Theme Articles
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Updating conservation techniques for paleontology collections associated with Florissant Fossil Beds National Monument

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ABSTRACT

Florissant Fossil Beds National Monument (FLFO) was established to protect the rare abundance and diversity of fossil resources preserved in the Florissant Formation. The majority of fossils are plants and insects preserved in laminated shale, which is prone to conservation issues. These issues result from the inherent thinness of individual laminae (≥0.1 mm) and high clay content, and, during collection, sharp fluctuations in relative humidity (RH) and moisture content. The purpose of this paper is to describe historical and current stabilization methods, and report on two current research efforts at separate institutions to mediate these issues using controlled drying techniques and selection of appropriate adhesives and consolidants following best practices in fossil preparation. Response of shale units to humidity is being investigated at the Western Archeological and Conservation Center facility in Tucson, Arizona, along with the viability of consolidation with tetraethyl orthosilicate (TEOS). This method controls humidity and moisture during drying procedures in order to prevent cracking, flaking, and delamination of the shale matrix for both untreated and treated samples. Results indicate that consolidation with TEOS can prevent damage from occurring during the drying process if the samples are maintained in a hydrated state while the TEOS forms a sol-gel. Without treatment, a slow and moderate drying cycle longer than 14 days without acute drops in (RH) is necessary to prevent damage. To test appropriate adhesives and consolidants that assist in preventing and repairing damage to fossil specimens, analysis is being performed at the National Center for Preservation Technology and Training in Natchitoches, Louisiana. The properties of two ethyl-cyanoacrylates, a medical-grade butyl-octyl cyanoacrylate, and Butvars B-98 and B-76 in 95% ethanol were compared against each other after treated paper shale samples were stressed under accelerated aging conditions in a weatherometer and analyzed using Fourier Transform Infrared Spectroscopy (FTIR). Results indicate that the paper shale samples treated with a consolidant using the two Butvar resins remain the most stable over time under extreme conditions. Updated methods of drying shale after extraction and using appropriate adhesives and consolidants will ensure conservation of the paleontological specimens at FLFO for ongoing research and public understanding into the future.

INTRODUCTION

Florissant Fossil Beds National Monument (FLFO) was established in 1969 to protect the abundant fossil resources in the upper Eocene (Chadronian) Florissant Formation. These fossils include large, petrified stumps as well as intricately preserved plants, insects, and vertebrates in lacustrine beds, intergrading from thin, laminated "paper" shale to thicker siltstone. The fossil assemblage includes specimens of approximately 1,800 taxa that have been collected and studied since the 1870s (Meyer 2003). Paper shale is vulnerable to various conservation issues due largely to its high clay content and thinness, and is susceptible to many types of deformation. The purpose of this paper is to describe

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historical and current stabilization methods, and report on how two new research efforts mediate deformation and conservation issues: development of controlled drying techniques, and determination of appropriate consolidants that conform to best practices in fossil preparation for use by the museum community.

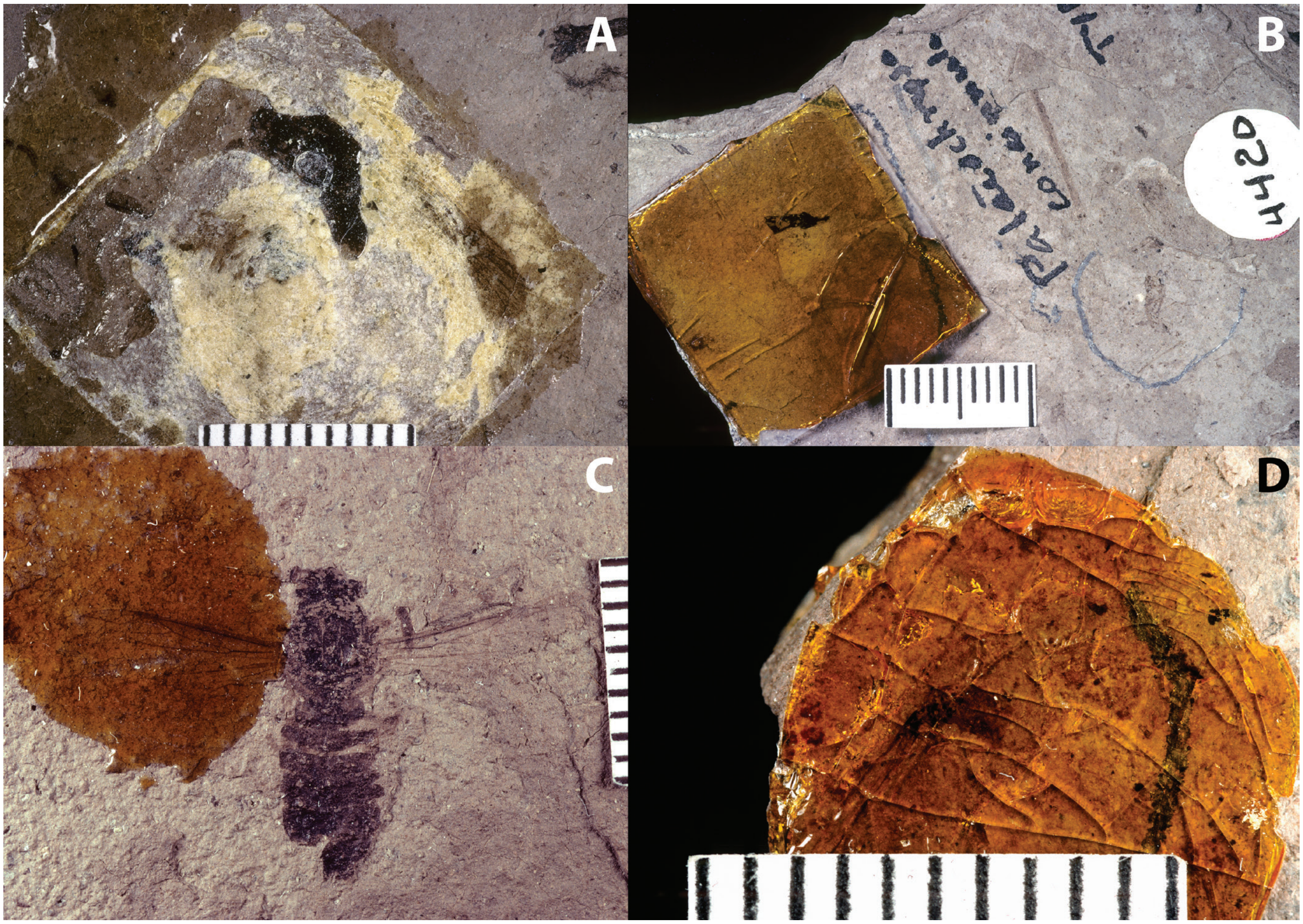
BACKGROUND

Scientific fossil collecting at Florissant began in the 1870s. These collections are housed in major museums around the world. Early efforts at conservation used undocumented substances in efforts to "protect" some of the fossils, although these have often had detrimental effects on the specimens over the course of decades. Some of the fossils had cover slips adhered to them using these substances. Those specimens, including holotypes, are now obscured by the crystallization occurring under the cover slip (Figure 1). However, most historical specimens have retained their inherent integrity and, therefore, were left untreated. Some historical collections may have unmeasured degradation (e.g., fading, accumulation of grime) due to poor storage conditions.

Collections made more recently are stored at Florissant Fossil Beds National Monument (FLFO) and are the primary focus of current research. Some of these FLFO specimens were collected under adverse conditions, including road construction salvage, and therefore have experienced more serious conservation issues due to extreme environmental fluctuations resulting from the need to rapidly collect in undesirable conditions.

The nature of the lithology of the shale units of the Florissant Formation is variable, ranging from laminae as thin as 0.1 mm to thicker interbeds of tuffaceous siltstone or mudstone up to several centimeters thick. Fossils can occur in either shale or siltstone layers in various abundances (Henning et al. 2012).

FIGURE 1. Historical use of unknown consolidants that have degraded over time that obscures the specimen: (a) yellowed and crystallized under cover slip (UCM-15880); (b) yellowed and cracked under coverslip (UCM-18609); (c) yellowing (MCZ-2028); (d) cracked and yellowing (UCM-8556). Scale in mm.

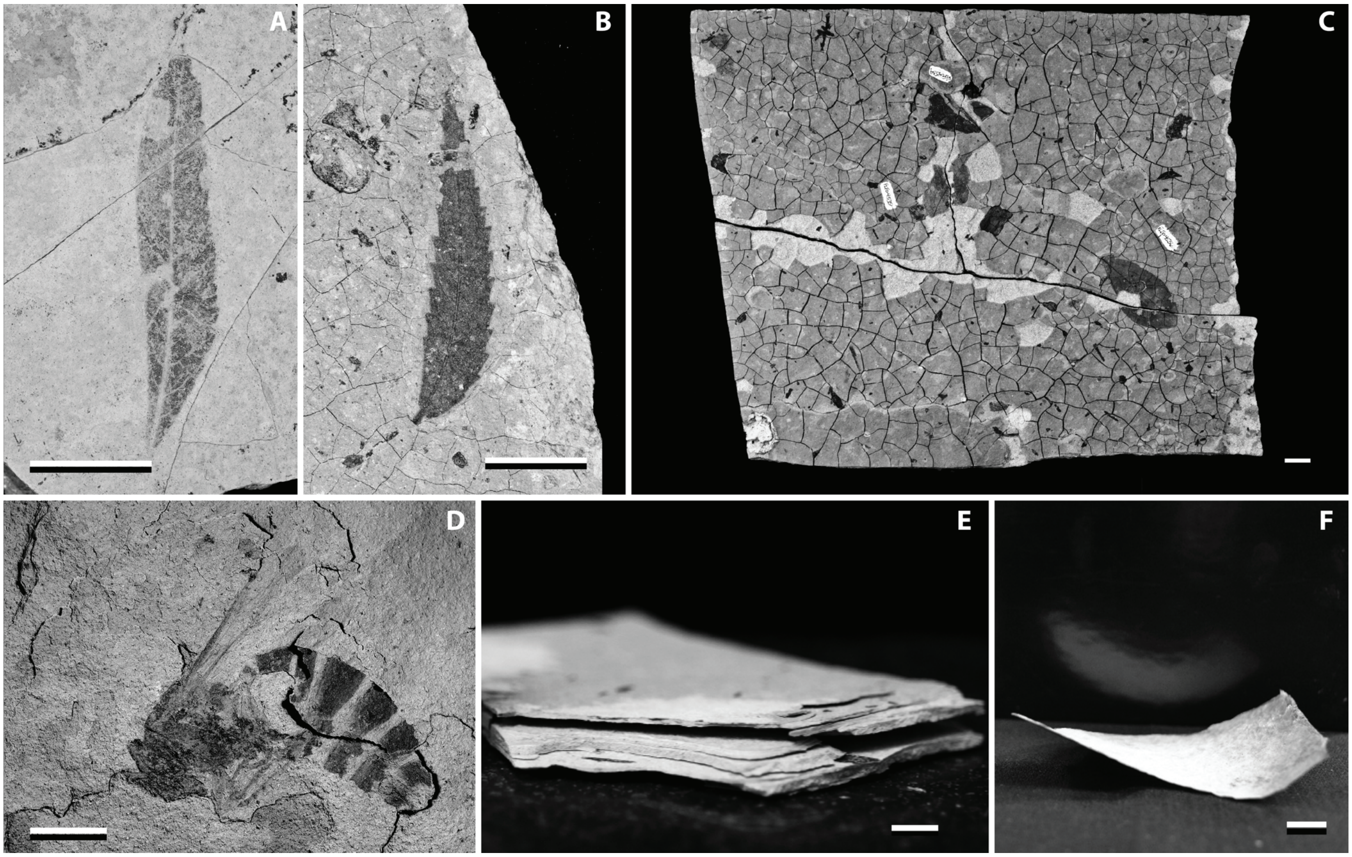


Successive thin laminae are most vulnerable to delamination or deformation, whereas adjacent coarser layers often form a stable base and are more durable. Thin laminae form as couplets of diatoms and smectite that settled in a lacustrine environment (O'Brien et al. 2002, 2008). The fossils preserved in very thinly laminated shale ("paper shales") are most prone to conservation issues. These issues result from the inherent thinness of individual laminae (0.1 to 1.0 mm) and high clay content, and, following collection, sharp fluctuations in external relative humidity (RH) and internal moisture content.

