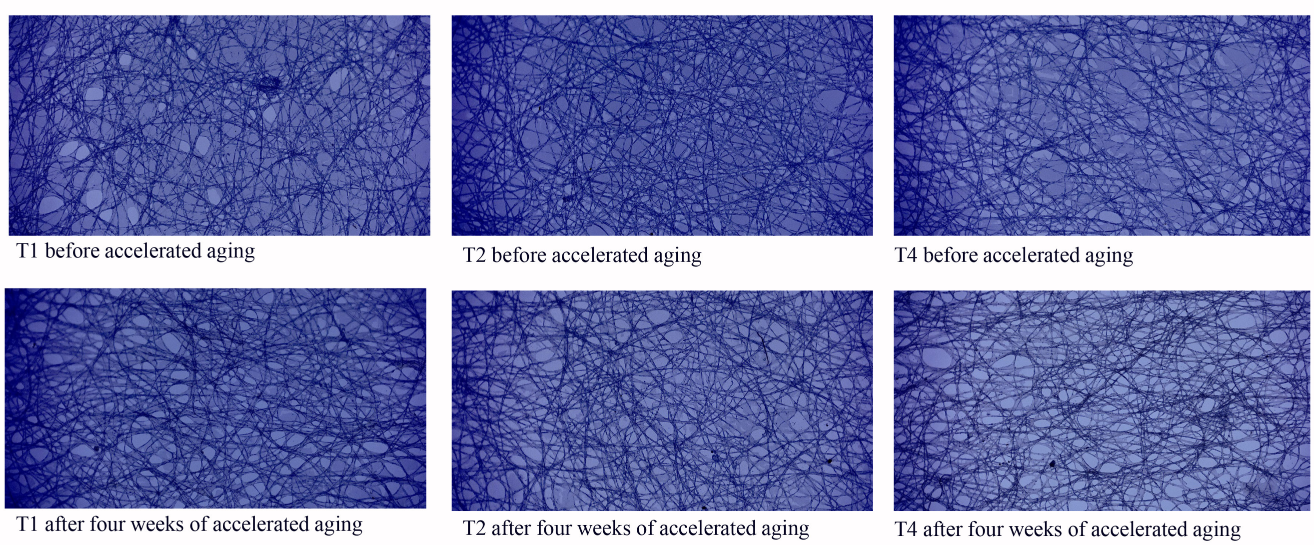


Fig. 13. "T1," "T2," and "T4" Tengujo tissue formulations before and after four weeks of aging under 40x magnification. While they all show some change to the adhesive coat suspended between the paper fibers, T4 show significant change after aging.

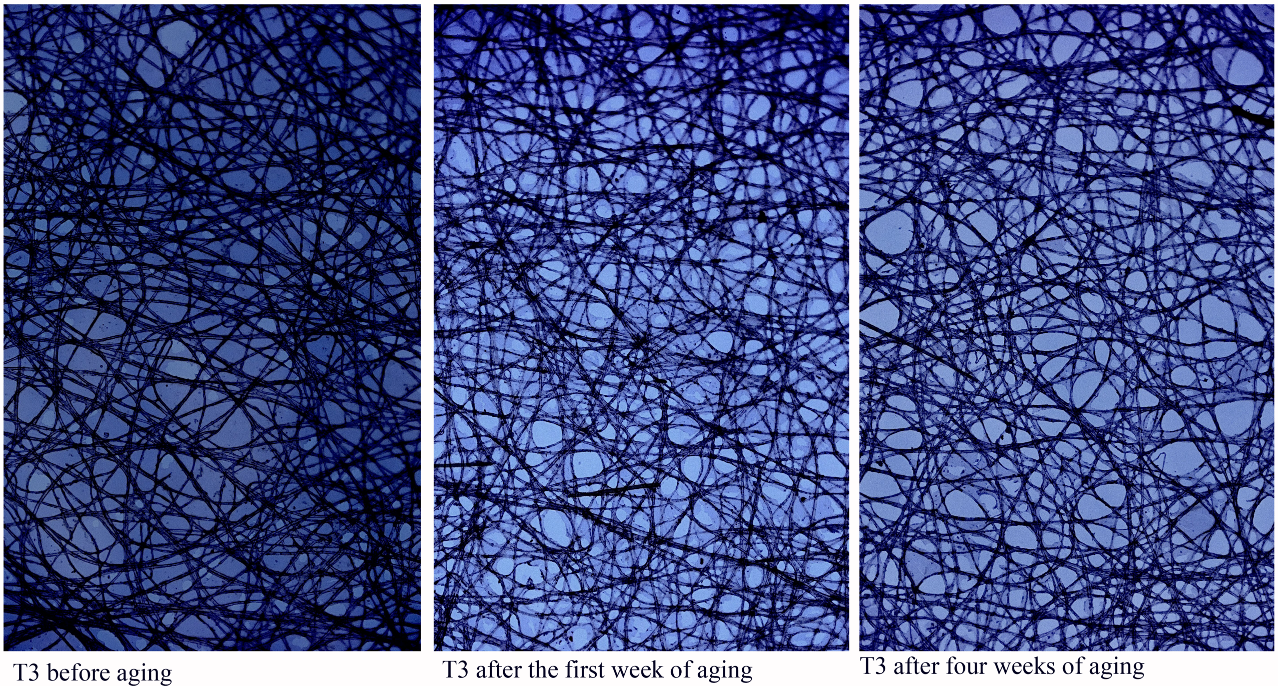


Fig. 14. “T3” Tengujo tissue formulation throughout the accelerated aging process under 40x magnification. The film suspended between the tissue fiber began to degrade as early as the first week of the aging process.

