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A Comparison of Fluids for Animal Glue Removal from Book Spines

INTRODUCTION

In book conservation, treatments involving repair of the binding can require removal of spine linings and adhesives. Removal of animal glue on book spines is often done by delivering water to the adhesive through a poultice, swelling and softening it enough so that it can be scraped away with a spatula. Possible additives in the adhesive, its proximity to covering materials like leather, and environmental conditions may encourage cross-linking in the adhesive over time, making it difficult to remove with conventional poultices like wheat starch paste or methyl cellulose. Even when it is possible to swell the adhesive, the combination of introducing moisture and the mechanical action required for removing the adhesive can cause fiber damage to the spine folds or create tide lines in the gutter of the text block.

Recent experimentations with new materials and techniques for cleaning have expanded treatment options in flat paper conservation, but their application to book spines has not been systematically tested. The multiple layers of paper and three-dimensionality of a book spine present challenges in adhesive removal that are not as prevalent in flat paper treatments, and solutions applicable for flat paper may not always be as successful for bound objects. Challenging spine adhesive removal treatments would benefit from an exploration of options presented by new and traditional methods and materials.

As there could be too many potential combinations of fluids and delivery methods to test, the project was divided into two parts: testing of fluids used for solubilizing adhesive on flat samples, and testing of delivery methods (e.g., gels) to apply the fluid to bound samples. Fluids and delivery methods to be tested were narrowed down based on affordability and ease of use, with a few novel materials added. This article focuses on identifying fluids that solubilize animal glue. Potential delivery methods will be tested in the future.

This article includes a review of the tests and results. For example, some techniques resulted in a more liquefied

adhesive, whereas others softened the adhesive to a more granular consistency, affecting the ease of mechanical removal and risk of penetration into the substrate. Some treatment circumstances may allow a one-step approach, whereas others may require utilization of multiple techniques for the removal of thick or final layers of adhesive and to prevent depositing new undesirable residues. Although there will be variabilities in actual treatments and no single method is appropriate for all circumstances, experimental results can help the conservator predict reactions and select an appropriate treatment based on the object's tolerance to moisture, heat, mechanical manipulation, and chemical reactivity.

RESOLUBILITY OF ANIMAL GLUE

In general, animal glue without additives swells readily in cold water even after prolonged aging. On its own, animal glue can be solubilized by warm water or steam above 40°C (Cannon 2015). However, it has been common historically to adjust glue recipes with additives like glycerin and honey, as well as other sugars, alcohols, polysaccharides, and salts, to improve adhesive strength, elasticity, wettability, or working time. Modification of the animal glue with such additives can affect the resolubility of the adhesive. In her own experience, the author has observed difficulties with spine adhesive removal on occasions where the text block spine was in direct contact with the leather cover, particularly with rebacked books where the reback leather has become red rotted. It is possible that exposure of the adhesive to tannins in the leather may have been a factor in its resistance to water. Schellmann (2007, 63) notes that “resolubility of animal glues may be reduced in cases where the protein has come into contact with metal ions (e.g., metal foils, tools, pigments), or with certain organic pigments and tannins, either before, during, or even after their application,” and that the lower the original concentration of the glue, the less it becomes resoluble.

The environmental conditions to which animal glue is exposed may also reduce solubility. After application, high internal stress and tensile forces develop in the glue matrix as it dries but relax over time under moderate relative humidity conditions. However, fluctuating environmental conditions

subject the glue matrix to further strains that can permanently impact the glue's stiffness and brittleness (Schellmann 2007, 62). Yannas and Tobolsky (1967) observed reduced solubility in gelatin after extended exposure to high temperatures and under vacuum. Gelatin also became partially insoluble over time under vacuum, even at temperatures as low as 25°C. They concluded that cross-linking in gelatin was a direct consequence of dehydration below a critical trace level (0.1–0.3 g water/100 g gelatin) rather than through pyrolytic decomposition at temperatures above 65°C.

EXPERIMENTAL DESIGN OF FLUID TESTS

Although the primary interest of this project is to identify successful techniques for animal glue removal from book spines, a decision was made to test the efficacy of selected fluids on animal glue solubility using flat paper samples. Conducting fluid tests on flat paper samples will reduce variables caused by three-dimensional surfaces such as the surface contact of a fluid with the adhesive, ease of mechanical removal, and vertical/lateral migration of a fluid into the substrate. Flat paper samples are also faster, easier, and cheaper to make than bound samples, and thus more flat samples can be tested within a limited time frame and budget. As such, using flat samples for the fluid tests will more efficiently pinpoint effective fluids for improving animal glue solubility. Using the more successful fluids from the fluid tests, different delivery methods can then be tested on the spine of bound paper samples to consider how they perform on three-dimensional surfaces.

Testing of different fluids was divided into two sections: studying the effect of selected fluids on the adhesive consistency over an extended period of time, and studying the influence of selected fluids on the ease of adhesive removal over different intervals of time. For both tests, five aqueous fluids were selected: deionized (DI) water, water adjusted with sodium chloride (NaCl) to boost conductivity, 3% w/v urea, 3% w/v citric acid, and a trypsin solution (see recipes in appendix 2). To test multiple fluids while controlling variables, low acyl gellan gum was selected as the only delivery method for the fluids. Both tests were conducted on samples of flat paper with artificially aged applications of animal glue.

Questions posed at the beginning of this research include:

- What can be used to improve solubility or swelling of the animal glue?
- Is there a way to reduce mechanical action during the adhesive removal?
- What are potential negative effects of the techniques used (e.g., mechanical damage, discoloration over time, impact on future treatments)?
- If the technique requires a clearing step, is there an adequate one?

Sample Preparation

Paper

The substrate for this experiment was chosen to represent the type of text block paper used in the 17th century. St Armand Old Master Papers in Frobisher (white) #57, a linen and cotton handmade laid paper, “reminiscent of the papers from the 17th century” (Talas 2020) was selected. The paper weight varies around approximately 90 g/m², an appropriate weight for text block use, and comes in sheets of 46 × 60.5 cm. The paper is semitextured and absorbent, making the paper challenging to work with for animal glue removal.

Animal Glue

Ground hide glue obtained from Talas was used in this experiment. The Talas catalog lists the Bloom strength of the adhesive at 222 g. The concentration of the glue was prepared as suggested by Talas, with 1 part glue to 10 parts water left to sit for half an hour. The glue was then heated on a hot plate until it reached a thick consistency. The temperature of the adhesive was monitored throughout preparation. It is often not recommended to allow animal glue to be heated beyond 60°C, as this can cause protein denaturation in the adhesive. In this circumstance, the animal glue was allowed to reach temperatures of 90°C to encourage the cross-linking that made animal glue removal on book spines difficult, with the additional reasoning that traditional bookbinders would likely have been less stringent in preparing their adhesives and may have likely let them overheat.

Sample Construction

Each sample was a piece of paper 5 cm square with a heavy layer of adhesive 2 cm square in the center. To prepare the flat samples, a polyester film template the same size as one full sheet of sample paper was made with a 2 cm² cutout per 5 cm². The template was then laid on top of a full sheet of the sample paper. A paint roller was dipped into the glue and rolled over the template to thickly coat the cutout area on the paper twice. After the sample sheets were dry, they were cut into 5 cm² squares. Samples underwent accelerated aging at the Library of Congress at conditions of 80°C and 65%RH for two weeks, with 10 of the flat samples retained and left unaged.

The conditions for accelerated aging were selected based on specifications used by Warda et al. (2007) and Van Dyke (2004).¹ After aging, the adhesive on the samples became noticeably harder, more brittle, and darker in color (fig. 1). Five-minute spot tests using droplets of water on samples before and after aging showed that adhesive on unaged samples quickly swelled, whereas that of aged samples remained hard.