Previous problems with Florissant shale were first reported by O'Connor et al. (2012), and later elaborated by Falkner (2016) and Shelton et al. (2016). These works recognized different types of deformation and the impacts of humidity variation, and proposed preliminary treatments. Types of deformation resulting from the expansion and contraction of the high clay content of the shale during desiccation or hydration include cracking, flaking, delamination, breaking, and warping. There are three subcategories of cracking: linear, crazing, and mudcracking. Linear, or curvilinear, cracks penetrate one or multiple surface layers (Figure 2a). Crazing is evident as a fine network of surficial cracks that are not deep enough to go past the surface layer (Figure 2b). Mudcracks are deep interconnected polygonal cracks that can split through multiple layers (Figure 2c). Flaking is the peeling of layers at the surface often associated with cracking (Figure 2d). Breaks go through the entire specimen and the matrix below it (Figure 2c). Delamination can detach complete layers along bedding planes throughout the specimen (Figure 2e). Warping of horizontal paper laminae is prevalent in very thin layers and cannot be reversed (Figure 2f) (Shelton et al. 2016).

In some instances, deformation issues are exacerbated by field collecting conditions, which are sometimes adverse due to increased ground saturation resulting from frequent summer rainstorms. Collecting techniques for historical collections were typically undocumented, although the majority of collections made in conjunction with planned National Park Service (NPS) projects since 1994 involved quickly wrapping individual specimens in toilet paper within minutes of collecting and allowing several weeks of drying before unwrapping and cataloguing.

FIGURE 2. Types of matrix deformation: (a) linear cracks; (b) crazing; (c) mudcracking and breaking; (d) flaking; (e) delamination; (f) warping. Scale 1 cm.



Current techniques employed by FLFO for stabilization of individual specimens in the collections involves customized nests using Tyvek that cradles the specimen and is constructed within an open-celled polyethylene foam base (Ethafoam) (Figure 3). Previous methods that created nests exclusively in Ethafoam or polyester batting caused damage because the open cells caused rough edges that abraded against the fossils and the batting fibers grabbed onto specimen edges. A method suggested by Falkner (2016) proposed the use of paper backings, such as sekishi kozogami tsuru, sekishu natural, and mulberry, applied with adhesives, but this method was not adopted; although the papers conformed to fragile, paper-thin specimens, they were not as sturdy as archival cardboard, which was also tested as an alternative method.

Storage room conditions can also affect the stability of specimens over time. Extreme fluctuations in environmental conditions, specifically RH, need to be minimized, especially with fragile or already damaged specimens. For example, the fragile delamination of the counterpart of the iconic Florissant fossil wasp (Figure 2d), which was kept on exhibit for many years, presents significantly more issues than the corresponding part, which is on a solid slab of shale that was always kept in collections storage. The FLFO visitor center was completed in 2013 and environmental controls were inadequately considered for the collections area during the design period. In 2017, two air conditioners were installed, one in the collections area and one in the paleontology area outside the collections area. Currently, FLFO can control the temperature and hence passively control humidity, though the collections storage area still does not allow for direct control of humidity. The seasonal fluctuation in climate at FLFO does not produce enough moisture for the air conditioner to control humidity regardless of the decrease in temperature.

FIGURE 3. Current housing standards at FLFO illustrated in a 15.24 x 20.32 cm specimen tray.

The application of particular adhesives to Florissant shale was examined previously by Falkner (2016) and Shelton et al. (2016). Butvars or Paraloid B-72 are the best consolidants previously recommended for use with paper shale specimens. Solvents for these were found to have markedly different reactions when directly applied to the shale, with ethanol being best, whereas acetone caused significant rapid shale deformation and water caused slow warpage of the shale. A 10% weight to volume (w:v) solution in 95% ethanol was recommended as the best for application to the shale. A problem with this is the viscosity of these consolidants, which do not easily penetrate the minute cracks often encountered in Florissant shale. Some applications (e.g., preparation of a Florissant fossil bird by C. Van Beek) have used cyanoacrylates, which rapidly wick into cracks but also permanently set just as rapidly. This can cause problems with reversibility if the matrix has not been joined correctly before the cyanoacrylate sets. The selection of appropriate consolidants must consider their usability in terms of viscosity, but also their effects on color, gloss, and texture following application to the matrix.

The sometimes fragile nature of the shale and/or the specimen requires specialized tools that are used both in removal of matrix and for application of chemical consolidants. The matrix surrounding the fossil sometimes must be removed in extremely small quantities, and tools such as wafer probes with 0.5-micron diameter tips (used in the computer industry), porcupine quills, and specially shaped carbide rods held by pin vises are used to achieve such highly focused preparation. As physical preparation treatments expose the fossil organisms, they can also reveal the need for chemical stabilization of issues such as flaking. Current tools used to apply consolidants are tweezers, oil dippers, syringes, and porcupine quills. Tweezers use the capillary action of the consolidants to draw small amounts into the tweezers, which then can be dispersed onto the appropriate location on the shale matrix. Oil dippers are traditionally used in the watch industry to apply oil to watch gears; they are used in much the same manner for application of consolidant on matrix. Syringes draw up the consolidant which can then be applied in small cracks or between delaminated layers of shale. The distal end of a porcupine quill is textured, enabling it to extract minute quantities of consolidants which can be applied to equally minute places.

One previous investigation on drying shale to mitigate the effects of humidity was completed as an informal project by FLFO (Slovacek 2015). This study collected samples of shale from two outcrops following a rainfall. The shale samples contained varying deformation types, including crazing, mudcracking, and delamination in variable lithologies of the shale. Three drying techniques were tested for a month. The control samples were placed in an archival specimen tray and freely allowed to dry in the room's ambient environment (RH range 7–30%). Test samples were placed in resealable plastic bags that were opened 15 minutes per day, while test samples placed in Tyvek envelopes were not observed until the end of the month. Other test samples were placed in a humidity chamber that was preconditioned to 35% RH and ran continuously for approximately 36 hours before it was turned off to allow the test samples to acclimate to the ambient room RH. Of the methods attempted, this study concluded that the least amount of visible damage occurred to the test samples in the Tyvek envelopes.

Most institutions that house specimens from Florissant are not using current techniques or methods utilized at FLFO. Our hope is that all institutions will apply the updated methods to safely store specimens with respect to stability of nesting in individual boxes, the use of appropriate adhesives, and environmental conditions of their facilities. This paper reports on two current research efforts at separate institutions to mediate these issues. The first, by author EB, investigates drying techniques for shale units to determine the best methods to prevent excessive desiccation due to too-rapid drying following collection under damp field conditions. It is being conducted at the Western Archeological and Conservation Center facility in Tucson, Arizona. The second, by author CC, examines the effects of utilizing cyanoacrylates and polyvinyl butyral solution adhesives as consolidants, with application of accelerated weathering methods to forecast long-range stability and suitability for use on the shale. It is being conducted at the National Center for Preservation Technology and Training in Natchitoches, Louisiana.

DRYING STUDY: METHODS

Sample description and set-up

Varying sample sizes and thicknesses (Figure 4) were excavated in November 2022 for this study. The samples were stored in an environmental chamber at 95% RH. The samples retained moisture, though were not fully saturated. Thirty-six samples were included in the study and categorized into "thin" (0.59–1.31 mm), "medium" (1.44–2.85 mm), and "thick" (3.2–5.93 mm) categories with 12 samples per category. Because of the overall variability and smaller piece sizes available for thin and medium thicknesses, some of the samples are actually composed of more than one piece treated as a single sample though tracked throughout the study as components "a" and "b" of the sample. The samples were separated into six sub-groups (1.1, 1.2, 2.1, 2.2, 3.1 and 3.2) with each sub-group composed of two samples from each thickness category.

The samples were documented with photography using a Nikon D850 SLR camera with a Sigma 24–70 mm lens and a pair of Profoto D1 mono-lights and an X-rite Pantone Classic Nano Color Checker. All photographs were taken in raw format and were color corrected, cropped, and processed into JPG using Adobe Bridge. The thickness of each sample was measured with analog Starrett calipers and the surface area of one side of each piece was measured from top-view photos using the area measurement tool in ImageJ.

Initial sample documentation and treatments were completed while working in a glove-chamber conditioned to between 65% and 75% RH due to the need to control RH prior to intentionally drying the samples. Given the space constraint, a Panasonic Lumix DMC-ZS100 compact camera was used instead of the SLR during the initial documentation phase.

The local environmental conditions of the samples were controlled using a variety of methods, including a Memmert environmental chamber with temperature and RH control, Hefty brand plastic containers with gasketed lids, silica gel, and vapor barrier envelopes constructed from Escal film, which is coated in ceramic polyvinyl alcohol to form a vapor barrier. The local conditions were monitored throughout the study, using Onset HOBOware dataloggers with data uploaded to eClimateNotebook for analysis and comparison.

Half of the samples were treated with tetraethyl orthosilicate (TEOS), also referred to as ethyl silicate and formerly named tetraethoxy silane. TEOS is an alkoxysilane compound that has been used for decades as a conservation treatment to consolidate silicate stone and earthen-based cultural material (Wheeler 2005; Ferron and Matero 2011; Tiennot et al. 2020); it has also been used to a lesser extent to consolidate fossilized bone specimens (Bisulca et al. 2009). TEOS is hydrolyzed by water to produce silanol molecules that condense, forming silicone polymer chains over the course of multiple weeks. The condensed silanol forms a sol-gel network that also bonds to the silica mineral material.

The samples were treated with ≥99.0% TEOS manufactured in Germany by Sigma-Aldrich that was catalyzed prior to use with 1% dibutyltin dilaurate (DBTL), which is a neutral pH organo-metallic catalyst commonly used in

FIGURE 4. Thick (left), medium thickness (middle), and thin (right) samples. Scale 1 cm.

commercial formulations for stone consolidation. The TEOS was catalyzed immediately prior to use. The samples were placed individually in a dish of TEOS, allowing it to be absorbed through capillary action. The samples were determined to be fully treated when the top surfaces were visibly saturated. TEOS was also applied by brush to the top surfaces of the thick category samples to ensure sufficient application to both top and bottom surfaces. No observable change occurred during treatment other than temporary darkening due to saturation. The TEOS was applied while working in a glove chamber for the first round of application. Samples exposed to uncontrolled environments were treated while working in a fume hood wearing nitrile gloves.