reversing adhesion (table 11). In subsequent testing, the solution was adjusted to 4:1 DiH_2O and EtOH. This change resulted in consistently high reversibility scores of 4.7 to 5 across all historical paper samples aged one to four weeks.

The efficacy of water in reversing adhesion supports the idea that the paste successfully encapsulates the Lascaux film and is the primary, if not only, source of adhesion in the coated tissue. After removing the tissue, no discoloration was observed in the samples with the “T2” formulation, and the tissue was removed easily, retained its original shape, and did not cause any delamination. Throughout reversibility testing, it was additionally observed that there were no signs of detachment occurring between the tissue and the paper samples without trying to actively remove them.

Only a few fibers of the historical paper remained attached to the tissue after reversibility testing. This seemed more likely when the paper was not sized. For the samples that had undergone resizing, there was less resistance to separating the tissue from the paper support, probably because of the sizing process consolidating historical paper samples to which it was applied.

One observation that encouraged caution was that some adhesive coating from the tissue remained on the secondary support after removal. While only visible under magnification, it was clear that the coating and tissue were separating, especially in the larger interstitial spaces between the fibers. Subsequently, after examining the secondary support when the tissue was removed, significant adhesive deposits left behind were not evident (unless mixed with fragments of Tengujo fiber), nor were they visible under magnification. The authors hypothesize that it could be due to the transparency of the adhesive, that the adhesive has broken or diminished as part of the mechanical action of removing it, or because it has broken down and migrated into the paper. This was evident for all tissue formulations and could be observed as early as after the first week of aging.

MECHANICAL TESTING

Since the main argument for the use of Lascaux adhesives in conjunction with paste for precoated repair tissue is that the acrylic film changes the morphology and strength of the Tengujo, the authors desired to add limited mechanical testing to see if it was possible to quantify that physical change. A control sample and “T1–T4” formulations were prepared both aged and unaged, and sent to SGS IPS Testing. This company conducts ISO and TAPPI standard tests on industrial paper-based products. Specific tests included ISO 12625-9 Ball Burst and ISO 12625-4 Tensile Strength (Dry).

ISO12625-9 Ball Burst

According to their website, SGS-IPS describes ISO 12625-9 Ball Burst as a test method for the determination of the resistance to mechanical penetration (ball burst strength

REVERSIBILITY OF AGED PRECOATED TENGUJO 5 GSM TISSUES T1–T4 Tissues Applied Over "Oversized" Treated Papers B1–B4 Temperature was maintained at 90°C, RH at 65% for a period of one month (Scale 1–5; 1=Low reversibility, 5=Highly reversible)						
OVERSIZED	AGING TIME	T1	T2	T3	T4	SOLVENT
B2	1st week	5	4.7	4.7	5	DiH_2O:EtOH (1:1)
B2	2nd week	5	4.7	4.7	5	DiH_2O:EtOH (1:1)
B2	3rd week	5	4.7	4.7	5	DiH_2O:EtOH (1:1)
B2	4th week	5	4.7	4.7	5	DiH_2O:EtOH (1:1)
B3	1st week	5	4.7	4.7	5	DiH_2O:EtOH (1:1)
B3	2nd week	5	4.7	4.7	5	DiH_2O:EtOH (1:1)
B3	3rd week	5	4	4.7	5	DiH_2O:EtOH (1:1)
B3	4th week	5	3	3	4	DiH_2O:EtOH (1:1)
B4	1st week	5	4.7	4.7	5	DiH_2O:EtOH (1:1)
B4	2nd week	5	4.7	4.7	5	DiH_2O:EtOH (1:1)
B4	3rd week	4.7	4.7	4.7	4.7	DiH_2O:EtOH (1:1)
B4	4th week	3	4.7	4.7	5	T1:DiH_2O:EtOH (1:1) T2–T4: DiH_2O only
MAPS	AGING TIME	T1	T2	T3	T4	SOLVENT
B2	1st week	5	5	4.7	5	DiH_2O:EtOH (4:1)
B2	2nd week	5	5	5	5	DiH_2O:EtOH (4:1)
B2	3rd week	5	5	5	5	DiH_2O:EtOH (4:1)
B2	4th week	5	5	5	5	DiH_2O:EtOH (4:1)
B3	1st week	5	5	4.7	4.7	DiH_2O:EtOH (4:1)
B3	2nd week	5	5	5	5	DiH_2O:EtOH (4:1)
B3	3rd week	5	5	4.7	4.7	DiH_2O:EtOH (4:1)
B3	4th week	5	5	5	4.7	DiH_2O:EtOH (4:1)
B4	1st week	5	5	5	5	DiH_2O:EtOH (4:1)
B4	2nd week	5	5	5	5	DiH_2O:EtOH (4:1)
B4	3rd week	5	5	5	5	DiH_2O:EtOH (4:1)
B4	4th week	5	5	5	5	DiH_2O:EtOH (4:1)
MINER	AGING TIME	T1	T2	T3	T4	SOLVENT
B2	1st week	5	5	5	5	DiH_2O:EtOH (4:1)
B2	2nd week	5	5	5	5	DiH_2O:EtOH (4:1)
B2	3rd week	5	5	5	5	DiH_2O:EtOH (4:1)
B2	4th week	5	5	5	5	DiH_2O:EtOH (4:1)
B3	1st week	5	5	5	5	DiH_2O:EtOH (4:1)
B3	2nd week	5	5	5	5	DiH_2O:EtOH (4:1)
B3	3rd week	5	5	5	5	DiH_2O:EtOH (4:1)
B3	4th week	5	5	5	5	DiH_2O:EtOH (4:1)
B4	1st week	5	5	5	5	DiH_2O:EtOH (4:1)
B4	2nd week	5	5	5	5	DiH_2O:EtOH (4:1)
B4	3rd week	5	5	5	5	DiH_2O:EtOH (4:1)
B4	4th week	5	5	5	5	DiH_2O:EtOH (4:1)