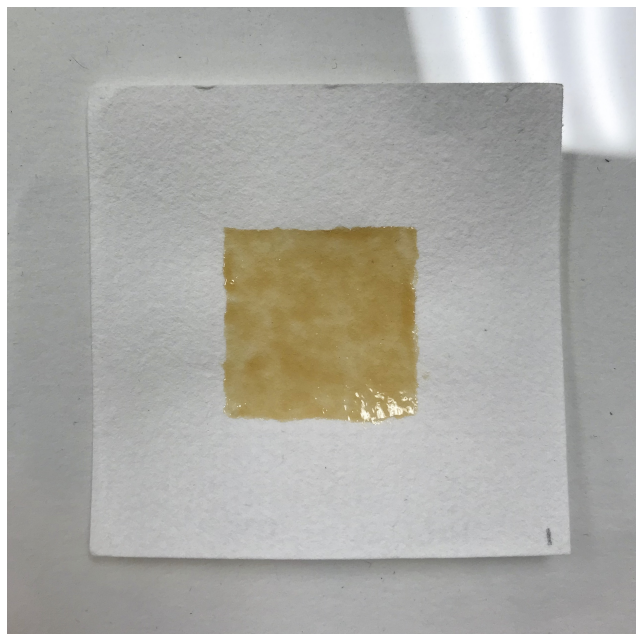


Fig. 1. Flat samples after accelerated aging.

Selected Fluids to Test

Appendix 1 presents suppliers and cost comparisons of selected fluids. All fluids have been made with DI water.

DI Water

In many cases, animal glue can be swelled with water and then mechanically removed. Environmental conditions or additives in the glue recipe can cause the adhesive to become less water soluble over time, making water insufficient for softening the adhesive.

NaCl-Adjusted Water

A solution with conductivity around 2.6 mS/cm² was tested. Tse (2001) notes that the addition of salt enhances the benefits of washing, as the higher ionic strength and conductivity of saline water allows it to draw out higher amounts of free acids from the substrate. Magee (2019) found success softening adhesive that was a mixture of animal protein and starch by raising the conductivity of water used in her recipe for high acyl gellan gum. She added NaCl to DI water, boosting the conductivity of the water up to 2 mS/cm².

Urea

A 3% w/v solution was tested. Hal Erickson (email to the author, November 22, 2018) first brought the use of urea as a small molecule surfactant for animal glue to the attention of the author. Structurally, urea molecules are very similar to protein R-groups, and following the concept of “like dissolves like,” Erickson suggested that urea’s wedge-shaped structure was optimal for opening up the surface area of

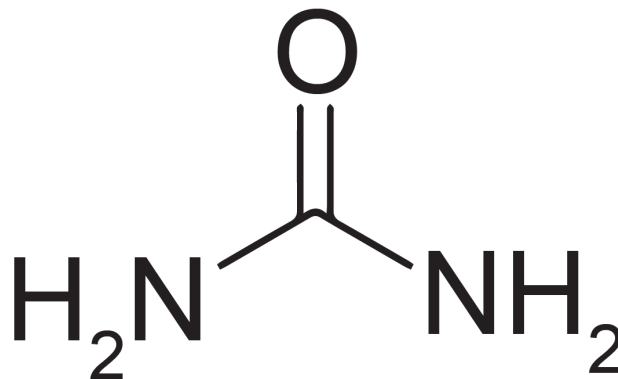


Fig. 2. Wedge-like structure of urea.

protein-based adhesives to water (fig. 2). Historically, urea has been added to animal glue to extend its open time or to make liquid glue at room temperature. Urea has also been commonly used to assess the stability of protein through chemical denaturation, and its ability to promote protein unfolding can be direct, by binding to the protein, or indirect, by altering the solvent environment (Bennion and Daggett 2003, 5142). Erickson (pers. comm., November 26, 2019) suggested applying a solution of 0.5 mol urea directly to the adhesive to improve swelling. Yasmeen Khan (pers. comm., January 28, 2019) also suggested applying a 2%–3% urea solution by brush to the adhesive for animal glue removal, but noted that urea is a hardener for animal glue and can make the adhesive layer more difficult to remove if it has been humidified with urea and allowed to dry. As the wedge-shaped structure of urea may also be capable of opening up the cellulose structure to atmospheric pollutants and oxidative-reductive reactions, Erickson recommended clearing urea after it comes into contact with the substrate, or to switch to another fluid as the adhesive layer becomes increasingly reduced.

Citric Acid

A 3% w/v solution was tested. Citric acid may help with the cleaning process as a chelating agent, binding calcium and metal ions. Besides urea, Khan (email to the author, January 28, 2019) also recommended using citric acid in low concentrations (approximately 3%) to open up the surface of hard, smooth animal glues, pointing out that although urea acted as a hardener for animal glue, the same issue was not found with citric acid. She suggested brushing a solution of either urea or citric acid onto the adhesive, which then breaks down into granules that can then be mechanically removed. As citric acid is acidic, she recommended against letting the solution come into contact with the text block substrate, switching to water when most of the adhesive has been reduced or clearing after use. Chris Stavroudis’s Modular Cleaning Program has

drawn recent interest in the use of citric acid and citrates for paper cleaning, which will likely produce further research on how to clear or neutralize these fluids after use.

Trypsin

Crystal Maitland notes that “since animal glues are not fully soluble (only swellable) unless enzymatically digested, no water-based system (even one with capillary pull like a gellan gum) is going to be able to fully remove the residues” (email to the author, July 23, 2019). Although enzyme use in cleaning and adhesive removal has been well documented for its efficacy, concerns about expense, ease of preparation and use, and negative impact of residues have often dissuaded conservators against its use. Clearing would be required after the use of enzymes. Although enzymes are often considered expensive, trypsin, a digestive endopeptidase commonly extracted from the bovine and porcine pancreas, was found to be surprisingly comparable in cost to other poultice materials and fluid solutions. Trypsin prefers to cleave adjacent to protonated lysine and arginine sites and can require high amounts of Ca^{+2} (approximately 0.02M) to retain activity (Erickson 2018). As specified by Sigma Aldrich, trypsin T0303 (lot #SLBX8983) contained 15,156 units/mg. A solution with a concentration of 400–500 activity units/mL, as recommended for use in gels by Van Dyke (2004), was tested.

Addition of Heat

When animal glue does not swell readily in room temperature water, the addition of heat can often increase its solubility, applied in the form of steam, heating pads, or through heated rigid gels. However, application of heat may be undesirable on parchment text blocks, where it may denature the parchment. On degraded paper text blocks, heat may also cause the substrate to absorb humidification unevenly or too rapidly, causing potential tide lines, or in conjunction with mechanical action cause fiber disruption.

Other Fluids Considered but Not Included in the Experiment

The addition of alcohol (often ethanol or isopropanol) has sometimes been suggested when working with animal glues that do not swell readily in water (Munn 1989). Saliva, which contains amylase and protease as two of the primary active ingredients, is also sometimes suggested where alternate fluids are unsuccessful for adhesive removal. Quandt (1991) describes using saliva with swabs to remove residual adhesive from a parchment text block spine. These fluids were not selected for the experiment due to the difficulty of incorporating them into various delivery methods.

Delivery Method of Fluid

Gellan gum was selected as the delivery method for the testing of the fluids due to its compatibility with all five of the fluids

selected, as well as for its ease of preparation and removal. As gellan gum leaves minimal residue when removed, the characteristics of the animal glue in reaction to the fluid can be observed clearly. A 2% w/v gel was selected, as lower concentrations can be too wet for the substrate, whereas higher concentration gels may be too dry to properly swell the adhesive, and can restrict delivery of fluids with larger molecules such as enzymes. Gellan gum with a thickness of approximately 3 mm was prepared with each of the five fluids selected, and cut into 2.5-cm² squares. Recipes for gellan gum prepared with each fluid are presented in appendix 2.

Although the delivery method may influence the efficacy of the fluid, consideration of optimal delivery methods will be conducted in the next phase of experimentation.

TEST 1: FLUID EFFECT ON ADHESIVE CONSISTENCY

Goal

This part of the experiment aimed to observe the reaction of animal glue to each of the tested fluids over the duration of an hour.

Experiment

- (1) Preparation of test samples and fluids in gellan gum have been previously described in section 3 (also see appendix 2).
- (2) For each sample, a fluid-impregnated piece of gellan gum was placed on top of the animal glue area on flat paper samples. A piece of polyester film and a small acrylic slab were placed on top of the gel. The gel was pressed lightly with fingers to ensure contact with the animal glue was being made and remained in place for an hour. At intervals of 2, 5, 10, 15, 20, 30, and 45 minutes and at the end of the hour, the gel was lifted off at one corner to check the consistency of the animal glue visually and by touch with a microspatula.
- (3) The introduction of heat was also tested for most fluids. Trypsin was tested only at room temperature (RT), as enzymatic response is not optimum at temperatures around 60°C. Heated samples (HT) utilized an 11-cm² gel bead heating pad, heated in the microwave until it reached 60°C and placed on top of the gel in lieu of the acrylic slab. Every 15 minutes, the heating pad was reheated to maintain its temperature.
- (4) This experiment was repeated on two samples for each of the fluid and heat combinations to confirm observations. Although a continuum, the phases of adhesive consistency were identified and described.