Experimental condition methodology

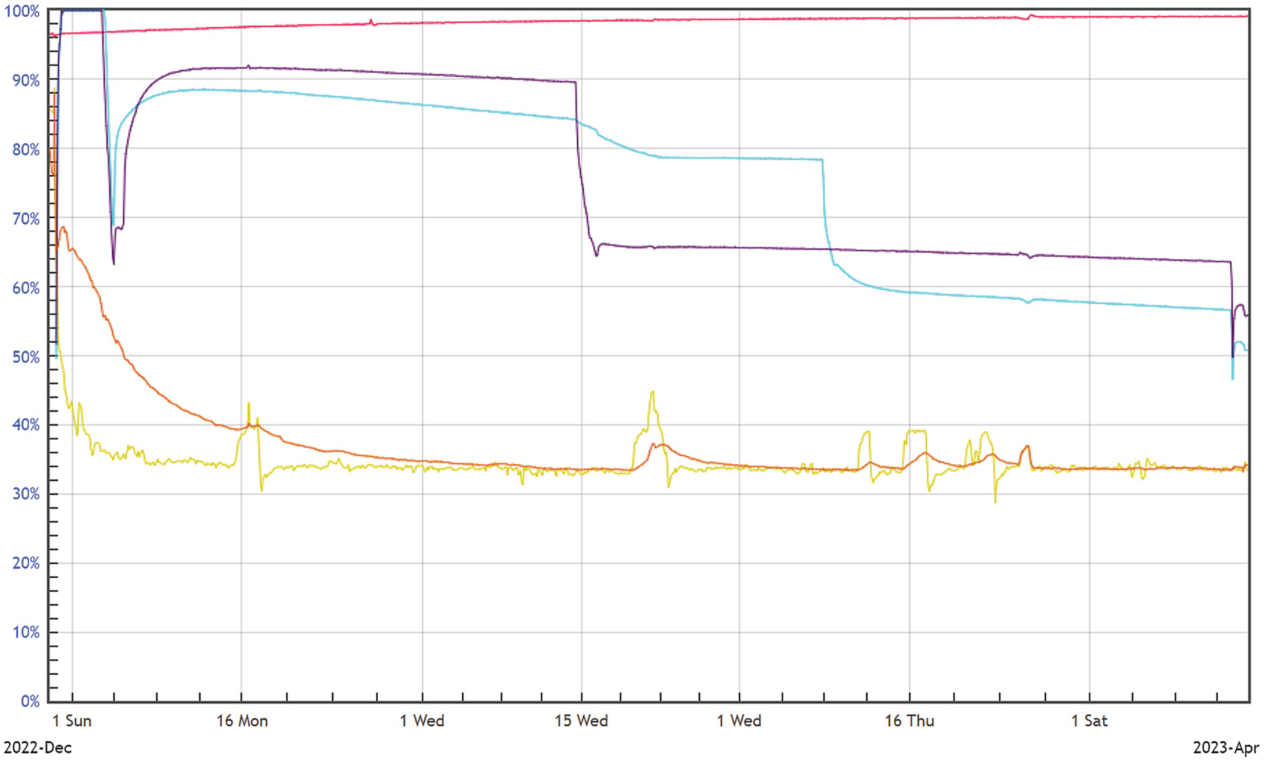
This study included different drying phase procedures followed by four variable-RH tests named A-1, B-1, B-2, and B-3.

The first 24 samples were documented and treated with TEOS while working in a glove chamber conditioned between 65% and 75% RH to minimize the uncontrolled effects of samples exposed to environmental change. Treated and untreated samples assigned to each sub-group were kept together so that they were exposed to the same environment throughout the study. Samples were transported from the environmental chamber to the conditioned glove box using gasketed boxes containing silica gel conditioned to about 70% RH.

After initial documentation and treatment with TEOS, the 24 treated and untreated samples in sub-groups 1.1, 1.2, 2.1, and 2.2 sat for 15 weeks to allow time for the TEOS to hydrolyze and condense. During this time the sample sub-groups already acclimated to a high-RH environment were exposed to different conditions while bringing their environment to ambient conditions to test the effects on both the paper shale and sol-gel formation (Figure 5; Table 1).

Twelve of the samples (sub-groups 1.1 and 2.1) were stored in two vapor barrier film envelopes and their internal environments modified using silica gel and air exchange. These environments began at 100% RH and were reduced to between 55% and 65% RH during the 15 weeks. The data show a steep drop in RH that soon recovered to about

FIGURE 5. Environmental RH during 15-week drying period for samples 1.1 (teal), 1.2 (yellow), 2.1 (purple), and 2.2 (orange).



Sample Description			Drying Experiments				
Count Treated with TEOS	Count Untreated	Sub-group Name	Initial 15-Week Drying Phase	A-1	B-1	B-2	B-3
3 total: 1 thin 1 medium 1 thick	3 total: 1 thin 1 medium 1 thick	**1.1**	High RH with intermittent drops	Reduced RH 2 percentage points every 2 hours to 19% RH	Reduced RH 2 percentage points every hour to 19% RH	Reduced RH 4 percentage points every hour to 19% RH	Reduced RH 6 percentage points every hour to 11% RH
3 total: 1 thin 1 medium 1 thick	3 total: 1 thin 1 medium 1 thick	**1.2**	35% RH and variable	NA			
3 total: 1 thin 1 medium 1 thick	3 total: 1 thin 1 medium 1 thick	**2.1**	High RH with intermittent drops	Reduced RH 2 percentage points every 2 hours to 19% RH			
3 total: 1 thin 1 medium 1 thick	3 total: 1 thin 1 medium 1 thick	**2.2**	35% RH and stable	NA			
3 total: 1 thin 1 medium 1 thick	3 total: 1 thin 1 medium 1 thick	**3.1**	High RH with quick drop during last 4 days	Removed from A-1			
3 total: 1 thin 1 medium 1 thick	3 total: 1 thin 1 medium 1 thick	**3.2**	High RH with quick drop during last 4 days	NA			
36	**36**	**72**					

TABLE 1. Sample descriptions and experimental methods by sub-group.

90% RH in both envelopes at the start of the study. Both chambers experienced two steep drops in RH of up to 30 percentage points about 6 weeks into the drying process after the sol-gel had already formed, and near the end of the 15-week period (teal and purple plots in Figure 5). These drops were caused by opening the envelopes and allowing air exchange to encourage drying.

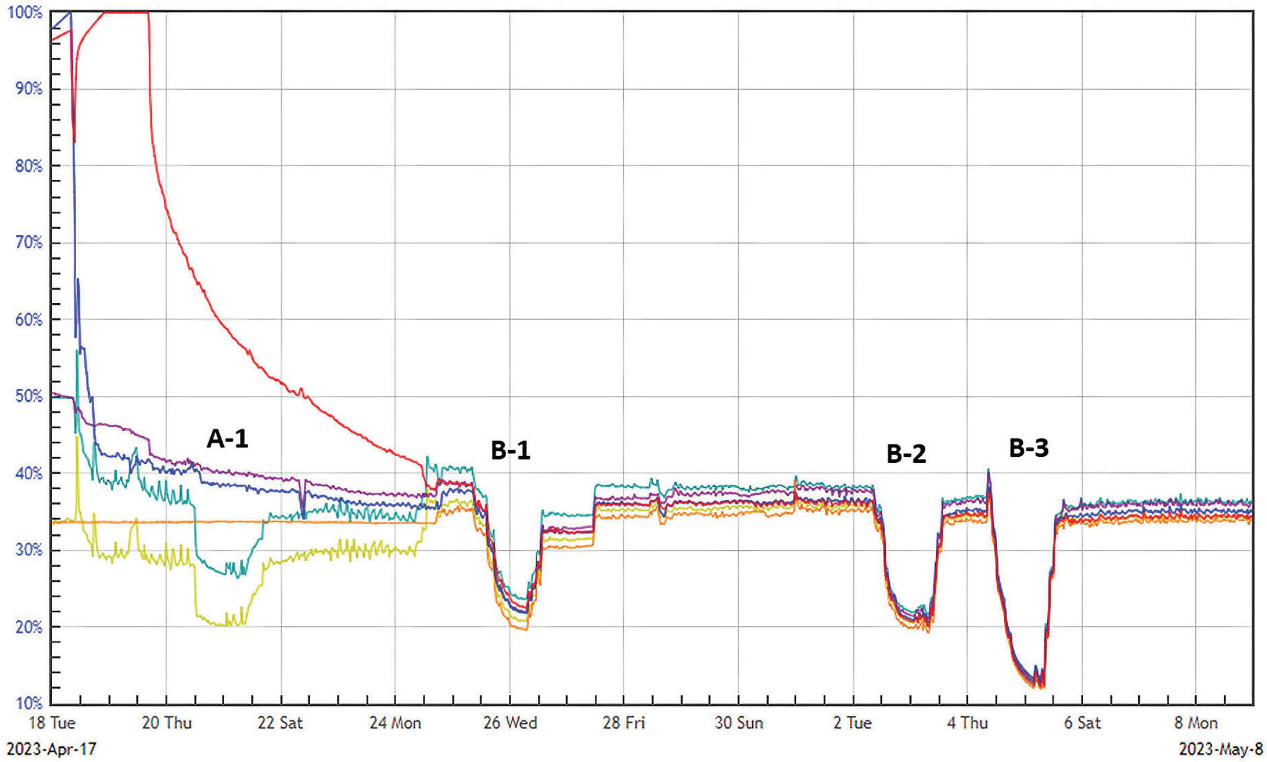
The other 12 samples were contained in Hefty brand plastic containers and exposed to the moderate environment of the lab beginning at 35% RH, six of which were left on the counter exposed to fluctuation of +/-5 percentage points (sub-group 1.2), while the other six were kept in a gasketed museum cabinet with the same average RH and minimal fluctuation (sub-group 2.2). For the first few days, lids were placed unlatched over the containers to allow for some level of drying control. According to the dataloggers near the samples, the local environments began at 70% RH and leveled off within days to 35%. The initial drying periods were more gradual over 21 days for samples in the cabinet (orange plot in Figure 5) compared to those left out in the open on the counter (yellow plot in Figure 5) that dried over 7 days with greater fluctuations during drying. After 15 weeks, the samples were documented in ambient conditions.

After the initial 15 weeks of preparation, 12 additional samples that had been stored above 95% RH (red plot in Figure 5) were assigned to sub-groups 3.1 and 3.2, documented, and half were treated with TEOS. The goal was to introduce these samples to the following stress tests for comparison to the samples allowed to reach 35%, though their introduction to the testing chambers made it difficult to incrementally reduce RH during the tests due to the high moisture content they released into the chamber as they dried. These groups were instead allowed to dry quickly to 35% in an uncontrolled environment prior to further testing. They were photographed with the standard photo set-up in an environment at 35% RH at the beginning of their drying phase.

Slow drying test A-1

The treated and untreated samples from Group 1 (1.1 and 1.2) were added to the humidity chamber for test A-1 (Figure 6). Samples from sub-group 3.1 that were anticipated to be in this test added too much moisture to the chamber due to their hydrated state and were removed from this test group. Sub-group 1.1 was conditioned to about 50% RH at the beginning of the study and sub-group 1.2 was conditioned to 35%. The chamber containing 1.1 and 1.2 was conditioned to 35% RH at the beginning of the test and incrementally decreased to 19% RH over the course of two days. This was achieved by reducing RH by two percentage points every two hours over the course of 16 hours, plateauing at 27% RH for 16 hours between days. The RH was held at 19% for 16 hours before increasing RH to 35% over the course of two days using the same method. Packets of silica gel desiccant had to be introduced to the chamber to reach RH below 27%, likely due to the buffering effects of the shale (moisture absorption of the clay content). The desiccated silica gel absorbed the excess moisture to create a lower RH environment. There was a discrepancy between the settings applied to the Memmert environmental chamber and what was achieved (Figure 6) throughout the chamber. At the end of A-1, the samples were removed from the

FIGURE 6. Stress tests A-1 and B-1–3 for samples 1.1 (teal), 1.2 (yellow), 2.1 (purple), 2.2 (orange), 3.1 (blue) and 3.2 (red). Samples 2.1, 3.1, and 3.2 were left in ambient room conditions held near 35% RH while running the A-1 experiment.



chamber and photographed on both sides and thin samples also in profile. Sub-group 3.1 was also photographed.