Table 11. Reversibility Observed in Precoated Tengujo on Historical

ANALYSIS OF FIVE UNAGED PRECOATED TENGUJO SAMPLES ISO 12625-9 Ball Burst specifies a test method for the determination of the resistance to mechanical penetration (ball burst strength procedure)					
Ball Burst (ISO 1265-9)	TCONTROL (UNAGED)	T1 (UNAGED)	T2 (UNAGED)	T3 (UNAGED)	T4 (UNAGED)
Bursting Strength (lbf)					
Average	0.430	1.335	1.413	1.467	1.481
Std. Dev.	0.0451	0.2033	0.2068	0.1953	0.2248
Maximum	0.512	1.582	1.763	1.793	1.789
Minimum	0.371	1.039	1.163	1.195	1.153
n=	10	10	10	10	10
Bursting Strength (N)					
Average	1.91	5.94	6.29	6.52	6.59
Std. Dev.	0.20	0.90	0.92	0.87	1.00
Maximum	2.28	7.04	7.84	7.97	7.96
Minimum	1.65	4.62	4.62	5.32	5.13
n=	10	10	10	10	10

Table 12. Physical Properties, Ball Burst of Unaged Precoated Tengujo

procedure) of tissue paper and tissue products. Testing Ball Burst consists of measuring the resistance force of a highly polished stainless steel burst ball, moving at a constant speed, penetrating a tissue test piece perpendicularly that is clamped between two concentric rings. SGS-IPS typically uses a clamping ring with an internal diameter of 4.45 cm.

Looking at the data (tables 12, 13) for both the aged and unaged Tengujo papers, adding a coating to the 5 gsm tissue imparts strength, even if it is only paste. That said, there is an increased resistance across all of the Lascaux-WSP samples compared to the "T1" sample coated only with WSP. Interestingly, of the Lascaux-WSP precoated tissues, "T3," with its layer of acrylic pigment followed by the adhesives, appears to have the smallest change in ball burst strength between unaged and aged samples.

ISO 12625-4 Tensile Strength (Dry)

SGS-IPS describes the ISO 12625-4 Tensile test as a method for the determination of the tensile strength, stretch at break, and tensile energy absorption of tissue paper and tissue

ANALYSIS OF FIVE AGED PRECOATED TENGUJO SAMPLES ISO 12625-9 Ball Burst specifies a test method for the determination of the resistance to mechanical penetration (ball burst strength procedure)					
Ball Burst (ISO 1265-9)	TCONTROL (AGED)	T1 (AGED)	T2 (AGED)	T3 (AGED)	T4 (AGED)
Bursting Strength (lbf)					
Average	0.226	0.482	0.577	0.713	0.585
Std. Dev.	0.0521	0.1221	0.0832	0.1325	0.0825
Maximum	0.337	0.711	0.737	0.924	0.708
Minimum	0.169	0.358	0.463	0.500	0.483
n=	10	10	10	10	10
Bursting Strength (N)					
Average	1.01	2.14	2.57	3.17	2.60
Std. Dev.	0.23	0.54	0.37	0.59	0.37
Maximum	1.50	3.16	3.28	4.11	3.15
Minimum	0.753	1.59	2.06	2.23	2.15
n=	10	10	10	10	10