Observations

Test 1 demonstrated that the fluids affected the consistency of animal glue adhesive in different but predictable ways.

The duration of contact and increased temperature were significant variables. As moisture was introduced, the adhesive moved from a solid to viscous liquid state. While passing through phases resembling those related to rheology and glass transitions, existing terminology is not specific to situations with increased moisture content. As such, terminology specific to this experiment was devised (fig. 3). Each phase presented risks and benefits to adhesive removal. For example, some fluids resulted in pliable softened phases that were not liquid, suggesting that removal may be possible with reduced risks of tide lines for those fluids. Others rapidly moved to a liquid phase that appeared easy to remove with minimal pressure, suggesting that less fiber damage may result. Not all experiments went through all phases of adhesive consistency change (fig. 4).

DI Water, RT

The surface of the adhesive began swelling at 10 minutes, with a granular consistency forming. At 15 minutes, the animal glue appeared mostly swelled. At 20 minutes and onward, the adhesive appeared swelled throughout. Paper fibers in contact with the gel appeared damp and swelled but without a harsh wet-dry interface forming. After one hour, the gel was slightly discolored, but no visible reduction in the adhesive layer was observed. The consistency of the adhesive remained granular throughout the hour.

DI Water, HT

The surface of the adhesive began swelling at 2 minutes. At 5 minutes, the adhesive appeared mostly swelled. At 10 minutes, some parts of the adhesive began to lose their granular consistency. The adhesive became slippery and could be pushed around with a microspatula at 15 minutes, and started spreading laterally at 20 minutes. At 25 minutes and onward, the adhesive continued to spread laterally, gaining a slightly gloppy consistency. The substrate became visibly damp and swelled, with noticeable lateral migration of water from the gel into the substrate at the end of the hour, but with no hard wet/dry interface formed. After an hour, the gel was more discolored in comparison to the one used at room temperature. There was no significantly visible reduction in the adhesive.

NaCl-Adjusted Water, RT

The surface of the adhesive began swelling at 5 minutes, and appeared swelled throughout by 20 minutes, with a granular consistency. By 30 minutes, the adhesive could be pushed into easily when prodded with a microspatula. The adhesive began to look less granular in consistency around 45 minutes. Where the gel was in contact with the substrate, paper fibers were damp and swollen, but there was no formation of a harsh wet-dry interface. There was no significantly visible reduction in the adhesive.

NaCl-Adjusted Water, HT

After 1 minute, the surface layer of the adhesive began swelling, and appeared swelled throughout by 5 minutes, being easily pushed into with a microspatula. It had a partially granular consistency. Around 10 minutes, the adhesive lost its granular consistency, becoming tacky. The adhesive began spreading laterally around 15 minutes. At 30–45 minutes, spreading of moisture beyond the edges of the gel was observed, although no sharp wet-dry interface was observed. The adhesive started to become gloppy at 45 minutes. After one hour, some amounts of adhesive clung to the gel as it was removed, and the gel was very discolored. After drying, a faint tide line was observed, indicating that some adhesive had solubilized and sunk into paper fibers.

3% w/v Urea, RT

At 5 minutes, the adhesive was mostly swelled, with a granular consistency. At 10 minutes, the adhesive appeared swelled throughout, beginning to lose its granular consistency. At 15 minutes, it gained a slightly more slippery consistency and moved easily when prodded with a microspatula. At 25 minutes, there was visible swelling and humidification of paper fibers where the substrate was in contact with the gel, but no hard wet-dry interface. When the gel was removed, it was only minimally discolored. There was no significantly visible reduction in the adhesive. Although the adhesive became less granular in appearance throughout the hour, it did not fully lose its granular consistency.

3% w/v Urea, HT

The adhesive surface layer began swelling after 1 minute, and appeared almost swelled throughout by 5 minutes, with a partially granular consistency. At 10 minutes, the adhesive appeared swelled throughout. The substrate area in contact with the gel was also visibly swelled and humidified, but without a harsh wet-dry interface. The adhesive began losing its partially granular consistency after 15 minutes, moving easily when prodded with a microspatula and gaining a tacky quality. At 30 minutes and onward, the adhesive became increasingly slippery in consistency. There was visible lateral migration of water beyond the gel area. The adhesive became increasingly wet and gloppy around 45 minutes. After one hour, some adhesive clung to the gel, which was slightly discolored, when it was lifted.

3% w/v Citric Acid, RT

The gellan gum turned opaque and more brittle on addition of the citric acid after heating—it behaved more like a sponge than a gel, and on applying pressure, fluid could be pressed out of the gel (fig. 5). At 1–2 minutes, the surface layer of the adhesive began to soften. Where the gel was in contact with the substrate, the paper was visibly swelled. At 10 minutes,

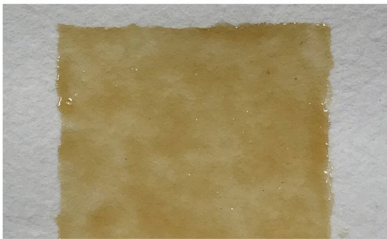





Term and Color Key	Image	Definition
Hard		The adhesive was considered “hard” if the adhesive felt solid or glassy when touched with a microspatula. Complete penetration of the adhesive layer was not possible. The adhesive remains a solid.
Swelled/ Granular		While the adhesive was considered “swelled” when the microspatula could be inserted all the way to the bottom of the adhesive and no part of the adhesive felt hard, the adhesive could continue to swell further and change in consistency. The adhesive is considered “granular” when it is a brittle, rigid gel. When prodded with a microspatula, the adhesive tends to break up into granules.
Partially Granular		As the adhesive continues to swell, it can begin to lose its granular consistency and become more rubbery. Adhesive fragments became smooth or rounded, and bounced back when indented with a microspatula. At this stage it is described as “partially granular.”
Tacky/ Slippery		Sometimes, the adhesive becomes “tacky” or “slippery” with longer exposure to a fluid. In both cases, the adhesive consistency becomes more coherent and gains elasticity. When the adhesive is “tacky,” it often appears sticky and clings to the microspatula when touched. When the adhesive is “slippery,” it has more stringy and wet consistency, feeling less sticky than when “tacky.”
Gloppy		As the adhesive becomes even more wet and loses coherence as a gel, some parts become watery while other parts remain semi-solid. The adhesive is described as “gloppy” at this stage, and smears easily when pressed into the substrate.
Runny		When there are no more semi-solid components to the adhesive and it takes on the consistency of a viscous liquid, it is considered “runny.”

Fig. 3. Phases of adhesive consistency.

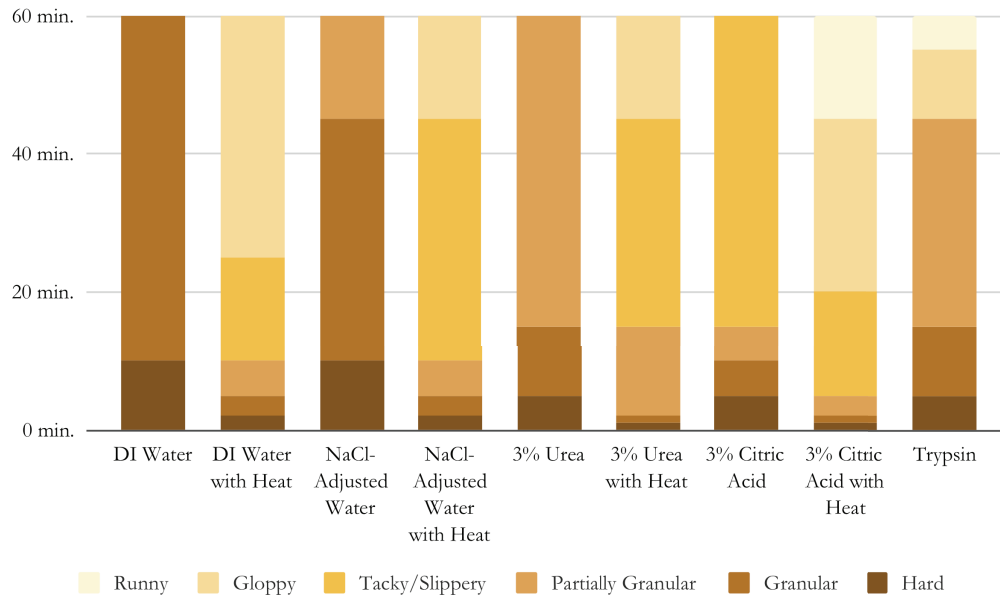


Fig. 4. Comparison of fluid effect on adhesive consistency.