Slow drying tests B-1, B-2, and B-3

All 36 samples from Group 1 (1.1, 1.2, and 1.3), Group 2 (2.1 and 2.2), and Group 3 (3.1 and 3.2) were introduced to a humidity chamber for successive tests B-1, B-2, and B-3 (Figure 6). Each test was initiated and completed at 35% RH with one day of decreased RH followed by one day of increase at the same rate the following day with a hold overnight at the minimum target RH. Test B-1 altered RH an average of two percentage points every hour, with a target RH of 19%; test B-2 altered RH by an average of four percentage points every hour, also with a 19% target RH; and B-3 altered RH by an average of six percentage points every hour down to 11% RH. Packets of silica gel desiccant were introduced for the chamber to reach below 27% RH likely due to the buffering effects of the paper shale samples. There was a discrepancy between the settings applied to the environmental chamber and what was achieved throughout the chamber because the chamber and added desiccant could not achieve the target RH quickly enough to compensate for the amount of water vapor released into the environment by the samples. All study samples were returned to ambient conditions using the same protocols and photographed on both sides; in addition, thin samples were photographed in profile. Photographs were not taken for sub-group 3.2 between initial drying and the start of B-1 as the original intention was to start B-1 with hydrated samples. The methodology was not updated alongside this adaptation in the protocol.

Evaluation methodology

Between each phase and test, the samples were photographed, except for photographs not taken between initial drying and the start of B-1 for sub-group 3.2. After all four rounds of stress testing were complete, the images were compared to identify at which phase different signs of damage developed with the following damage categories: linear cracking, crazing, surface flaking, delamination, and warping. Each progression or development of damage was considered an individual instance. Damage incurred for sub-group 3.2 during the drying phase and B-1 test were counted together as a single instance.

CONSOLIDANT STUDY: METHODS

Thirty samples of paper shale matrix were cut into 4.45x4.45-cm squares using a Hillquist saw with a diamond blade. These samples were split into groups of five for treatment with different consolidants, including two ethyl cyanoacrylates (Aron Alpha 241F and PaleoBond PB40); one clear, undyed, medical-grade butyl-octyl cyanoacrylate blend (90% butyl, 10% octyl) (GluStitch GluSeal) (composition confirmed by GluStitch, personal communication, January 2020); and two polyvinyl butyral solution adhesives (5% w:v Butvar B-76 in 95% ethanol and 5% w:v Butvar B-98 in ethanol). The three cyanoacrylates are proprietary blends made by their respective manufacturers and may contain other ingredients for maintaining their viscosity. For example, PaleoBond PB 40 contains ethyl-2-cyanoacrylate, 2-ethoxyethyl 2-cyanoacrylate, and 1,4-dihydroxybenzene (composition confirmed by PaleoBond, personal communication, January 2020); these other compounds may also impact samples. Treatment included flooding the surface of each sample with the respective consolidant and adhering a chip of paper shale to the upper left corner of the sample. When treating samples with ethyl cyanoacrylate, goggles, nitrile gloves, and a half-mask respirator with organic vapor filters were worn. Five samples of paper shale were left untreated as controls.

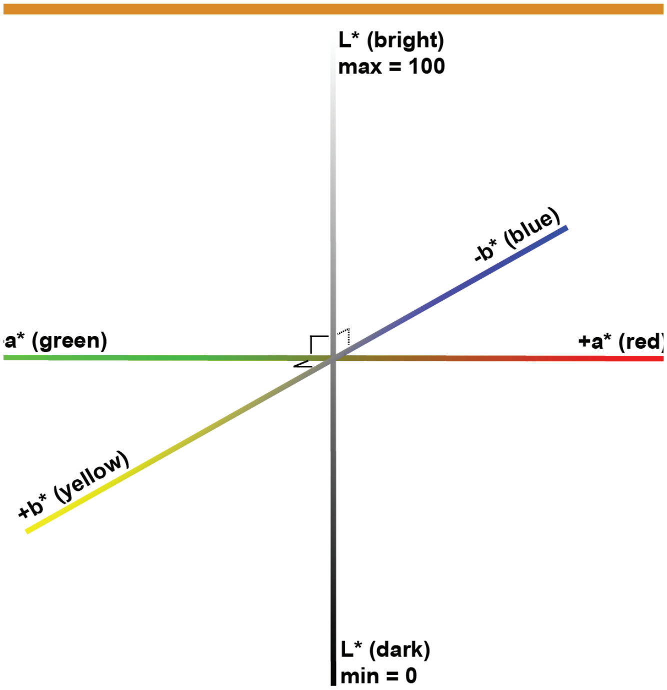
Samples were weathered following the American Society for Testing and Materials (ASTM) standard D904-99 (2013) in a QUV weatherometer; this procedure necessitated exposing the samples to constant direct ultraviolet A (UVA) light at 60°C. The samples were weathered in 200-hour increments for a total of 800 hours. Data were collected at each stage: before treatment with consolidants, after treatment with consolidants, and at each 200-hour weathering increment to document change in the consolidants across weathering.

Color measurements were taken with a Konica Minolta Chromometer 400; the instrument was programmed to take five measurements across the sample surface for an average value reported as three numbers along a three-dimensional coordinate plane: L—light to dark, a—green to red, and b—yellow to blue. The absolute change in color of a sample (ΔE*) was calculated using the coordinate values of the before-treatment color measurement (pT), and the after-treatment (T) or after-weathering measurements using the formula

$$\Delta E^* = \sqrt{(L_2 - L_1)^2 + (a_2 - a_1)^2 + (b_2 - b_1)^2}.$$

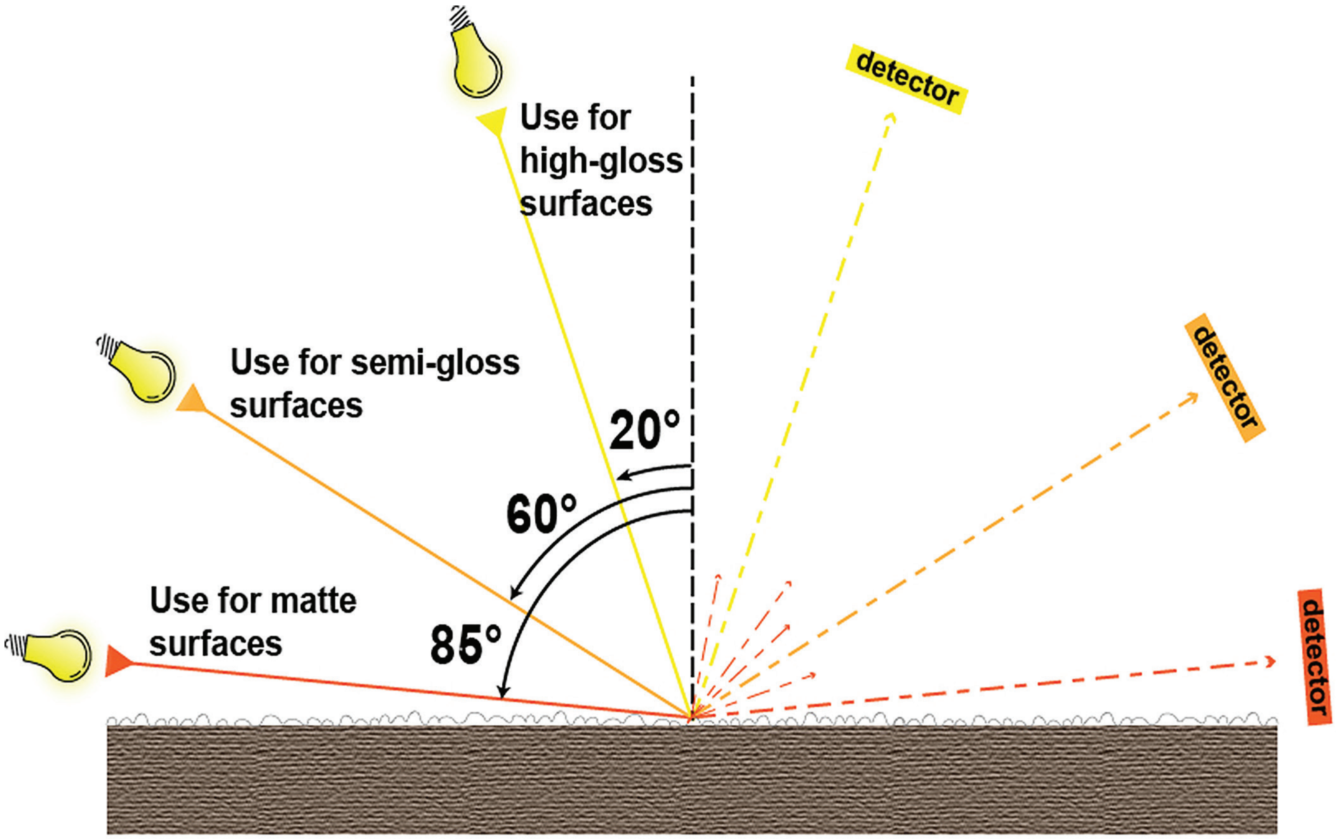
A color change of ΔE* ≥ 2 is considered significant and perceptible to the human eye (Pretzel 2008). ΔE* was calculated between the baseline color measurement and each subsequent treatment and weathering increment (Figure 7).

Gloss measurements were taken with a BYK Gardner micro tri-gloss glossmeter, which shines a light on the surface of a sample at specified angles and collects light that bounces off the surface at the instrument's paired detector (Figure 8). Paper shale fossils are relatively matte, so the 85º angle for matte surfaces



▶ **FIGURE 7.** Axes of three-dimensional space in which colorimeter measurements were made, with x, y, and z coordinates. Changes in these coordinates over time can be used to calculate absolute color change.

▼ **FIGURE 8.** Diagram illustrating how light in the glossmeter is bounced off the surface of the sample and measured at the paired detector. Surface roughness can cause the light to bounce and scatter away from the detector so that it is not measured, and the surface is measured as less glossy.



was used. Five measurements were taken and used to calculate an average for each sample at each measuring timepoint.

A Keyance VR-3200 optical light profilometer was used to collect both photographs and surface texture scans of the sample surfaces. The instrument reports many measurements around surface topography; the SPC (Arithmetic Mean Peak Curvature) measurement indicates the average peak curvature across the sample, where the higher the SPC value, the sharper the peaks on the surface (Figure 9).