Table 13. Physical Properties, Ball Burst of Aged Precoated Tengujo

ANALYSIS OF FIVE UNAGED PRECOATED TENGUJO SAMPLES ISO 12625-4 Tensile strength test specifies a method for the determination of the tensile strength, stretch at break, and tensile energy absorption					
Tensile (ISO 12625-4)	TCONTROL (UNAGED)	T1 (UNAGED)	T2 (UNAGED)	T3 (UNAGED)	T4 (UNAGED)
Tensile Strength (Newtons per meter)					
Average	106	311	354	457	307
Std. Dev.	16	72	24	44	25
Maximum	126	456	394	514	347
Minimum	84.3	232	323	390	276
n=	10	10	10	10	10
Tensile Strength (pounds-force per inch)					
Average	0.60	1.78	2.02	2.61	1.75
Std. Dev.	0.090	0.411	0.140	0.250	0.141
Maximum	0.72	2.61	2.25	2.94	1.98
Minimum	0.481	1.33	1.84	2.23	1.58
n=	10	10	10	10	10
Stretch at Maximum Force (%)					
Average	1.9	3.6	3.3	3.9	3.5
Std. Dev.	0.31	0.60	0.20	0.20	0.29
Maximum	2.3	4.8	3.7	4.2	3.9
Minimum	1.3	2.7	3.0	3.5	2.9
n=	10	10	10	10	10
Tensile Energy Absorption (Joules per square meter)					
Average	1.4	2.6	7.7	11.0	7.0
Std. Dev.	0.30	0.60	0.87	1.17	0.71
Maximum	1.8	4.8	9.0	12.3	7.8
Minimum	0.8	2.7	6.4	8.5	5.3
n=	10	10	10	10	10
Test Parameters					
Width (mm)	50	50	50	50	50
Nominal Gage Length (mm)	100	100	100	100	100
Crosshead Speed (mm/min)	50.0	50.0	50.0	50.0	50.0

Table 14. Tensile Properties of Unaged Precoated Tengujo

products. It uses a tensile-testing apparatus operating with a constant rate of elongation. The authors provided multiple test pieces of Tengujo paper of given dimensions. The samples were then stretched to break at a constant rate of elongation using a tensile-testing apparatus that measures and records the tensile force as a function of the elongation of the test piece. From the recorded data, the tensile strength, the corresponding stretch at break, and the tensile energy absorption were calculated.

The results of the tensile strength testing were consistent—applications of coatings to the Tengujo 5 gsm did improve performance. In this case, where the unaged "T3" formulation vastly outperformed the other sample preparations, it also lost the most strength after aging. Another notable finding in this data is that it appears that without considering the stretch at maximum force or the tensile energy absorption, the initial tensile strength data shows that all other tissue formulations tested besides "T3" actually increase in tensile strength after aging (tables 14, 15).

Although tear resistance was initially another area of mechanical testing that the authors were interested in, there was not a tear resistance testing protocol that was for tissue weight papers. More mechanical testing in the future would help deepen the understanding of how the precoatings are

ANALYSIS OF FIVE AGED PRECOATED TENGUJO SAMPLES ISO 12625-4 Tensile strength test specifies a method for the determination of the tensile strength, stretch at break, and tensile energy absorption					
Tensile (ISO 12625-4)	TCONTROL (AGED)	T1 (AGED)	T2 (AGED)	T3 (AGED)	T4 (AGED)
Tensile Strength (N/m)					
Average	184	343	327	258	321
Std. Dev.	59	29	47	47	38
Maximum	323	382	397	335	396
Minimum	127	281	257	186	285
n=	10	10	10	10	10
Tensile Strength (lbf/in)					
Average	1.05	1.96	1.87	1.47	1.83
Std. Dev.	0.335	0.164	0.269	0.271	0.217
Maximum	1.85	2.18	2.26	1.91	2.26
Minimum	0.73	1.61	1.47	1.06	1.63
n=	10	10	10	10	10
Stretch at Maximum Force (%)					
Average	1.0	1.4	1.8	2.1	1.7
Std. Dev.	0.16	0.19	0.32	0.37	0.32
Maximum	1.2	1.8	2.1	2.8	2.2
Minimum	0.7	1.1	1.3	1.6	1.2
n=	10	10	10	10	10
Tensile Energy Absorption (J/m^2)					
Average	1.0	2.4	3.4	3.1	3.0
Std. Dev.	0.45	0.40	0.54	0.80	0.69
Maximum	2.1	2.9	4.3	4.3	4.2
Minimum	0.5	1.5	2.6	2.1	2.1
n=	10	10	10	10	10
Test Parameters					
Width (mm)	50	50	50	50	50
Nominal Gage Length (mm)	100	100	100	100	100
Crosshead Speed (mm/min)	50.0	50.0	50.0	50.0	50.0

Table 15. Tensile Properties of Aged Precoated Tengujo

affecting the physical behavior and working characteristics of the Tengujo.