the adhesive appeared mostly swelled but still with a granular consistency. At 15 minutes, the adhesive appeared swelled throughout and lost its granular consistency, becoming tacky. Liquid appeared to be pooling on the top of the gel. At 20 minutes, the adhesive continued to swell and remained tacky when prodded with the microspatula. Lateral spreading of the fluid on the paper increased. From 25 minutes onward, the adhesive continued to swell and become more slippery and could be easily slid around when prodded with a microspatula. Lateral spreading of the fluid on the substrate continued. By one hour, small areas of the adhesive clung to the gel when it was removed, and the gel was quite discolored.



Fig. 5. Gellan gum made with DI water (left) vs. with 3% citric acid (right).

3% w/v Citric Acid, HT

The adhesive began swelling at 1 minute and appeared mostly swelled by 5 minutes, after which moisture began spreading laterally on the substrate beyond the gel area. By 10 minutes, the adhesive had swelled further and gained a tacky consistency. It continued to swell, and at around 20 minutes, the adhesive became gloppy in consistency, sliding around when prodded with a microspatula. It began to become runny around 45 minutes, spreading laterally. When the gel was removed after one hour, the adhesive clung to the gel in several areas. The gel was noticeably discolored, and residual adhesive on the substrate was a viscous liquid (fig. 6). A harsh wet-dry interface was noticeable and dried into a tide line.

Trypsin Solution, RT

Results between samples were inconsistent—some samples showed minimal adhesive reduction, and some samples showed significant adhesive reduction after one hour. Trypsin may not have been distributed evenly when the gellan gum was cast, as the gel was beginning to set when it reached appropriate temperatures to add in the enzyme. The following description is of the sample most noticeably affected by the gel. The adhesive began swelling at 1 minute, and appeared mostly swelled by 5 minutes, at which point it had a granular consistency. By 15 minutes, the paper fibers in contact with the gel area were noticeably swelled, and it appeared that moisture had deeply penetrated into the paper fibers. The adhesive continued to soften and gradually became less granular in consistency; by 45 minutes, parts of the adhesive became gloppy and almost runny. After

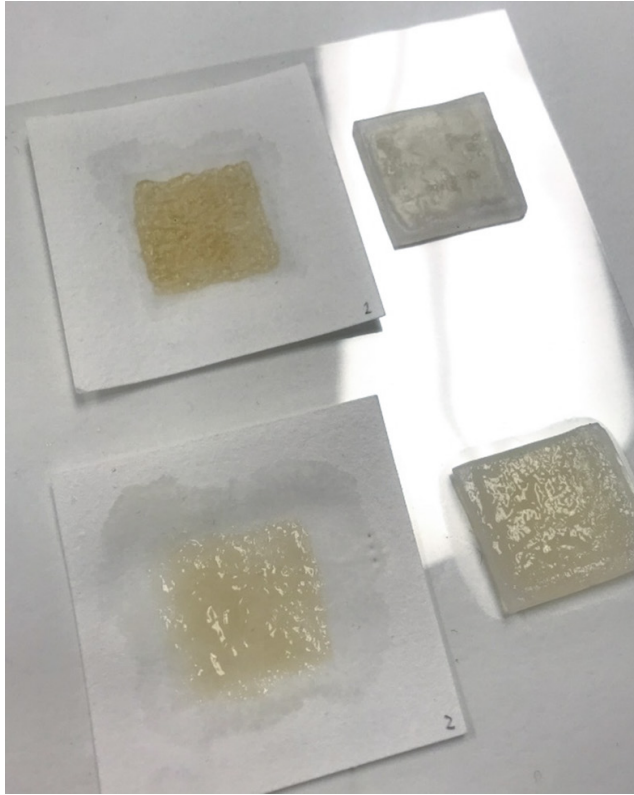


Fig. 6. Samples treated with citric acid at room temperature (top) vs. with heat (bottom).

one hour, some adhesive remained on the paper, but a large portion had been removed. Of all of the fluids tested, it was most clear that the gel with trypsin had absorbed some solubilized adhesive rather than simply having adhesive cling onto the surface of the gel (fig. 7). After drying, there

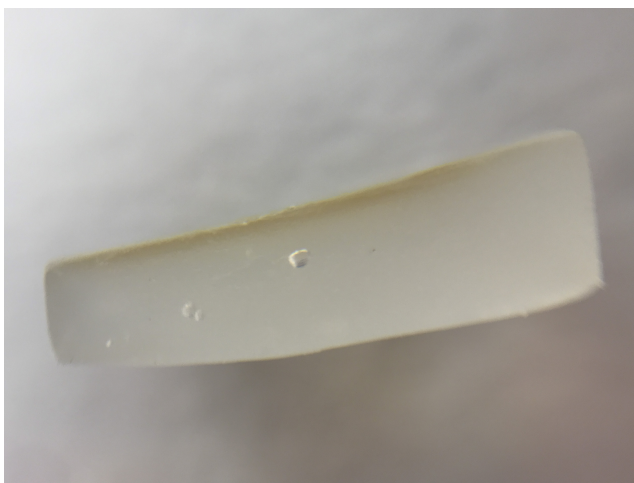


Fig. 7. Gellan gum with trypsin with visible adhesive absorption.

was a faint tide line where the gel had been placed on the substrate, indicating that some adhesive had solubilized and sunk into paper fibers (fig. 8).

TEST 2: FLUID EFFECT ON ADHESIVE REMOVAL

Goal

In the previous test, the changes in adhesive consistency suggested that the difficulty of adhesive removal and risks such as fiber disturbance and formation of tide lines may not be linear or the same for all fluids. Building on that information, this test attempts to discover what stage of adhesive consistency was the easiest to remove and with the least risk for the object for each fluid/heat combination.

Experiment

This experiment simulates removal of adhesive when multiple cycles of poultice are used. The initial heavy layer of adhesive is often removed after the first poultice (P1), and then a thinner, residual layer is removed in a second poultice (P2). Durations of each poultice have been expressed here in parentheses after the poultice abbreviation—for example, P1 (5 min.) indicates a first poultice with a duration of 5 minutes:

- (1) Preparation of test samples and gellan gum prepared with each fluid have been previously described in section 3 (also see appendix 2).
- (2) Various fluids were applied via gellan gum placed on top of the animal glue area of flat paper samples. The fluid/gum was left in place undisturbed for different durations of time (5, 10, 15, 20, 30, and 45 minutes). Where noted, heat was applied with an 11-cm² gel bead heating pad, heated in the microwave until it reached 60°C and placed on top of the gel. Where applicable, the heating pad was reheated every 15 minutes to maintain its temperature. At the designated time interval, adhesive removal was attempted using a microspatula.
- (3) The ease of removal after P1 was observed and rated on a scale from 0 to 6, which indicates how much adhesive was removed, the type of residue left, how much pressure was required, and other risks (fig. 9).
- (4) After the initial adhesive removal phase, P2, a second, fresh application of the same fluid/gel/heat combination was applied for a standard 15 minutes to half of the cleaned adhesive area, and removal was attempted with a microspatula. This meant that samples that had adhesive cleaned after P1 (5 min.) had the remaining adhesive exposed to an additional 15 minutes (P2), and that samples cleaned after P1 (45 min.) also had an additional 15 minutes (P2).
- (5) After drying, the half of the adhesive sample that had only been treated with P1 was compared with the half that had been further treated with P2.



Fig. 8. Test 1 and 2 fluid experiments.