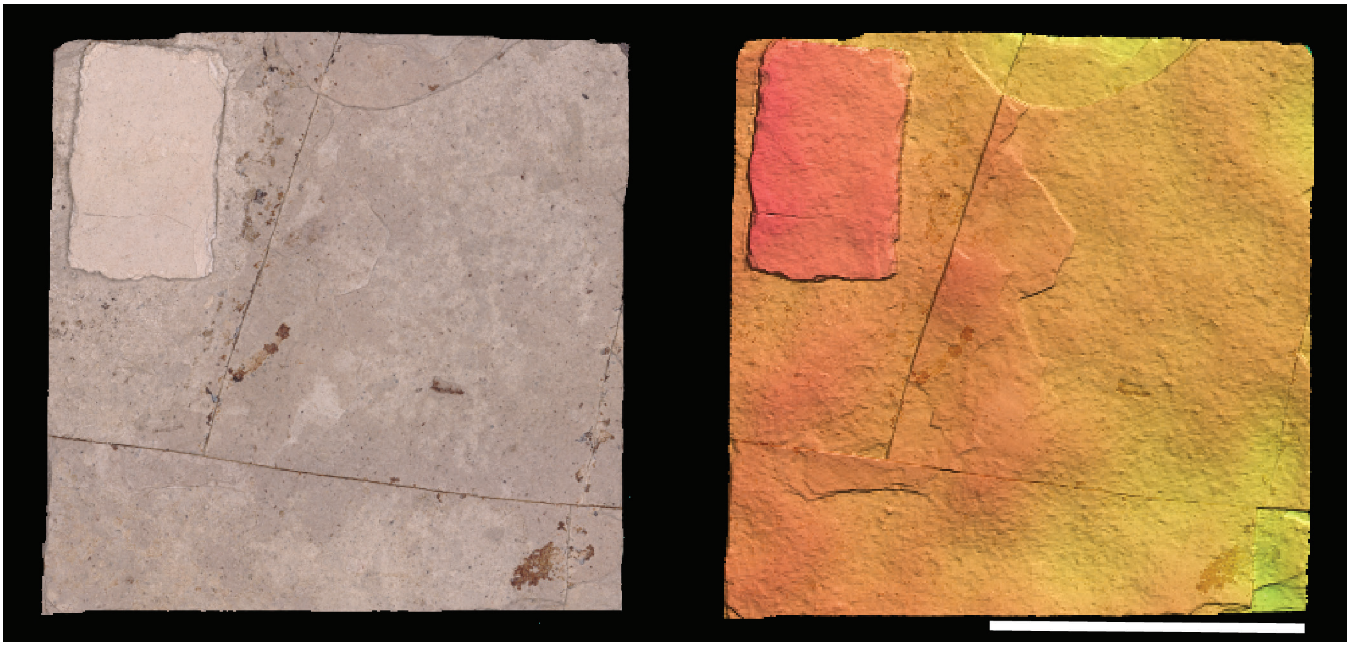
During analysis of the data, "failure" was defined as (1) loss of material either through the detachment of the paper shale chip or through delamination and flaking of the main sample block, either of which might cause loss of fossil information, and/or (2) change in appearance as measured by color, gloss, and surface roughness, each of which might impede interpretation or study of the fossil.

Fourier-transform infrared (FTIR) spectroscopy was used to identify the chemical stability of the consolidants. FTIR spectroscopy uses infrared radiation to indicate what functional groups are present in the material; it also identifies potentially unique chemical patterns that serve as "fingerprints" in each sample. This study was mainly interested in spectral consistency rather than functional groups or fingerprints. Pure samples of consolidants were dried and analyzed using a Thermo Fisher Nicolet iS50 ATR (attenuated total reflection) spectrometer. A Thermo Fisher Nicolet iN5 FTIR microscope in reflection mode was used to collect qualitative data from samples without removing material and to compare how the spectra changed over the course of 800 hours of weathering.

DRYING STUDY: RESULTS

Overall, 66.7% of the samples became visibly deteriorated over the course of the study due to mechanical response to environmental fluctuation. Visible evidence of damage was induced during both the initial drying phase and during variable RH stress testing, which indicates that more gradual drying periods result in fewer instances of damage. Samples in Group 3, which had the most severe drying cycles over less than four days, developed some form of damage on 11 of the 12 samples (91.7%). Samples with more controlled drying in Groups 1 and 2 developed visible damage on 13 of the 24 samples (54.2%). The additional round of stress testing completed for Group 1 compared to Group 2 did not have an apparent impact. Samples in sub-groups 1.1 and 2.1 that were dried to 35% RH over 15 weeks developed damage on five of 12 samples (41.7%). Sub-groups 1.2 and 2.2 that reached ambient conditions of

FIGURE 9. Two types of images are captured by the Keyance optical light profilometer. The left image is a high-resolution white-light photograph of the surface; the right image is a 3D-rendered image showing the surface texture with the colors used to indicate surface height from the viewing stage and topography. Scale is 2 cm.



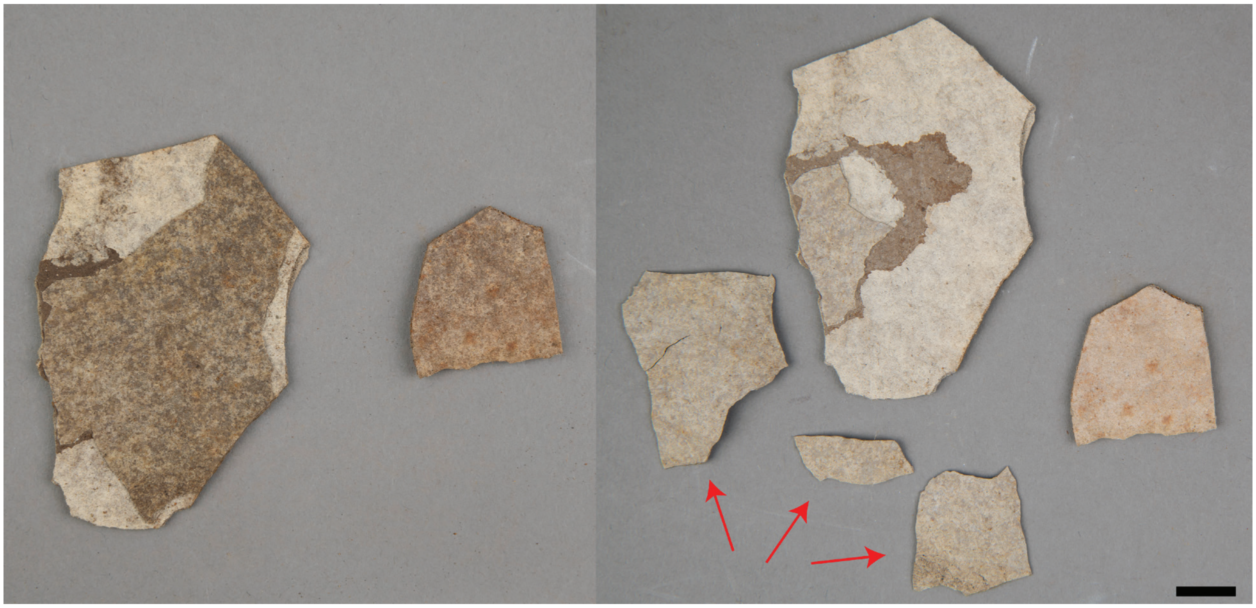
35% within 7 and 21 days, respectively, developed damage during the study on four samples within each subgroup, totaling eight of the 12 samples (66.7%).

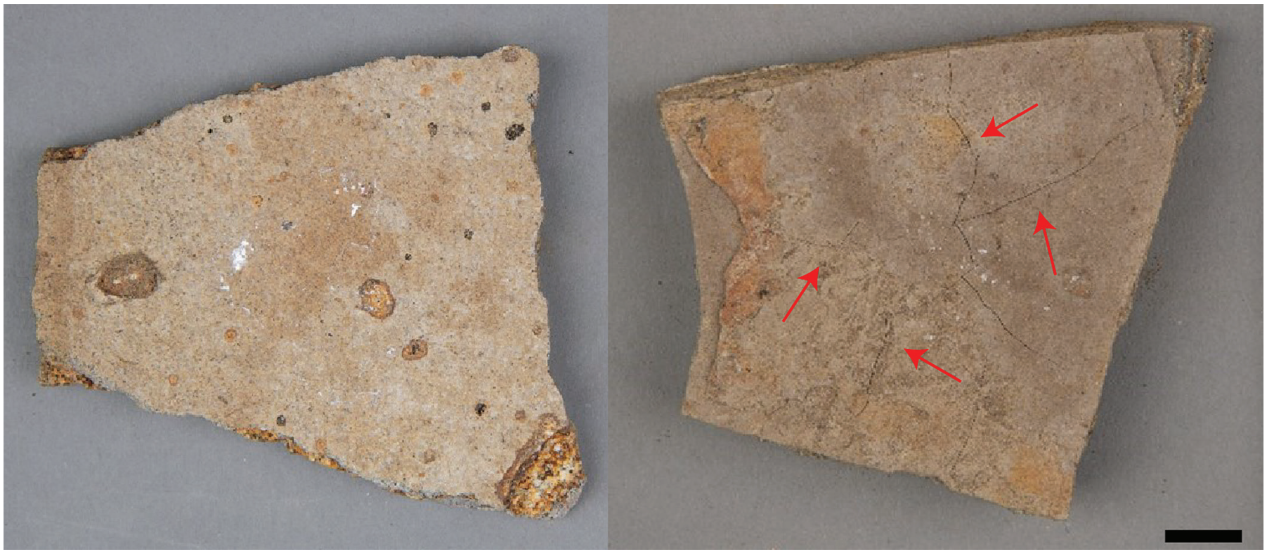
Eleven of the 24 samples (45.8%) that dried in controlled conditions (Groups 1 and 2) developed visible evidence of damage after the initial drying phase and two additional samples developed damage during the following stress tests. Six of the 12 TEOS-treated samples in Groups 1 and 2 were undamaged by the end of the study and the other half developed only one instance of damage per sample. Five of 12 untreated samples in the same groups were undamaged and of the seven that were damaged, four developed multiple instances (57.1% of damaged samples). While all TEOS-treated samples in Group 3 with uncontrolled drying were damaged, only one sample developed multiple instances, compared to the untreated samples in Group 3, where all samples that developed damage incurred multiple instances (Figure 10).

Samples that were treated with TEOS and dried to ambient conditions over 15 weeks (sub-groups 1.1 and 2.1) performed better than all other samples (Figure 11). None of these samples developed damage during the initial

▼ **FIGURE 10.** Thin samples in sub-group 3.2 without TEOS treatment, before drying (left) and at the conclusion of the study (right). Three sections from the largest sample detached and warped (arrows). Scale is 1 cm.

▼▼ **FIGURE 11.** Sample treated with TEOS in sub-group 1.1 (left) compared to sub-group 2.1 (right) at the conclusion of the study. Damage indicated by arrows. Scale is 1 cm.





drying process and only one warped during the fourth round of stress testing. These two sub-groups are the only two where TEOS was allowed to form a gel prior to undergoing the mechanical stress of initial drying and additional fluctuation in subsequent stress tests. The untreated samples in the same sub-groups incurred damage likely caused by the decreases in RH about six weeks into the drying period and near the end of the drying period. The treated samples were not impacted by the decrease, potentially because the TEOS had formed a sol-gel by this point in the drying process.

In contrast, samples treated with TEOS then immediately dried to ambient conditions over 7 and 21 days (sub-groups 1.2 and 2.2, respectively) during the 15-week curing time developed damage during the initial drying phase on five of the six samples (83.5%) and performed worse than untreated samples in the same environments, where only half of the untreated samples were damaged during initial drying (Table 2). Though quick drying of TEOS-treated samples developed cracks, there was only one sample with more than one instance of damage.

TABLE 2. Instances of damage for each sample in Groups 1, 2, and 3. The experiment cycle during which the observed damage occurred is listed for each sample, with “drying” referring to the conclusion of the initial 15-week drying phase. Instances of damage (indicated by arrows in Figures 10 and 11) were only photographed before drying and after B-1; damage incurred may have taken place in either or both phases.