CONCLUSIONS

While any adhesive can eventually affect the aging characteristics of paper, the evidence presented here supports the idea that precoating repair tissue with a Lascaux-WSP combination does not present serious risks to paper objects, even under extreme aging conditions. While the decrease in pH is observed, it is stable and predictable and not as much as one might expect based on the observable color shift. The properties of adhesion and reversibility remain high after aging, suggesting that the proposed precoated tissue formulation is safe for most special collections materials. Furthermore, the mechanical testing results confirmed that the Tengujo's overall strength was improved upon with acrylic coatings, which supported the original goal of having a fine but strong tissue for conservation work.

Although the development of this tissue formulation addressed a very specific need, this research has unlocked other possible avenues of inquiry. Additional testing of the same formulations focusing on application methods or continued experimentation with other concentrations or proportions of the adhesives could provide useful iterations. However, the aging data and performance of Lascaux 303

HV acrylic adhesive have encouraged the authors to consider other book and paper conservation applications, such as Lascaux as an alternative to Klucel G in leather consolidation.

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APPENDIX 1. PROCEDURE FOR THE PREPARATION OF PRECOATED TENGUJO TISSUE WITH LASCAUX 303 HV ADHESIVE + 2% ZEN SHOFU WHEAT STARCH PASTE

Introduction

Precoated/remoistenable tissue is a common material used in book and paper conservation. It is useful for mending tears, guarding gatherings, and sometimes even facing or lining fragile paper supports. Thin paper especially Hiromi's Tengujo tissues which are available on the market from 1.6 grams on wards, is ideal for certain conservation treatments because of its thinness. Traditional conservation adhesives, such as gelatin, WSP, or methylcellulose, are commonly used for these preparations, but their chemical structure can produce a crystallization effect on the paper fibers to which they are applied. This can sometimes cause rigidity and weakness, especially in fine papers, which can be hard to work around.

The following methods outlines how to prepare fine tissues with a precoating of Lascaux Acrylic 303 HV followed by a low concentration of WSP. This yields a repair paper that has the elasticity and flexibility of an acrylic dispersion and the same adhesive and reversibility properties of precoated tissues prepared with paste.

Material and Equipment

Deionized water (200 mL)	Stir bars
Uncooked Zen Shofu wheat starch powder (2 g)	Gloves
Lascaux Acrylic 303 HV (3 g)	Silicone release Mylar
Hiromi Tengujo 5 gsm tissue	Trays
Beaker	Spatula
Glass stirring rod	Hake brushes (2)
Heated magnetic stir plate	Hair dryer

Procedure

1. Prepare a 3% solution of Lascaux Acrylic 303 HV:
   a. Measure 3 g of Lascaux 303 HV into an empty beaker.
   b. Add 100 mL of deionized water.
   c. Using a magnetic stir bar and plate, thoroughly mix the adhesive in the water until the mixture appears homogeneous.
2. Apply one layer of 3% Lascaux Acrylic 303 HV to Tengujo paper:
   a. Start with a clean sheet of silicone release Mylar that is 2 to 3 inches larger than the sheet to be coated on all sides. Then lay out the sheet of Tengujo.
   b. Using a large hake brush, apply an even layer of the Lascaux mixture to the Tengujo on top of the silicone release Mylar:
      i. Begin in the center and work your way out in a cross or starburst pattern to make sure the Tengujo wets out evenly.
      ii. Be gentle to avoid disrupting the paper fibers.
   c. Once the Tengujo is fully and evenly coated, clean the perimeter of the silicone release Mylar with a laboratory wipe or paper towel to remove any drops of adhesive solution (this makes handling in the following steps easier and ensures that the silicone release Mylar can be used multiple times).
   d. Using a hair dryer to speed drying time and prevent dust from settling, fully dry this first coat of adhesive before proceeding to the next step.
3. Prepare a 2% solution of WSP:
   a. Measure 2 g of powdered WSP into an empty beaker.
   b. Add 100 mL of deionized water.
   c. Using a hotplate with a magnetic stir bar and a temperature probe, thoroughly cook and mix the adhesive in the water until the mixture reaches 100°C.
   d. Remove from heat; add a volume of deionized water until the total volume is 100 mL (this replaces any liquid that evaporated during the cooking process).
4. Apply two to three layers of the WSP solution on alternating sides of the Tengujo, allowing it to dry between coats:
   a. Start with a clean sheet of silicone release Mylar that is 2 to 3 inches larger than the sheet to be coated on all sides. Then lay out the sheet of Tengujo coated with Lascaux.
   b. Using a large hake brush, apply an even layer of the paste solution to the Tengujo on top of the silicone release Mylar:
      i. Begin in the center and work your way out in a cross or starburst pattern to make sure the Tengujo wets out evenly.
      ii. Be gentle to avoid disrupting the paper fibers.
   c. Once the Tengujo is fully and evenly coated, clean the perimeter of the silicone release Mylar with a laboratory wipe or paper towel to remove any drops of adhesive solution (this makes handling in the following steps easier and ensures that the silicone release Mylar can be used multiple times).
   d. Using a hair dryer to speed drying time and prevent dust from settling, fully dry this first coat of adhesive before proceeding to the next step.
   e. Carefully remove the Tengujo from the silicone release Mylar, and flip over. Apply a second coat of paste to the new side.

f. Repeat, if desired, for three layers of paste. *Note: More than three layers of paste are not recommended, since they greatly diminish the elasticity of the Lascaux.*
5. To use in mending, reactivate using gellan gum or deionized water directly applied to the shiny side of the tissue.
6. Consult https://www.youtube.com/watch?v=SBV8J372scQ&t=189s for a full video tutorial.