0		No adhesive could be removed
1		Small amounts of adhesive could be removed, high pressure required
2		Majority of adhesive layer was removed with some solid residue, high pressure required
3		Majority of adhesive layer was removed with some solid residue, slight pressure required
4		Majority of adhesive layer was removed with minimal residue, slight pressure required
5		Majority of adhesive layer was removed with minimal residue, no pressure required
6		Paper was too wet or the adhesive became too messy to remove, increasing risk of tidelines, adhesive sinking, and paper fiber damage

Fig. 9. Scale for ease of P1 adhesive removal.

Observations

At room temperature, the optimal time for adhesive removal after P1 with each fluid appeared to range between 20 and 30 minutes. The addition of heat accelerated the swelling of the adhesive so that 5 minutes of P1 with any fluids was adequate for easy adhesive removal. Given adequate time, all fluids were able to swell the adhesive enough for removal of the distinct adhesive layer on top of the substrate, but discoloration remained in the substrate where the adhesive had been, indicating that some adhesive had sunk into the paper fibers either on application, during aging, or while being softened.

At room temperature and with heat, continued exposure of fluid to the adhesive after it became swelled changed adhesive consistency in ways that affected ease of removability. Several of the fluids were so successful during P1 that the majority of the adhesive layer was already removed and P2 was not necessary for further adhesive removal. With some fluids, although a distinct adhesive layer was no longer present after P1, P2 helped in reducing the residual adhesive discoloration. With other fluids, P2 was detrimental to the treatment after the majority of adhesive had already been removed—the sample rapidly became too wet and potentially damaged with the additional poultice.

The ease of adhesive removal after P1 was observed and rated on a scale from 0 to 6, and color coded based on the ease of removal from dark red (most difficult to remove) to dark green (easiest to remove). Where damage occurred due to tide lines, adhesive sinking, or paper fiber damage, gray was used as the color code (see fig. 9). These observations are recorded in figure 10.

DI Water, RT

After P1 (5–20 min.), significant amounts of adhesive residue remained that could not be removed even when pressure was

applied with a microspatula. P2 swelled remaining adhesive residue, which could be removed without pressure after 15 minutes. As the majority of adhesive had been removed after P1 (30, 45 min.), P2 did not further remove significant amounts of adhesive. No visible tide lines or adhesive sinking was observed after P2 for all durations.

DI Water, HT

After all application durations of P1, most of the adhesive had been removed with a microspatula. In all tests with P2, lateral migration of moisture created tide lines. Especially after P1 (30, 45 min.), the paper was so wet that even light pressure with a microspatula after P2 could damage paper fibers.

NaCl-Adjusted Water, RT

Significant amounts of adhesive that remained after P1 (5, 10 min.) were not sufficiently swelled after the P2 to be easily removed. After P1 (15, 20 min.), some amounts of adhesive residue remained. These were able to be removed after P2 with slight pressure. Most of the adhesive layer was removed after P1 (30, 45 min.), and P2 did not further remove significant amounts of adhesive. After drying, where the adhesive had been exposed to P1 for durations of 15 minutes or longer, P2 appeared to reduce the discoloration on the substrate left by the adhesive. No tide lines were observed after P2 for all durations.

NaCl-Adjusted Water, HT

After all application durations of P1, the majority of the adhesive had been removed with a microspatula. For P1 (5 min.), a small amount of adhesive was further removed with a microspatula using light pressure after P2. In all other instances, no significant adhesive layer existed to remove after P2. After drying, where the adhesive had been exposed to the

	DI water, RT	DI water, HT	NaCl-adjusted water, RT	NaCl-adjusted water, HT	3% Urea, RT	3% Urea, HT	3% Citric acid, RT	3% Citric acid, HT	Trypsin solution, RT
5 min	1	4	1	4	2	5	2	5	2
10 min	1	4	1	5	3	5	2	5	3
15 min	2	5	2	5	3	5	3	5	3
20 min	3	5	2	5	4	5	3	5	3
30 min	4	6	4	5	4	5	5	6	4
45 min	4	6	4	5	4	5	5	6	5

Fig. 10. Ease of adhesive removal with a microspatula after P1.

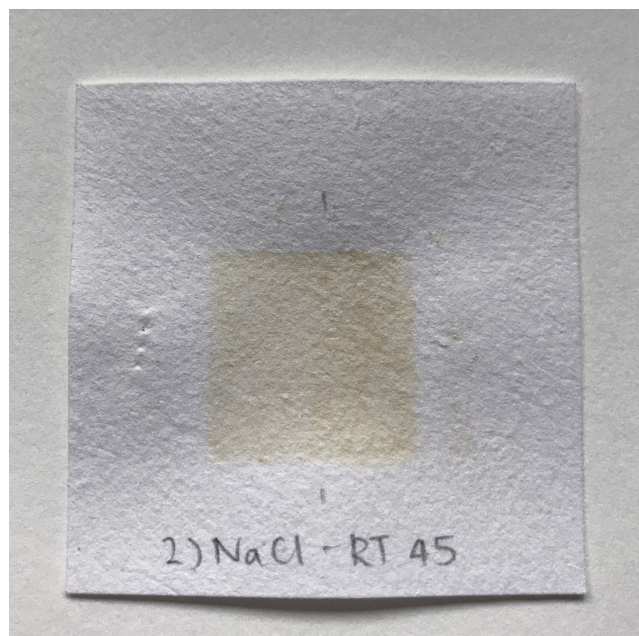


Fig. 11. Slight reduction in discoloration after P2 (45 min.), on the left side of sample, from P1 (45 min.), on the right side of the sample, using NaCl-adjusted water at room temperature.

P1 for durations of 15 minutes or longer, P2 appeared to reduce the discoloration on the substrate caused by the adhesive (fig. 11). No tide lines were observed after the application of P2 for all durations.

3% w/v Urea, RT

Small amounts of adhesive residue that could not be removed after P1 (5, 10 min.) were removed after P2, although pressure was required to remove areas with heavier adhesive residue. For P1 (15 min.) samples, remaining adhesive residue was fully softened with P2 and could be removed easily. As the majority of adhesive had been removed after P1 (20–45 min.), P2 did not further significantly reduce adhesive amounts. After drying, where the adhesive had been exposed to P1 for durations of 10 minutes or longer, P2 appeared to reduce the discoloration on the substrate caused by the adhesive. No visible tide lines were observed after the application of P2 for all durations.

3% w/v Urea, HT

After all application durations of P1, the majority of the adhesive had been removed with a microspatula, and no significant amount of adhesive was further removed with P2. After drying, where the adhesive had been exposed to P1 (5–20 min.), P2 appeared to reduce the discoloration on the substrate caused by the adhesive. For samples exposed to P1 (30, 45 min.), P2 did not appear to reduce discoloration on the substrate. No visible tide lines were observed after the application of the P2 for all durations.

3% w/v Citric Acid, RT

Small amounts of adhesive residue that could not be removed after P1 (5, 10 min.) were removed after P2, although pressure was required to remove areas with heavier adhesive residue. Adhesive residue left on the samples after P1 (15, 20 min.) were further reduced after P2 with less pressure. For P1 (5–20 min.), no visible tide lines were observed after P2. After exposure to P1 (30, 45 min.), the majority of adhesive had already been removed. In these samples, there was no significant distinct adhesive layer to remove after P2, and a faint tide line was visible beyond gel areas after drying.

3% w/v Citric Acid, HT

After all application durations of P1, the majority of the adhesive had been removed with a microspatula, and no significant adhesive layer existed for removal after P2. Lateral migration of moisture beyond gel areas was observed for all durations with P2, resulting in tide lines. Where P1 had been applied for 15 minutes or longer, the paper was so moist after P2 that slight pressure with a microspatula could cause fiber damage (fig. 12). After P1 (30, 45 min.), the adhesive sunk into the paper, and P2 may have absorbed some of the sunk adhesive as the substrate appeared slightly less discolored after drying.