Sample #	Treatment	Thickness Category	Total Instances of Damage	Linear Cracks	Crazing	Flaking	Delamination	Warping
1.1	TEOS	1	0					
		2	0					
		3	0					
	Untreated	1	1			A-1		
		2	2			Drying		
		3	0					
2.1	TEOS	1	1					B-3
		2	0					
		3	0					
	Untreated	3	0					
		2	1	Drying				
		1	3	Drying		B-2	Drying	
1.2	TEOS	1	1		Drying			
		2	1		Drying			
		3	1		Drying			
	Untreated	1	3			A-1	Drying	Drying
		2	0					
		3	0					
2.2	TEOS	1	1					Drying
		2	0					
		3	1		Drying			
	Untreated	1	1					Drying
		2	0					
		3	2		Drying	Drying		
3.1	TEOS	1	1	Drying				
		2	1	Drying				
		3	1		Drying			
	Untreated	1	2		Drying		Drying/A-1	
		2	2		Drying		B-1	
		3	2		Drying	Drying/ B-3		

Sample #	Treatment	Thickness Category	Total Instances of Damage	Linear Cracks	Crazing	Flaking	Delamination	Warping
3.2	TEOS	1	4	B-3		B-3	Drying-B-1*	Drying-B-1*/ B-3
		2	1	Drying B-1*				
		3	1		Drying-B-1*			
	Untreated	1	3		Drying-B-1*	Drying-B-1*		Drying-B-1*
		2	0					
		3	3	Drying-B-1*	Drying-B-1*	Drying-B-1*		
1.1 Total			3	1		2		
2.1 Total			5	2		1	1	1
1.2 Total			6		3	1	1	1
2.2 Total			5		2	1		2
3.1 Total			9	2	4	1	2	
3.2 Total			12	3	3	3	1	2
Number of instances of damage			40	8	12	9	5	6

Total Count of Samples Damaged	
1.1	2
2.1	3
1.2	4
2.2	4
3.1	6
3.2	5
All Samples	24
Percent of 36 Samples	66.7%

TABLE 2 (cont'd). Instances of damage for each sample in Groups 1, 2, and 3. The experiment cycle during which the observed damage occurred is listed for each sample, with "drying" referring to the conclusion of the initial 15-week drying phase. Instances of damage (indicated by arrows in Figures 10 and 11) were only photographed before drying and after B-1; damage incurred may have taken place in either or both phases.

During the study, visible damage developed on 30 of the 56 individual pieces that comprise the 36 samples (53.6%) (Figure 12). Of these, 60% in the thin category and 64% in the thick category developed visible damage, compared to only 35% in the medium category. This is likely because medium-thickness pieces have already had fragile layers detach during excavation or transport, making them thinner than the thickest samples and potentially more stable than the thicker pieces that retain fragile outer layers. Thin samples were more prone to warping and delamination than medium and thick samples. Samples in all thickness categories had evidence of surface cracks and surface flaking.

CONSOLIDANT STUDY: RESULTS

Over the course of treating and weathering the samples, all retained their adhered paper shale chips, and none lost other surface material. There was some delamination beginning to occur to lower layers in the shale, but no loss. Thus, all the consolidants passed the first measurement and did not fail.

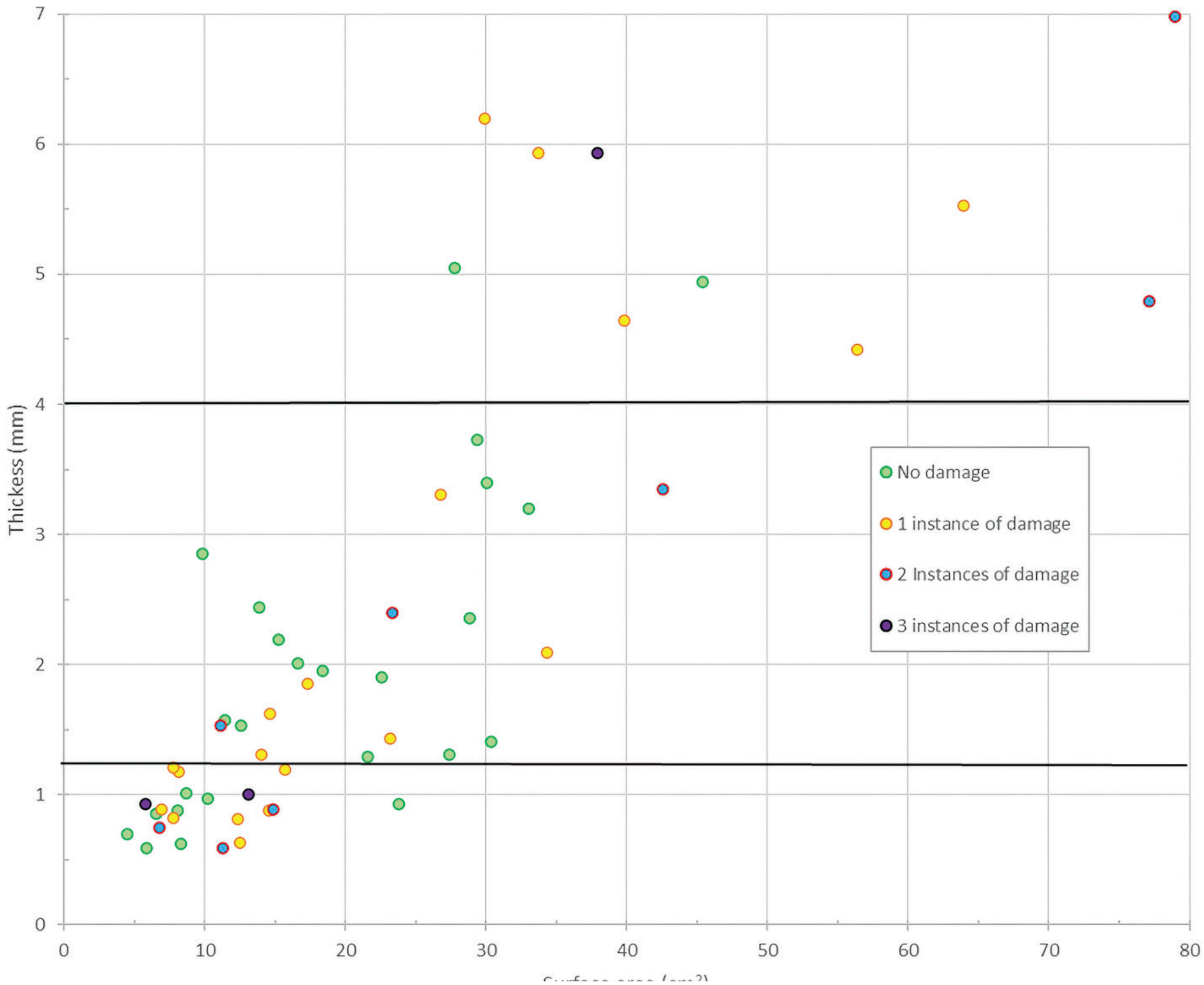


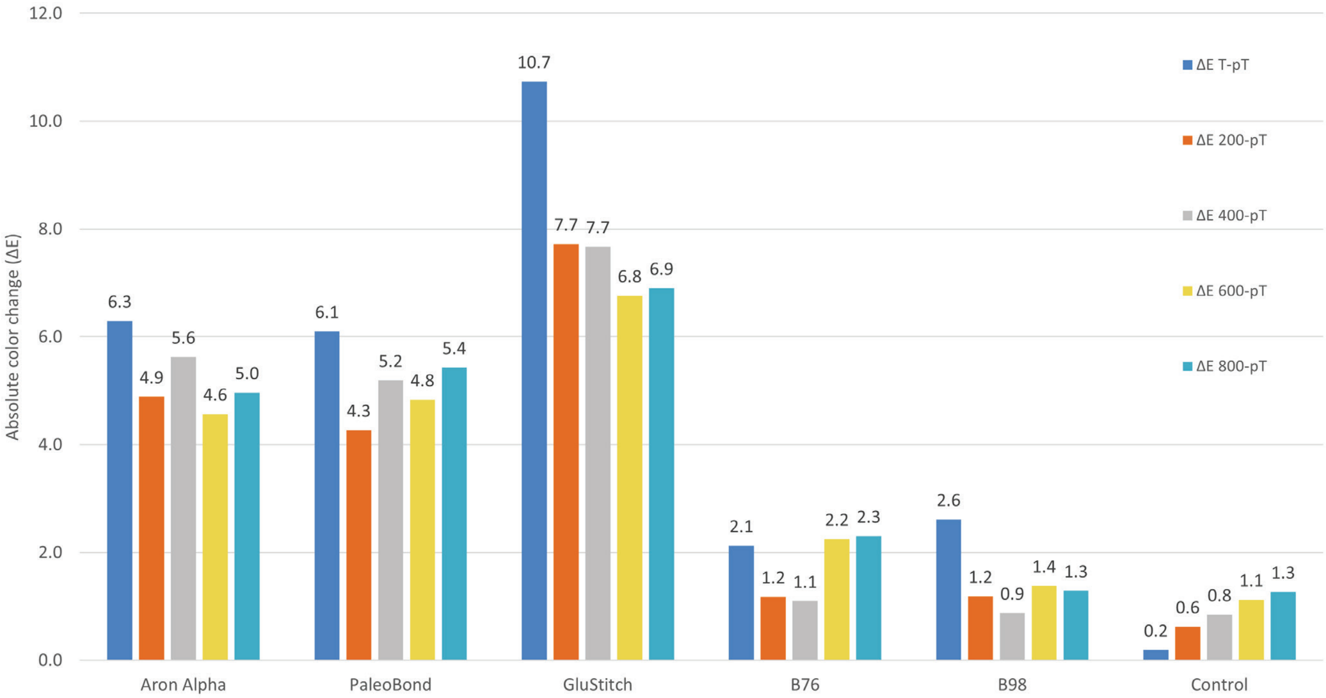
FIGURE 12. Sample pieces plotted by thickness against surface area categorized by instances of damage incurred during the course of the study.

The greatest color change from the baseline untreated samples (pre-treatment, or pT) occurred just after treatment (treated, or T) with the respective consolidants and before weathering; this is seen most distinctly in the samples treated with GluStitch, which immediately yellowed after application (Figure 13).

The exposure to UVA light did cause some bleaching of the consolidant-treated surfaces over the course of the 800 hours of weathering. Overall, the greatest trend was the change in color, with the largest color change observed in samples treated with the butyl-octyl cyanoacrylate blend, and the least color change in the Butvar resins. Butvar B-98 bleached to within the detectable ΔE* ≥ 2 range during UVA exposure, resulting in values closest to the control (Figure 14). There is also a trend of the greatest color change from baseline occurring immediately after treatment and before weathering.

Gloss and surface roughness measurements were interpreted together because the surface roughness can impact gloss readings. Increased surface texture can scatter the light away from the paired detector, resulting in a lower gloss measurement (Figure 15). The various consolidants had different trends: the Butvars behaved roughly the same, increasing in gloss after treatment and then decreasing again with weathering, PaleoBond and GluStitch both decreased the glossiness of the surface, and Aron Alpha appeared slightly glossier.

The gloss readings indicated that the ethyl cyanoacrylates caused the least absolute change in gloss across the experiment. This differed from qualitative observation of the surfaces, which indicated a beaded glossy surface (Figure 16).