APPENDIX 2. PROCEDURE FOR SAMPLE PREPARATION FOR LASCAUX-WSP TISSUE APPLIED OVER HISTORICAL PAPERS

Note: Because the tissue is activated with a 10% EtOH gellan gum formulation, the moisture evaporates fairly rapidly, leaving the dried mending tissue in place. If the area of the mending tissue is larger, the object needs to stay weighted down to dry longer.

1. Use gellan gum to moisturize the paper:
   a. Cut or tear the precoated paper to obtain the piece you need for the conservation treatment.
   b. Place the piece of paper over the gellan gum.
   c. Place a piece of silicone release Mylar over the paper.
   d. When the paper is wet enough (about 4 or 5 seconds), remove both the silicone release Mylar and the paper together.
2. Place the paper and the silicone release Mylar over the item to be treated.
3. Apply pressure using a Teflon or bone folder. Pressure is essential to obtain good adhesion.
4. Remove the silicone release Mylar.
5. Place the following four layers in order on top of the treated area:
   a. first, a piece of Hollytex;
   b. on top of the Hollytex, a piece of blotter;
   c. then a glass;
   d. and finally, a weight.
6. Wait until the treated area is flat and dry. This will take a maximum of 5 minutes.
7. If you need to remove the precoated paper from the document, apply light moisture with a brush or a piece of gellan gum.

APPENDIX 3. COLOR ANALYSIS USING THE COLOR MUSE COLORIMETER

Parameters for Color Data

- Evaluation is to be performed both on prepared historical paper samples and test tissues.
- Photo Doc will need to be performed to capture the color shift.
- Incorporate the use of Color Muse to quantify the color shift and to what extent it is impacted (if at all) by the presence of mending tissue on the historical sample.
- Testing includes the recto/verso of each historical treated paper with mending and without mending.

Instructions for the Use of a Color Muse Colorimeter

1. Download the Color Muse app on a smartphone. Open the app and press "Connect device."
2. Follow directions to connect and calibrate the device.
3. Once calibrated, remove the lens protection, place the device on a flat paper surface, and press it lightly so it is flush with the paper.
4. Hit "reference" on the app and record data from whichever color space is desired.
5. Repeat at least three times to collect enough data to average, moving the device randomly to different areas of the sheet of paper. Do not select the placement; rather, try to place it randomly, as this will give the most accurate data.

Link to Color Muse information and app: https://colormuse.io/color-muse.html.

APPENDIX 4. COMBINATION PH ELECTRODE INSTRUCTION MANUAL + EXPERIMENTAL USE

Introduction

This combination pH electrode comes in many styles and is designed for maximum reliability, accuracy, and ease of use. The outer body can be glass, epoxy, or other plastic materials. The plastic body electrode is available with a permanent nonremovable bulb guard or a removable guard that is shipped and attached to the cable. The reference half-cell can be refillable or permanently sealed at the factory and underneath the cap at the top of the electrode. All styles of electrodes are shipped with an electrode soaker bottle wetting solution.

Preparation

1. Remove the electrode soaker bottle covering the pH bulb, and raise the bulb area with deionized water or pH buffer. Gently shake the electrode downward in the same manner as a clinical thermometer to remove any air bubbles that might be trapped behind the pH bulb.
2. For electrodes shipped with fill hole plugs, remove the shipping tape covering the rubber fill hole plug and withdraw the plug to expose the fill hole. For electrodes shipped with a sleeve over the fill hole(s), slide the rubber sleeve down and remove the shipping tape to expose the fill hole. Fill the refillable electrodes with the proper fill solution(s) to a level just below the fill hole(s).
3. Attach the removable bulb guard if provided with an epoxy electrode by sliding the guard over the end of the electrode.
4. If stored dry for an extended time, immerse the pH bulb in the pH buffer for 30 minutes. This hydrates the pH

bulb and wets the reference junction for optimum performance. The electrode is now ready for use.

Required Materials

1. *pH meter*: This electrode will work with any commercially available pH and/or millivolt meter. Connect the pH electrode to the meter or operate the meter.
2. *Buffers*: For precise electrode standardization, two buffers are required, one of which should be close to the desired pH.