Trypsin Solution, RT

Small amounts of adhesive residue that could not be removed after P1 (5, 10 min.) with the first poultice were removed after P2, although slight pressure was required to remove areas with heavier adhesive residue. In all other instances, the adhesive layer was mostly removed after P1, with negligible

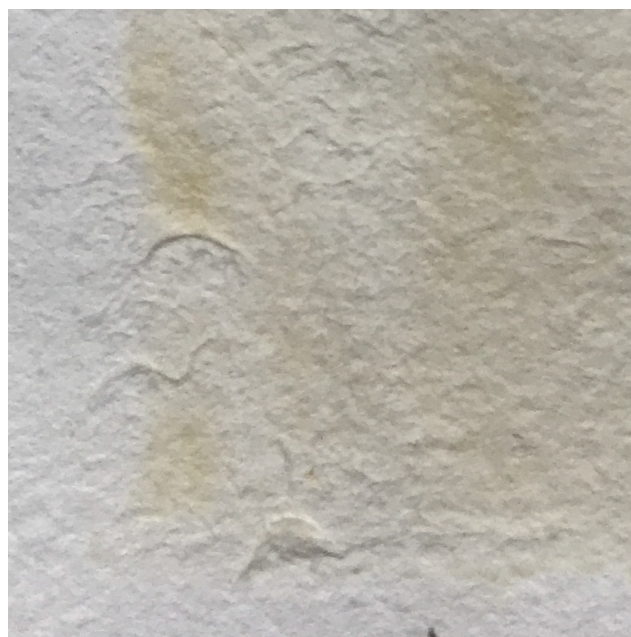


Fig. 12. Fiber damage from overwetting.

amounts of residue remaining for P2. Lateral spreading of moisture and/or sinking of adhesive resulting in tide lines occurred after P2 for all samples.

CONCLUSION

Speed of Adhesive Swelling

At room temperature, adhesive poulticed with DI water and the NaCl-adjusted water were the slowest to reach a removable state. Adhesive was difficult to remove until around 20 minutes and at that point still required high pressure and left significant residues. At room temperature, citric acid, urea, and trypsin were easy to remove at around 10–15 minutes, requiring light or minimal pressure and leaving minimal residues. For all fluids, the speed of adhesive swelling was significantly increased with the addition of heat, reducing the necessary poultice duration down to 5–10 minutes. It should be noted that the length of time required for swelling the adhesive increases in relation to the thickness of the adhesive, so the recorded times for these experiments may not correlate exactly with actual treatments.

Effect of Fluid on Adhesive Consistency and Absorbance in Gellan Gum

Except with trypsin, adhesive on test samples retained a mostly cohesive structure (granular or tacky/slippery) even with prolonged exposure to all room temperature fluids. With the addition of heat to all fluids, the adhesive became increasingly liquid-like after prolonged poultices. For instance, DI water at room temperature was the least successful at solubilizing the adhesive, which even when swelled throughout remained brittle and granular. With the addition of heat to DI water, the adhesive began losing its granular consistency after 10 minutes and became increasingly gloppy from 25 minutes onward. Urea and citric acid improved the rate of solubility in similar ways, although citric acid appeared to be slightly more successful at solubilizing the adhesive. It is interesting to note that adhesive exposed to urea took longer to dry and reharden than with other fluids. Although exposure to urea may reduce the resolubility of adhesive once it has dried after poulticing, there is a longer working time for adhesive removal while it remains softened. Although results between samples were inconsistent, trypsin was the only fluid capable of making the adhesive runny at room temperature.

As the duration of gel to adhesive contact increased in Test 1 experiments, the gel became increasingly discolored, indicating that some solubilized components of the adhesive had been absorbed by the gel. After prolonged poulticing, significant discoloration in gels at room temperature containing NaCl and citric acid, as well as where heat was applied, suggest that these fluids and the addition of heat increase the success of solubilizing components of the adhesive. For Test 1 experiments, DI water at room temperature resulted in the least adhesive reduction without mechanical action, and trypsin at room

temperature resulted in the highest reduction, with heated citric acid coming in second (fig. 14). In Test 2 experiments, P2s with NaCl-adjusted water and urea were more successful than other fluids in reducing the discoloration of the substrate after the majority of adhesive had been removed in P1. This suggests that the addition of NaCl or urea may also increase the success of solubilizing the adhesive, as more of the adhesive has been absorbed by the gel after poulticing.

Effect of Fluid on Substrate

With both Test 1 and 2 experiments at room temperature, no harsh wet-dry interfaces or lateral migration of the fluid occurred with DI water, NaCl-adjusted water, or urea. Lateral migration of the fluid beyond gel areas occurred with citric acid at room temperature, as well as with the addition of heat to other fluids. This suggests that lateral spread of water into the paper occurs faster and more extensively with heat than at room temperature, which can increase tide line risks and damage to paper fiber during mechanical removal of adhesive. Trypsin also had adverse effects on the substrate—although no lateral migration of the fluid beyond gel areas was observed, the fluid and solubilized adhesive penetrated deeply into paper fibers, resulting in visible tide lines where the gel was placed. And although minimal tide lines were observed on samples treated with urea, a noticeable precipitate formed on dried pieces of gellan gum after use, further suggesting that clearing is necessary after any direct contact of urea with the substrate.

In some samples for Test 2 experiments with DI water, HT, citric acid, RT and HT, and trypsin, RT, most of the adhesive layer was removed after P1. For these samples, P2 was more likely to make the substrate too wet for safe mechanical manipulation, develop tide lines, or become discolored. More fluid was introduced to the substrate with less adhesive to absorb the bulk of the moisture, and it is likely that remaining adhesive residues after P1 were solubilized during P2 and sank into the substrate. However, at both room temperature and with heat, P2s with NaCl-adjusted water and urea reduced the discoloration of the substrate after the majority of adhesive had been removed in P1, suggesting that these fluids allowed the gel to absorb small amounts of further-solubilized adhesive residue rather than depositing them further into the substrate, reducing the risk of tide lines and improving discoloration.

Ease of Adhesive Removal vs. Risk of Damage

Test 1 and 2 results suggest that in different treatment circumstances, some fluids may be more suitable than others. Although more pressure is required during mechanical removal of adhesive in a granular state and less pressure is required as the adhesive progresses toward the more liquid, runny state, removal of adhesive at both ends of the spectrum presents pros and cons. Where the adhesive remains in a more cohesive, gelatinous state throughout poulticing, such as with DI water and NaCl-adjusted water at room temperature, there appears

Fluid (in Gellan Gum)	Speed of Adhesive Swelling	Effect on Adhesive Consistency/Gellan Gum	Effect on Substrate	Additional Comments
DI water, RT	Slowly swelled, ~20 minutes	<ul style="list-style-type: none"> Remained granular throughout poulticing Gel became slightly discolored 	<ul style="list-style-type: none"> Pressure mostly required with mechanical removal Low risk of over-wetting and tidelines 	<ul style="list-style-type: none"> The slowest method tested, may be appropriate for removal of final residues, when overwetting is most likely
DI water, HT	Softened quickly, ~5-10 minutes	<ul style="list-style-type: none"> Became gloppy after prolonged poultices Began spreading laterally after 20 minutes Gel became more discolored than at room temperature 	<ul style="list-style-type: none"> Minimal pressure required with mechanical removal Lateral migration of fluid beyond gel area Risk of over-wetting and tidelines after prolonged poulticing 	<ul style="list-style-type: none"> More prone to tidelines than at room temperature
NaCl-adjusted water, RT	Slowly swelled, ~20 minutes	<ul style="list-style-type: none"> Remained partially granular throughout poulticing Gel became slightly discolored 	<ul style="list-style-type: none"> Pressure mostly required with mechanical removal Low risk of over-wetting and tidelines 	<ul style="list-style-type: none"> Not significantly different from DI water (RT)
NaCl-adjusted water, HT	Softened quickly, ~5 minutes	<ul style="list-style-type: none"> Became gloppy after prolonged poulticing Began spreading laterally around 15 minutes Clung to gel after an hour Gel became very discolored 	<ul style="list-style-type: none"> Minimal pressure required with mechanical removal Lateral migration of fluid beyond gel area Risk of tidelines and adhesive sinking into paper fibers after prolonged poulticing 	<ul style="list-style-type: none"> More discoloration in gel indicates more solubilization of adhesive
3% Urea, RT	Softened at moderate pace, ~10 minutes	<ul style="list-style-type: none"> Remained partially granular throughout poulticing Gel became slightly discolored 	<ul style="list-style-type: none"> Slight pressure required with mechanical removal Low risk of over-wetting and tidelines 	<ul style="list-style-type: none"> May require clearing Adhesive retains moisture after swelling for an extended period of time, but will become less resolvable after drying
3% Urea, HT	Softened quickly, ~5 minutes	<ul style="list-style-type: none"> Became gloppy after prolonged poulticing Easily removed after about 5 minutes Gel became slightly discolored 	<ul style="list-style-type: none"> Minimal pressure required with mechanical removal Lateral migration of fluid beyond gel area Slight risk of tidelines 	<ul style="list-style-type: none"> May require clearing Adhesive remained swelled for an extended period of time, but will become less resolvable after drying
3% Citric acid, RT	Softened at moderate pace, ~15 minutes	<ul style="list-style-type: none"> Became tacky/slippery after prolonged poulticing Clung to gel after an hour Gel became very discolored 	<ul style="list-style-type: none"> Slight pressure required with mechanical removal Lateral migration of fluid beyond gel area Risk of tidelines 	<ul style="list-style-type: none"> May require clearing
3% Citric acid, HT	Softened quickly, ~5 minutes	<ul style="list-style-type: none"> Became runny after prolonged poulticing, spreading laterally Clung to gel after 1 hour Gel became very discolored 	<ul style="list-style-type: none"> Minimal pressure required with mechanical removal Lateral migration of fluid beyond gel area Risk of tidelines, over-wetting, and adhesive sinking 	<ul style="list-style-type: none"> May require clearing Most effective at solubilizing the adhesive, but also the most risks
Trypsin, RT	Softened at moderate pace, ~10-15 minutes	<ul style="list-style-type: none"> Some areas became almost runny after prolonged poulticing Gel absorbed some adhesive after 1 hour Gel became slightly discolored 	<ul style="list-style-type: none"> Slight pressure required with mechanical removal Risk of tidelines, over-wetting, and adhesive sinking 	<ul style="list-style-type: none"> Only fluid at room temperature to significantly solubilize the adhesive May require clearing Gellan gum may have impacted mobility of enzymes