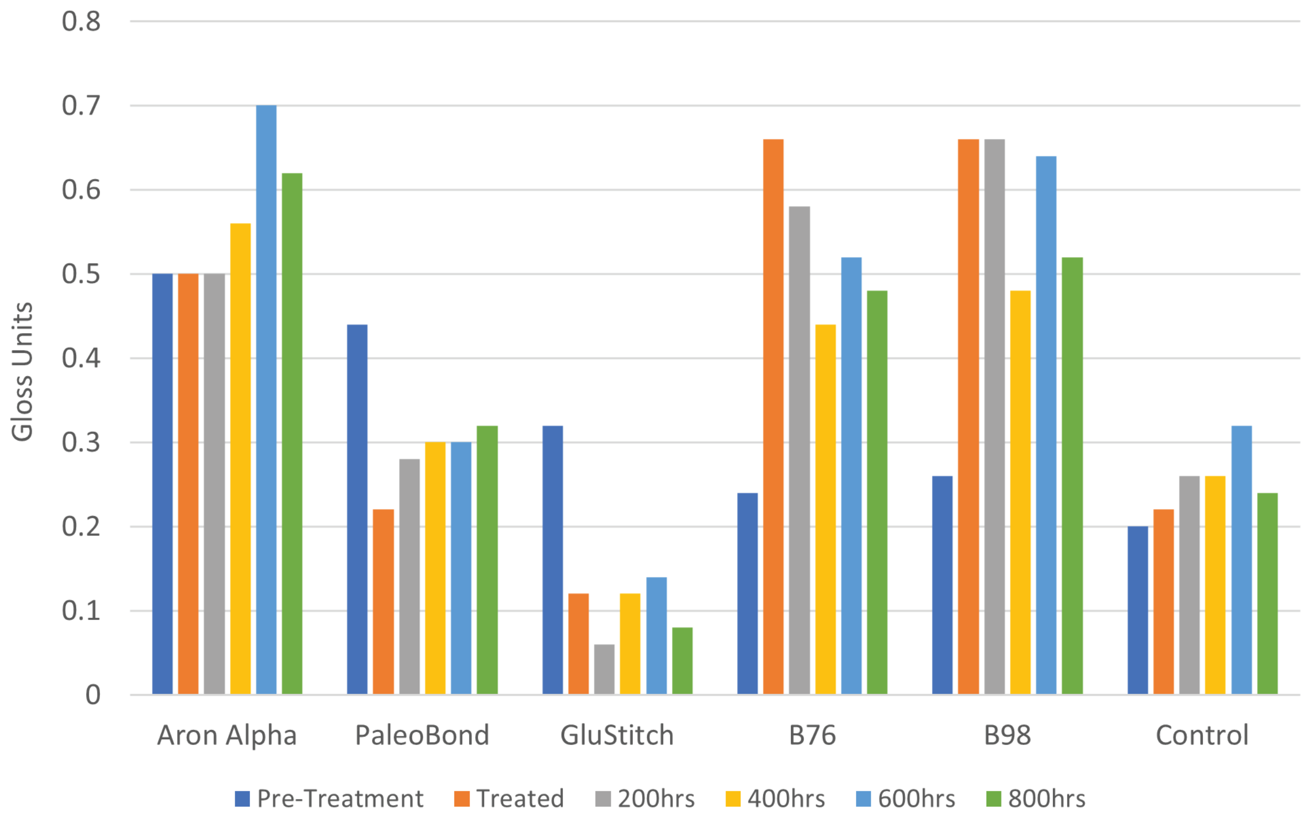


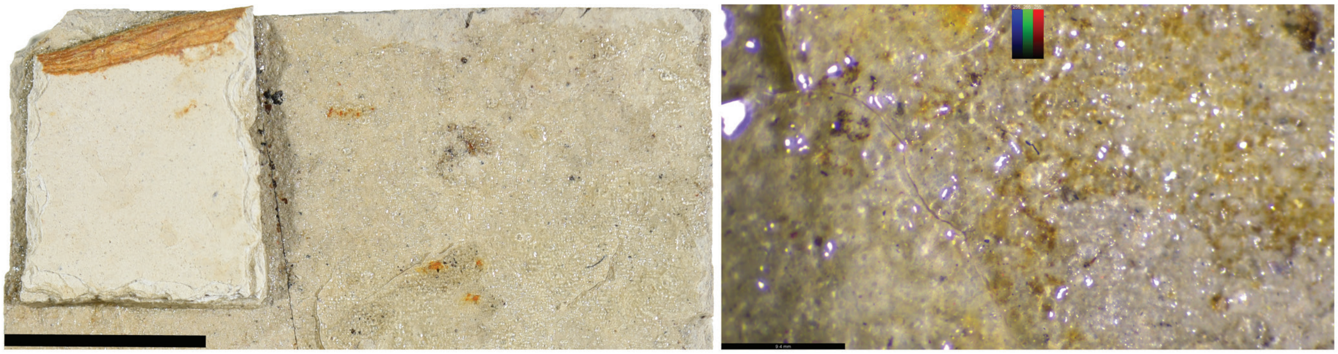
▲▲ **FIGURE 13.** 1GS1 sample pre-treatment and just after treatment with GluStitch and paper shale chip. The butyl-octyl cyanoacrylate not only changes the color but also increases the contrast between the matrix and the fossil inclusions. Scale is 1 cm.

▲ **FIGURE 14.** Bar graph showing the average of the absolute color change from baseline at each of the 200-hour weathering increments for each of the different consolidants.

The surface roughness measurements support the qualitative observations: SPC values increased for samples treated with the cyanoacrylates, and the surface texture remained relatively constant for both types of Butvar and the control across the experiment (Figure 17). The increased texture on the cyanoacrylate treated samples was scattering the light, causing a lower gloss measurement.

Results from this experiment indicate that the two Butvars cause the least overall change in appearance when compared to the control samples. Butvar B-98 evidenced the least overall color change over the course of the experiment, closely followed by Butvar B-76 (Figure 14). The Butvars did not alter the surface roughness of the samples appreciably.





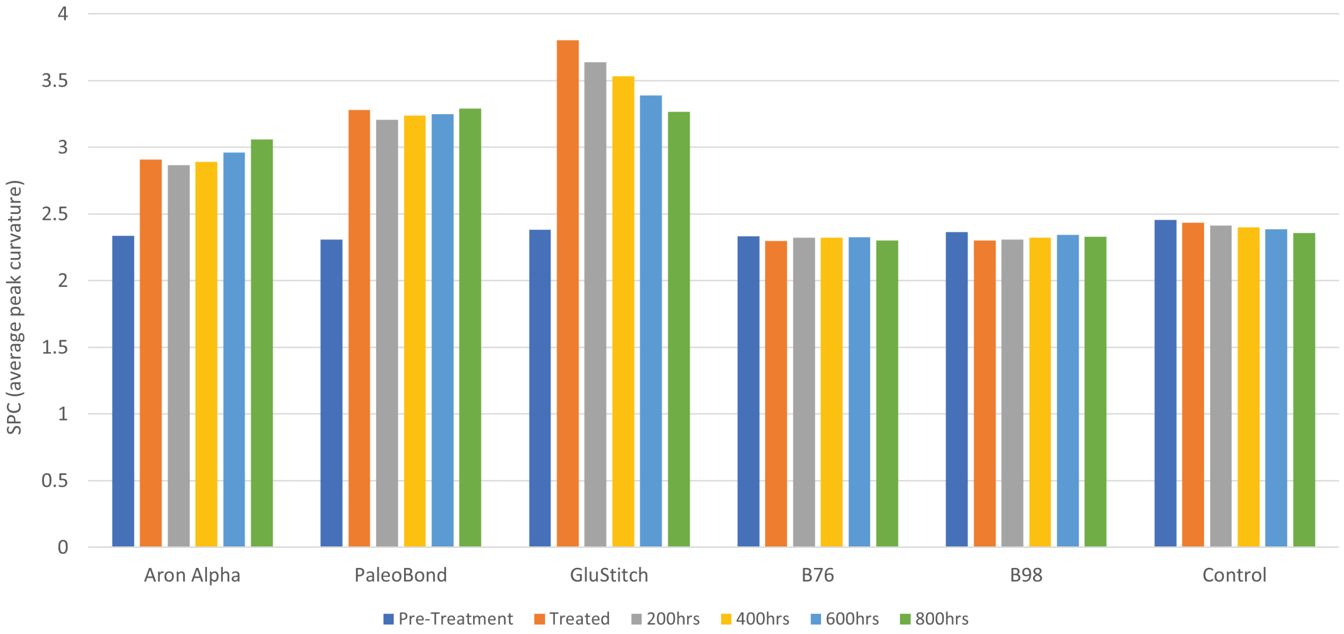
▲▲ **FIGURE 15.** Average gloss values for each of the different sampling periods—pre-treatment through 800 hours.

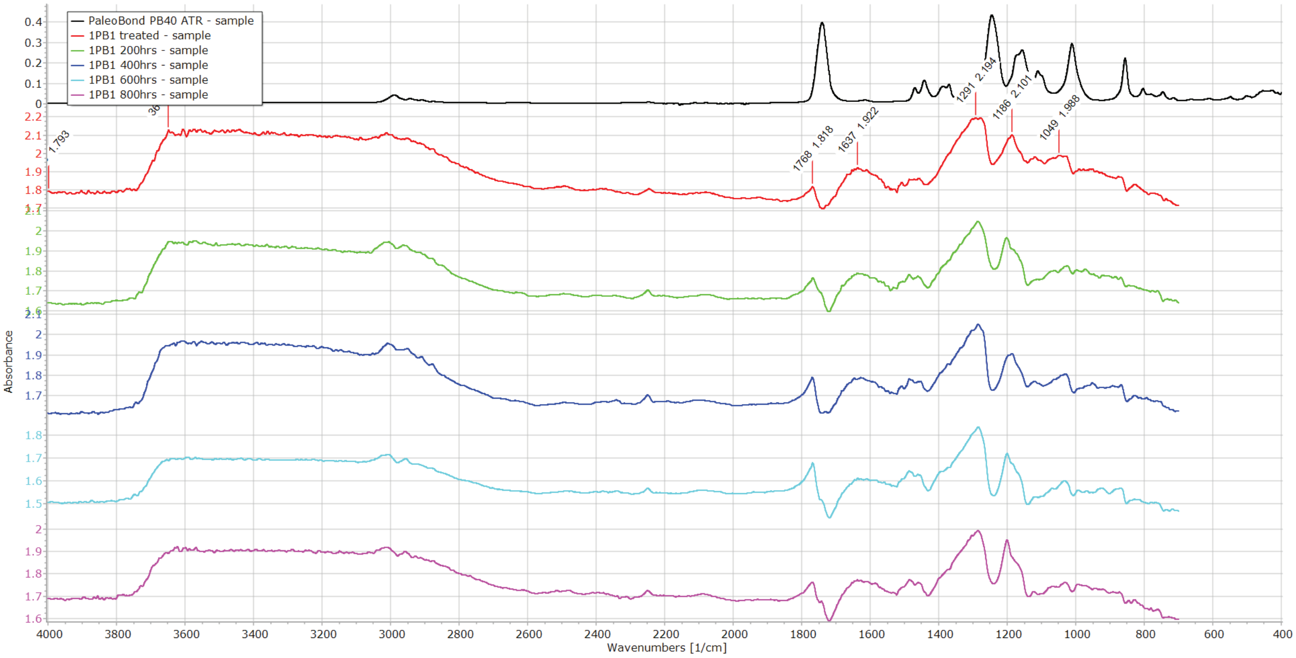
▲ **FIGURE 16.** Sample 1AA4 after treatment with Aron Alpha, showing the beaded, sparkly surface created by the consolidant. Left scale is 1 cm. Right scale is 9.4 mm.

Consolidants were also assessed for chemical stability over the course of weathering using a FTIR spectrometer. Changes in consolidant spectra over the course of weathering were tracked by taking FTIR spectra at each experimental stage. In the following graphs (Figures 18–20), the first spectrum (black) is the adhesive examined as a cured thin film using the ATR attachment on the Thermo Fisher Nicolet iS50 FTIR. The second through sixth spectra (red, green, blue, turquoise, purple) represent the treated sample after increasing hours of exposure to UVA collected using the Nicolet iN5 microscope attachment in reflectance mode. Slight shifts in the wavenumber of the bands is expected due to the difference in the analytical methods.