Electrode Standardization

1. Place the electrode in a fresh pH 7.00 buffer and stir. Allow the meter to read and stabilize for 30 seconds to 1 minute. Adjust the meter to read 7.00 with the standardization control.
2. Rinse the electrode in distilled water and place it in a fresh pH 4.00 buffer or fresh pH 10.00 buffer depending on whether the sample is acidic or basic. Stir and allow the meter reading to stabilize for 30 seconds to 1 minute. Adjust the meter reading to the second pH value with either the slope or temperature compensation adjustment. If impossible, see the cleaning procedure.
3. Rinse the electrode with distilled water. Place in the sample and stir. Allow the meter reading to stabilize for 30 seconds to 1 minute. Record the reading. For best accuracy, the temperature of the buffers and samples should be identical and at room temperature.

Electrode Storage

For the best results, always keep the pH bulb wet, preferably in pH 4.00 buffer with 1/100 part of saturated KCl added. Other pH buffers or tap water are acceptable storage media, but avoid storage in distilled water. The electrode storage bottle filled with a buffer will provide an ideal storage chamber for long periods.

Electrode Cleaning

Electrodes that are mechanically intact with no broken parts can often be restored to normal performance by one of the following procedures:

1. *Salt deposits*: Dissolve the deposit by immersing the electrode in 0.1M HCl for 5 minutes, then immerse in 0.1M NaOH for 5 minutes and thoroughly rinse with distilled water.
2. *Oil/grease films*: Wash the electrode pH bulb in a little detergent and water. Rinse the electrode tip with distilled water.
3. *Clogged reference junction*: Heat a diluted KCl solution to 60°C to 80°C. Place the sensing portion of the pH electrode into the heated KCl solution for approximately 10 minutes. Allow the electrode to cool while immersed in some unheated KCl solution.
4. *Protein deposits*: Dissolve the deposit by immersing the electrode in a 1% pepsin solution with a background of 0.1M HCl for 5 minutes, followed by thorough rinsing with distilled water.

If these steps fail to restore normal electrode response, replace the electrode.

Modifications to Recommended Procedure for Experimental Application (MVVP 1/2024)

1. Before each use, calibrate the pH probe. Slightly humidify the testing paper with deionized water. The absorption time varies depending on the type of paper: handmade paper absorbs quickly, whereas machine-made, lignin-rich paper absorbs more slowly. Allow the electrode measurement to stabilize, which may take up to 3 minutes, with a minimum of 1 minute needed to ensure an accurate reading.
2. After each measurement, cleanse the electrode with deionized water and corroborate the results by taking a second reading at a different location using the same procedure.
3. If the readings appear anomalous—either excessively high or low or not progressing as expected—halt the process and recalibrate the electrode according to the manufacturer's instructions.
4. Verify the results using pH strips. First, immerse the strip in deionized water, then place it on the test paper (which should be isolated on Mylar to prevent additional absorption from surrounding materials). Allow the strip to remain in contact with the test paper for approximately 1 minute.

APPENDIX 5. PROCEDURE FOR EVALUATING ADHESION OF AGED LASCAUX TISSUE SAMPLES AND SAMPLES APPLIED OVER HISTORICAL PAPERS

- The purpose of this criterion is to gauge how well-aged tissues can adhere to the paper and therefore be able to draw conclusions on the ability of Lascaux-coated paper to retain its working characteristics over time.
- For adhesion testing, we applied small samples from our prepared test tissues according to the standard application procedure. The test tissue can be applied over any high-quality Western paper stock.

Procedure for Applying Prepared Tissue Samples to Paper

1. Use gellan gum to moisturize the paper:
    a. Cut or tear the precoated paper to obtain the necessary piece for the experiment.
    b. Place the piece of paper over the gellan gum.
    c. Place a piece of silicone release Mylar over the paper.

   d. When the paper is wet enough (about 4 or 5 seconds), remove both the silicone release Mylar and the paper together.
2. Place the paper and the silicone release Mylar or polyethylene strapping over the item to be treated.
3. Apply pressure using a Teflon or bone folder. Pressure is essential to obtain good adhesion.
4. Remove the silicone release Mylar.
5. Place the following four layers in order on top of the treated area:
   a. first, a piece of Hollytex;
   b. on top of the Hollytex, a piece of blotter;
   c. then a glass;
   d. and finally, a weight.
6. Wait until the treated area is flat and dry. This will take a maximum of 5 minutes.

Procedure for Evaluating Adhesion of Applied Tissue to Stock Paper

1. Before proceeding, observe the level of adhesion extant between the dried tissue and the support paper:
   a. Does it appear uniformly stuck down?
   b. Do you observe delamination between the tissue and the paper?
   c. Under raking light, are any fibers visible out of plane with the paper?
   d. Is there any puckering, cupping, warping, or other planar distortion in the area where the tissue is applied to the paper?
2. In its dried state, without applying any moisture, begin trying to remove the tissue from the paper by peeling the strip from the paper at a sharp angle. Based on your observations, classify the adhesion process based on the following scale, where
   a. (1) equals little to no adhesion with exerted force to remove; the tissue comes cleanly away from the support paper without delamination or separated fibers
   b. (2) equals minor observed resistance with exerted force to remove, but with effort, the tissue and the paper still separate cleanly
   c. (3) equals minor observed resistance with exerted force to remove, resulting in light delamination of the tissue only but leaving the paper relatively intact
   d. (4) equals moderate observed resistance with exerted force to remove, resulting in delamination of the tissue as well as possible minor skinning of the surface of the paper
   e. (5) equals strong adhesion at work between the tissue and paper; resistant to separation with exerted force to remove, resulting in significant tearing, skinning, and delamination of the paper
3. Record your observation and additional comments in the “Qualitative Testing: Adhesion” Form.