Fig. 13. Summary of fluid experiments.

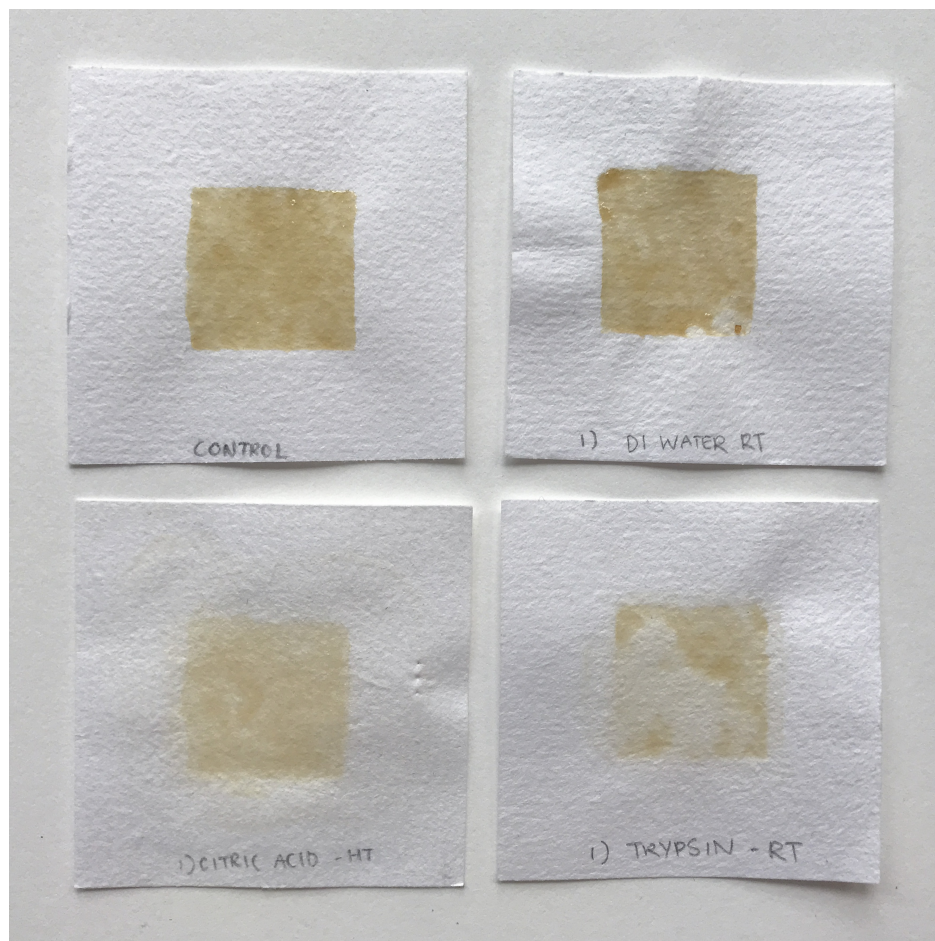


Fig. 14. Comparison of adhesive reduction after Test 1 experiments (clockwise): control; DI water, RT; trypsin, RT; and citric acid, HT

to be less risk of adhesive sinking, lateral migration of the fluid resulting in tide lines, or risk of damage to paper fibers through overwetting in combination with mechanical removal. As such, although more pressure is required when using these fluids, they may be preferable for working with thinner layers of adhesive, as well as with substrates that are heat or water sensitive, or have poor wet strength.

Urea, citric acid, and trypsin at room temperatures, and the addition of heat to all fluids, were more successful at solubilizing the adhesive and rendering it to a more liquid state. This makes the adhesive easier to remove mechanically with little to no pressure on the spatula, reducing the risk of paper fiber disruption. As the adhesive became gloppy, it became possible to gently wipe away the adhesive rather than using a scraping motion. However, the more solubilized the adhesive, the more risks of tide lines and adhesive sinking. Furthermore, when the adhesive became too runny, as with prolonged exposure to citric acid with the addition of heat, mechanical removal became more challenging, as the adhesive smeared into the substrate very easily. Although less

pressure is required with the addition of heat or when using urea, citric acid, and trypsin, they may be preferable for working with thicker layers of adhesive, as well as with substrates that are more highly sized or less hydrophilic.

Although no single fluid excelled above all others with consideration to different treatment circumstances, experimental results have guided the formation of a fluid selection approach when removing animal glue. Poulticing with DI water should be tested first to see if water alone is sufficient to swell the adhesive. If the adhesive is not sufficiently swelled and the substrate is not heat sensitive or severely degraded, the addition of heat should then be considered. The tests confirm that heat will dramatically speed the softening process and reduce the need for mechanical manipulation. However, the conservator should keep in mind that heat will also increase the risk of tide lines and adhesive sinking, especially when used on residual adhesive.

When removing particularly heavy adhesive layers, using one of the faster working, heated fluids is most expedient—heated citric acid was the fastest. If heat cannot be used on the treatment, urea and citric acid should be tested, as they were the most

successful poultices at room temperature.² To prevent urea or citric acid from contacting the substrate and leaving potentially harmful residues, room temperature NaCl-adjusted water may be used to remove the final adhesive layer. Removing the final adhesive layer and adhesive that has sunk into the paper requires a slower acting fluid—NaCl-adjusted water was seen as the best option for that because it was slow and the gel was discolored, implying that it drew more adhesive out of the paper than did DI water. The sodium component may also help in neutralizing citric acid residues in the substrate. These fluids, used in combination, should maximize efficiency of bulk adhesive removal while providing the safest and most complete cleaning option for the residual adhesive layer.

Through the fluid experiments, it became clear that mechanical removal is necessary to successfully reduce animal glue softened with each of the tested fluids in gellan gum. The next step in experimentation would be to test different delivery methods to see if there are application methods where the solubilized adhesive is more successfully absorbed into the delivery method, therefore requiring less mechanical action. Sequentially applying heated citric acid and room temperature NaCl-adjusted water in gellan gum was the most successful for removing heavy adhesive layers on flat paper in the fluid experiments. To maximize the efficacy of this combination, further examination of the fluid's delivery method, as well as working on the sculptural form of a book spine, is needed. As such, delivery method experiments should be tested on bound samples to determine the success of each delivery method on a three-dimensional surface.

FURTHER STUDY: TESTING OF DELIVERY METHODS

Although further studies could not be undertaken at this time due to the Covid-19 pandemic, future testing of delivery methods will optimize the ease of adhesive removal and reduce risk of damage to the substrate during adhesive removal. These risks, identified during the fluid experiments, include adhesive sinking, tide lines, and paper fiber damage caused by overwetting in combination with mechanical action. Clearing of potential residues from the adhesive removal process will also be examined.