Appendix 6. Worksheet: Adhesion of Aged Lascaux Tissue Samples and Samples Applied over Historical Papers

Please evaluate each test sample for adhesion using the procedure described in “Procedure for Evaluating Adhesion of Aged Lascaux Tissue Samples and Samples Applied Over Historical Papers.

Low High

1				5

Adhesion Rubric

(1) equals little to no adhesion with exerted force to remove; the tissue comes cleanly away from the support paper without delamination or separated fibers

(2) equals minor observed resistance with exerted force to remove, but with effort the tissue and the paper still separate cleanly

(3) equals minor observed resistance with exerted force to remove, resulting in light delamination of the tissue only but leaving the paper relatively intact

(4) equals moderate observed resistance with exerted force to remove, resulting in delamination of the tissue as well as possible minor skinning of the surface of the paper

(5) equals strong adhesion at work between the tissue and paper; resistant to separation with exerted force to remove, resulting in significant tearing, skinning, and delamination of the paper

Tissue Sample Name	Support Paper	Adhesion Scale 1–5
Additional comments:		

Tissue Sample Name	Support Paper	Adhesion Scale 1–5
Additional comments:		

APPENDIX 7. PROCEDURE FOR EVALUATING REVERSIBILITY OF AGED LASCAUX TISSUE SAMPLES AND SAMPLES APPLIED OVER HISTORICAL PAPERS

- The purpose of this criterion is to gauge how fully removable mending tissue is immediately after application versus after aging and whether the aging process on the acrylic medium of the Lascaux has an impact on how reversible it remains over time.
- For reversibility of Lascaux-WSP precoated papers (particularly if the samples have undergone accelerated aging), a 4:1 solution of DiH_2O to EtOH is recommended.
- Testing included historical paper samples with applied tissue before and after aging.

Procedure for Testing Reversibility of Applied Tissue to Stock and Historical Papers

1. Before proceeding, observe the level of adhesion extant between the dried tissue and the support paper:
   a. Does it appear uniformly stuck down?
   b. Do you observe delamination between the tissue and the paper?
   c. Under raking light, are any fibers visible out of plane with the paper?
   d. Is there any puckering, cupping, warping, or other planar distortion in the area where the tissue is applied to the paper?
2. Using a brush, apply the 80:20 solution of DiH_2O mixed with EtOH over a small area of the sample where the tissue has adhered to the paper.
3. Directly after application, using fingers, a spatula, or a pair of blunt tweezers, attempt to separate the tissue from the paper in the area where the solvent was applied. Based on your observations, classify the reversibility process based on the following scale, where
   a. (1) equals not reversible at all with applied solvent to remove; resistant to separation with exerted force to remove, resulting in significant tearing, skinning, and delamination of the paper
   b. (2) equals low observed reversibility with applied solvent to remove, resulting in delamination of the tissue as well as possible minor skinning of the surface of the paper
   c. (3) equals minor observed resistance to reversibility with applied solvent to remove, resulting in light delamination of the tissue only but leaving the paper relatively intact
   d. (4) equals paper negligible observed resistance to reversibility with applied solvent to remove, but with effort, the tissue and the paper still separate cleanly
   e. (5) equals high reversibility with applied solvent to remove; the tissue comes cleanly away from the support paper without any delamination or separated fibers

APPENDIX 8. WORKSHEET: REVERSIBILITY OF AGED LASCAUX TISSUE SAMPLES AND SAMPLES APPLIED OVER HISTORICAL PAPERS

Please evaluate each test sample for reversibility using the procedure described in “Procedure for Evaluating Adhesion and Reversibility of Aged Lascaux Tissue Samples and Samples Applied Over Historical Papers.

Low High

1				5

Reversibility Rubric

(1) equals not reversible at all with applied solvent to remove; resistant to separation with exerted force to remove, resulting in significant tearing, skinning, and delamination of the paper

(2) equals low observed reversibility with applied solvent to remove, resulting in delamination of the tissue as well as possible minor skinning of the surface of the paper

(3) equals minor observed resistance to reversibility with applied solvent to remove, resulting in light delamination of the tissue only but leaving the paper relatively intact

(4) equals paper negligible observed resistance to reversibility with applied solvent to remove, but with effort, the tissue and the paper still separate cleanly

(5) equals high reversibility with applied solvent to remove; the tissue comes cleanly away from the support paper without any delamination or separated fibers

Tissue Sample Name	Support Paper	Reversibility Scale 1–5
Additional comments:		

Tissue Sample Name	Support Paper	Reversibility Scale 1–5
Additional comments:		

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