Bound samples for experimentation with delivery methods were prepared using the same paper and animal glue and then aged at the Library of Congress at the same time as the flat fluid experimentation samples (fig. 15). As the limited quantity of bound samples and time constraints make testing of delivery methods in combination with each of the fluids from the fluid experiments impractical, fluid choices for this part of the experiment will be narrowed down to DI water and citric acid. DI water was selected because it is the most common and routine poultice fluid for animal glue removal. Citric acid was selected because it was the most successful in the fluid experiments for reducing thick adhesive layers.

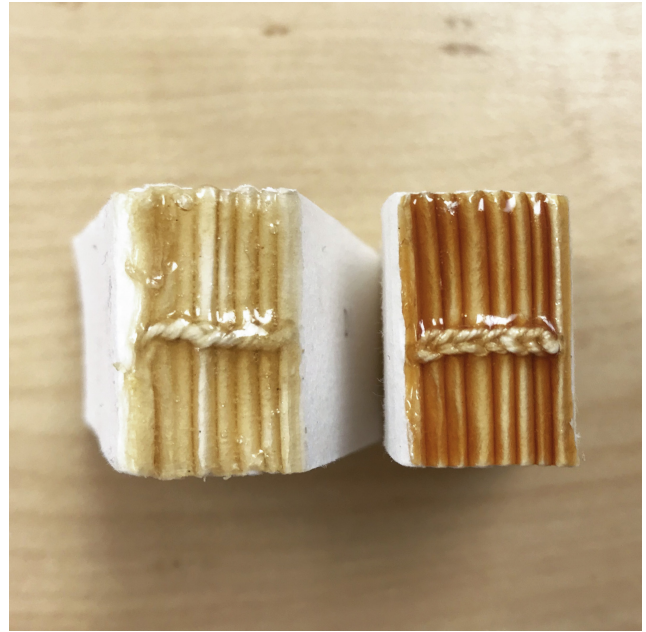


Fig. 15. Bound samples before (left) and after (right) accelerated aging.

Delivery methods currently considered for testing include wheat starch paste, methyl cellulose, rigid gels including low acyl gellan gum, agarose, and Nanorestore Peggy 6, and application of a neat solvent by brush. Although wheat starch paste and methyl cellulose are perhaps the most commonly used poultice materials for spine adhesive removal, the range of fluids that can be incorporated into paste is limited, and methyl cellulose may sometimes be too wet, risking tide lines and overwetting the substrate. The use of rigid gels including gellan gum, agarose, and Peggy 6 are particularly appealing for cleaning treatments, as they absorb solubilized degradation products into the gel network via capillary action so that theoretically no mechanical action is required (Hughes and Sullivan 2016). Reduction of mechanical action during spine adhesive removal would reduce risk of spine fold damage, especially if the text block paper is deteriorated or has poor wet strength. Peggy 6, a poly(vinyl) alcohol gel, is of particular interest for spine adhesive removal, being flexible and elastic with good adherence to very rough and irregular surfaces, as well as stable at high temperatures. Both Erickson (pers. comm., November 22, 2018) and Khan (pers. comm., January 1, 2019) recommended application of urea and citric acid by brush in combination with mechanical action using a microspatula or swab for animal glue removal, noting that these two fluids work on the surface of the adhesive.

In combination with the results of the fluid experiments, delivery method experiments should provide conservators with direct comparisons between these materials and techniques that should help the conservator predict potential treatment issues and results during removal of animal glue on book spines.

ACKNOWLEDGMENTS

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Appendix 1. Fluid Suppliers and Cost Comparisons

Fluid	Supplier	List Price	Cost/L
DI water	Northwestern University Libraries Conservation Lab	N/A	N/A
NaCl, CAS 7647-14-5 (ACS reagent grade)	Calbiochem, via Sigma Aldrich	\$34.20/500 g	For a conductivity of approximately 2.6 mS/cm ² , \$0.068/L
Trypsin T0303	Sigma Aldrich	\$125/g	For 0.033 g/L, \$4.125/L
Urea 99.5% for analysis	Acron Organics, via Fisher Scientific	\$52.15/1 kg	For a 3% solution, \$1.56/L
Citric acid monohydrate	Sigma Aldrich	\$75.5/1 kg	For a 3% solution, \$2.265/L

Appendix 2. Recipes for Fluid Experiments

Fluid	2% Gellan Gum Recipe	Cooking Instructions/Additional Notes
DI water	<ul style="list-style-type: none"> • 100 mL DI water • 0.04 g calcium acetate • 2 g gellan gum 	Dissolve the calcium acetate in DI water. Add the gellan gum to the water while whisking. Heat in the microwave until fully dissolved. Pour into a tray to cool and set.
NaCl- adjusted water	<ul style="list-style-type: none"> • 100 mL DI water • 0.04 g calcium acetate • 0.1 g NaCl • 2 g gellan gum 	Follow the preceding instructions, dissolving NaCl in the DI water before adding in the gellan gum.
3% w/v urea	<ul style="list-style-type: none"> • 100 mL DI water, divided • 0.04 g calcium acetate • 3 g urea • 2 g gellan gum 	Dissolve urea in 10 mL of DI water and put aside. Prepare gellan gum with the remaining water as usual. After the gellan gum has been dissolved and removed from heat, stir in the urea solution. Pour into a tray to cool and set.
3% w/v citric acid	<ul style="list-style-type: none"> • 100 mL DI water, divided • 0.04 g calcium acetate • 3 g citric acid • 2 g gellan gum 	Follow instructions for the urea gellan gum, replacing urea with citric acid. The formed gel is opaque, white, and more brittle than other gels. The gel feels more like a sponge than a true gel—when pressure is applied, liquid is expelled from the gel, pooling up at the top or bottom of the gel.
Trypsin	<ul style="list-style-type: none"> • 100 g DI water adjusted with calcium hydroxide to pH 7.5, divided • 0.04 g calcium acetate • 0.0065 g Trypsin • 2 g gellan gum 	Dissolve the trypsin in 10 mL of pH-adjusted DI water. Prepare gellan gum with the remaining water as usual. After the gellan gum has been dissolved and removed from heat, stir the gel until it cools to 40°C. Stir in the trypsin solution. Pour into a tray to cool and set.

NOTES

1. During discussions with Fenella France, chief of the Preservation Research and Testing Division, France (email to the author, April 30, 2019) noted that 80°C was a high temperature, with practices at the Library of Congress tending toward lower temperatures to reduce generating samples dissimilar to real-life circumstances. Nevertheless, a decision was made to continue with these parameters

after Andrew Davis (email to the author, April 30, 2019) pointed out that they were what would be used for the aging of standard Library of Congress ISR/CLASS paper samples.

2. Although at room temperature trypsin showed the most success at solubilizing animal glue, it also created the most tide lines and adhesive sinking, and effective clearing of enzymes is debated. As such, trypsin is not recommended except as a last resort.

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SOURCES OF MATERIALS

- Old Master Papers in #57, Frobisher (White)
St Armand, via Talas
330 Morgan Ave.
Brooklyn, NY 11211
212-219-0770
<https://www.talasonline.com/St-Armand-Old-Master-Papers>
Gellan Gum and Ground Hide Glue
Talas
330 Morgan Ave.
Brooklyn, NY 11211
212-219-0770
<https://www.talasonline.com/>
- Citric Acid Monohydrate (CAS 5949-29-1) and Trypsin from Porcine Pancreas (CAS 9002-07-7)
Sigma Aldrich Corp.
St. Louis, MO 63178
800-325-3010
<https://www.sigmaaldrich.com/>
- OmniPur Sodium Chloride (CAS 7647-14-5)
Calbiochem, via Sigma Aldrich Corp.
St. Louis, MO 63178
800-325-3010
<https://www.sigmaaldrich.com/catalog/product/mm/7710op?lang=en®ion=US>
- Urea, 99.5% for Analysis (CAS 57-13-6)
Acros Organics, via Fisher Scientific
300 Industry Dr.
Pittsburgh, PA 15275
724-517-1500
<https://www.fishersci.com/shop/products/urea-99-5-analysis-acros-organics-3/AC140750010>
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