The band shapes remain relatively consistent across all five scans of the fossils treated with PaleoBond, with some slight variation in the resolution of peaks around 2900 wave numbers and in the fingerprint region (Figure 18). This stability of the spectral pattern across weathering is seen in samples treated with either ethyl cyanoacrylate, PaleoBond or Aron Alpha.

The spectra of the samples treated with the butyl-octyl cyanoacrylate blend were similarly consistent. The peaks and region around 2900 wave numbers of sample 1GS3 are more defined than in the ethyl cyanoacrylate samples, but again only show slight variation across the experiment (Figure 19).



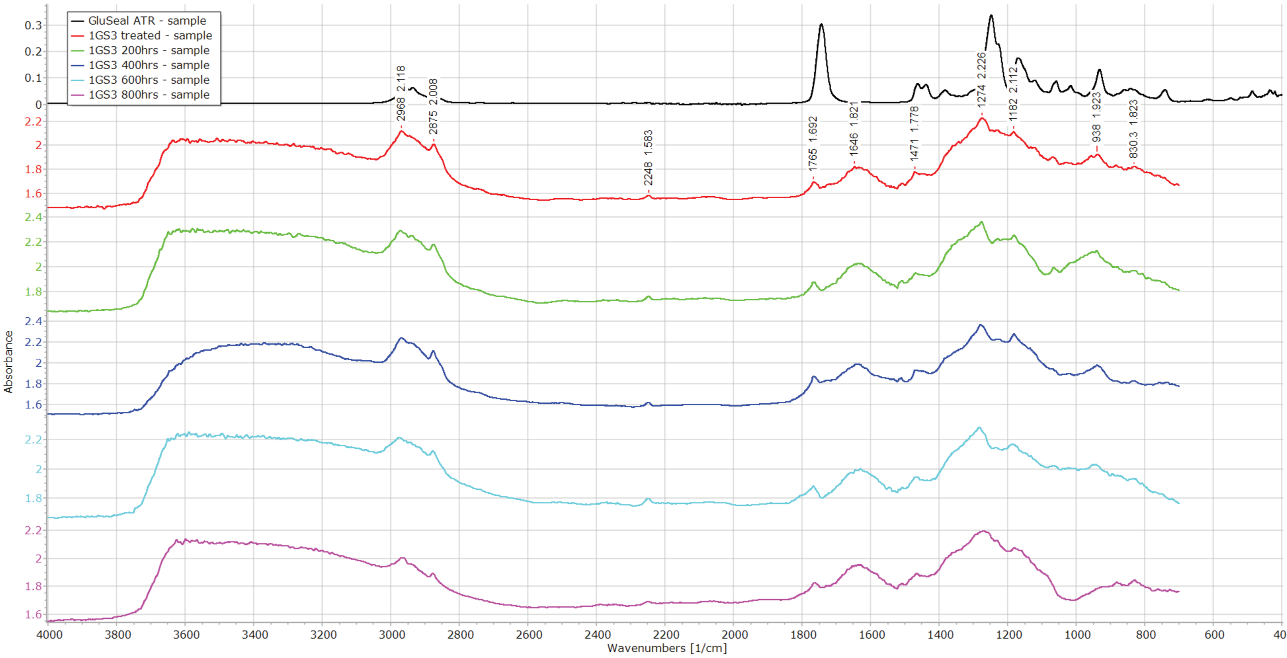


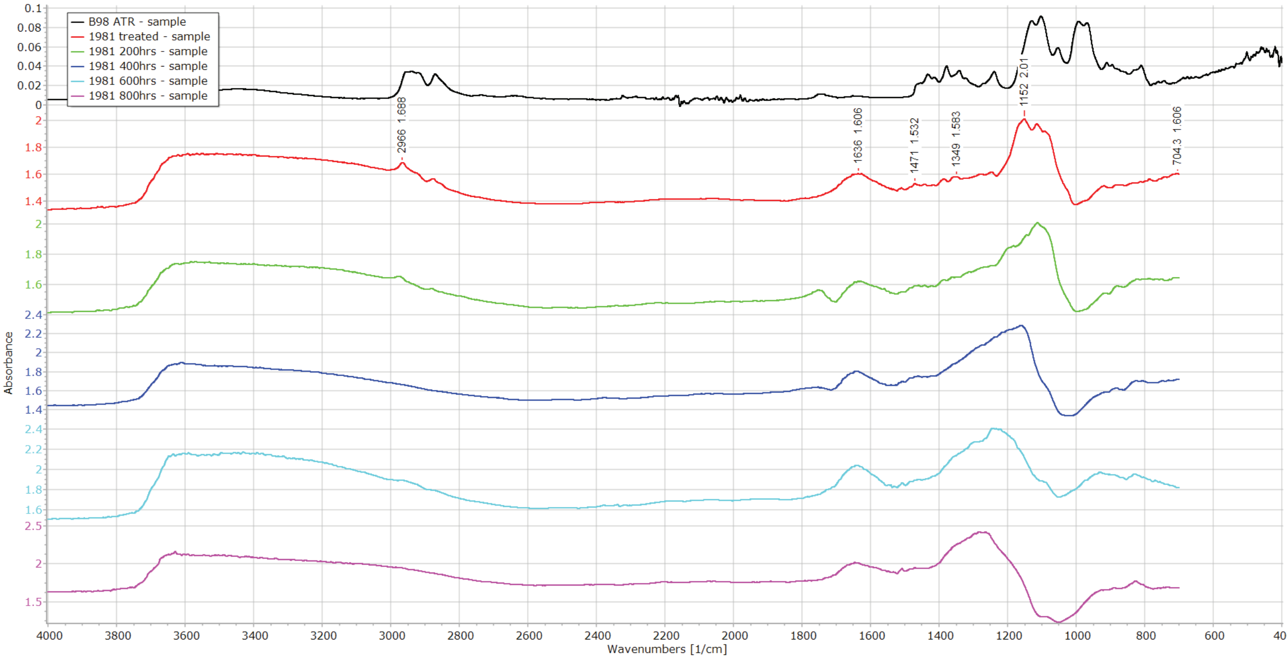
▲▲ **FIGURE 17.** Average SPC (peak curvature value) for each of the different sampling periods—before treatment through 800 hours.
▲ **FIGURE 18.** Stacked FTIR spectra taken from sample 1PB1 showing peak consistency across weathering from directly after treatment (red) through 800 hours of UVA exposure (purple).

This spectral consistency across weathering is not seen in either of the Butvar resins. Sample 1981 treated with Butvar B-98 shows the greatest peak definition in the first spectrum taken just after treatment, with the peaks become decreasingly less defined across the 800 hours of weathering (Figure 20). This could be a result of the qualitative nature of the experiment combined with not sampling the exact same location each time. However, this pattern exists across the samples treated with Butvar B-98 and Butvar B-76, which indicates that this is likely more than an artifact of sampling error.

DISCUSSION

Current research on samples from the shale units of the Florissant Formation are focused on how to best preserve the fossils for future examination. The studies can be applied to the care of previously collected specimens, which





▲▲ **FIGURE 19.** Stacked FTIR spectra taken from sample 1GS3 showing peak consistency across weathering from directly after treatment (red) through 800 hours of UVA exposure (purple).
▲ **FIGURE 20.** Stacked FTIR spectra taken from sample 1981 showing peak smoothing (degradation) across weathering from directly after treatment (red) through 800 hours of UVA exposure (purple).

have been in various collections since excavations began in the late 1870s, as well as best practices for preparing specimens immediately post-excavation. In both cases, the critical application of the experiments reported here is to ensure that the distinguishing features of the fossils are preserved for future study.

Those working on previous fossil excavations at FLFO have observed that a change in the color of the matrix can be beneficial to discovering fossils. As the matrix gradually dries naturally or fades in sunlight, the contrast between the matrix and the fossil organism increases, especially in cases where the fossil is a faint impression. Similarly, the change in color observed in this study following application of cyanoacrylates increases the contrast between the fossils and the matrix. While the contrast is beneficial, the butyl-octyl cyanoacrylate blend changed the color of the sample (Figure 13), and the ethyl cyanoacrylates increased the gloss and surface texture of the samples (Figures 16 and 21).

FIGURE 21. 1PB1 sample pre-treatment and just after treatment with PaleoBond PB40. The ethyl cyanoacrylate increases the contrast between the fossil inclusions and the paper shale matrix while also creating surface sparkles. Scale is 1 cm.

The reflectivity of light resulting from gloss on the surface of a specimen can negatively impact the quality of photography and microscopic examination and create challenges in achieving optimal lighting angles. An example of this can be seen in Figure 16, where the fossil's original surface is obscured by the adhesive layer and bouncing light. Surface texture is not always apparent to the naked eye; in the case of the ethyl cyanoacrylates, the texturizing is noticeable. Texturizing of the surface can decrease the measurement of gloss or the apparent gloss of the sample, as found in the samples treated with GluStitch. Although some treatments may improve the appearance of the fossils, leaving them untreated is always optimal.

Another potential drawback of chemical treatments is that a gradual change in the appearance of a consolidant can indicate it is failing mechanically or chemically. Historically, treatments covering a fossil may have been chosen for their transparent characteristics, but over time these treatments have degraded and created problems such as darkening, crystallization, and opaqueness, which can cause specimens to become completely obscured (Figure 1). Most of the earliest authorities on fossil preparation made conservation a priority (Brown 2013) and considered long-term effects of the methods and materials used. However they did not have the capacity to study the effects of weathering and aging over time, nor how chemical treatments being used would limit or impact future analysis. Thorough and accurate documentation of all preparation in collection records is critical to enable future evaluation of the techniques used.

The selection of adhesives and consolidants for use on Florissant shale is influenced largely by practicality. The minute scale involved often demands the use of very low-viscosity fluids, a condition that cyanoacrylates satisfy. Although the low viscosity enables penetration for use as a consolidant, brittleness due to poor aging is a disadvantage for use of cyanoacrylates as an adhesive. Another limitation is their ineffectiveness when in the presence of moisture (Elder et al. 1998). Cyanoacrylates are generally disfavored among conservators because they are not completely reversable and some formulations require personal protective equipment for handling and application. It must be considered that application of *any* chemical is irreversible regardless of what is used, and will cause molecular changes. Although we consider that the question of reversibility is secondary to the stabilization of the fossil, further research is needed to investigate the possibility of reversal techniques for historical treatments where consolidants have degraded to the point of obscuring type specimens.

The results of the consolidant study thus far indicate that the Butvar resins alter the appearance of the fossil samples the least of the five compounds tested. These Butvar resins did exhibit some non-visible chemical changes over the course of accelerated weathering and UVA exposure. Though not a metric that determines success or failure, consideration of the future chemical stability of the consolidants is also important. Previous experimentation with butyl cyanoacrylates has shown that they are more stable than ethyl cyanoacrylates (Down and Kaminska 2006), but the alteration of sample color and relative expense of the chemical make it a less ideal choice. Butvar resins are considered to be less chemically volatile than cyanoacrylates, with any degradation products less likely to cause damage or toxicity (Davidson and Alderson 2009). Even though this study shows potential degradation of the Butvar resins over time, the chemical changes did not cause detrimental visual alterations during the course of the experiment. The impacts of these chemical changes could be explored more fully in future research. Other avenues for research could include exposure to cyclical indoor environmental conditions, penetration capabilities of consolidants, and methods for applying adhesives and consolidants.

Chemical alteration and degradation of a consolidant are important long-term conservation considerations. As with preparation of any fossil, it is always better to do nothing if the specimen is stable. The tradeoffs between conservation and scientific study must always be considered since chemical treatment can impact or limit future analytical techniques.

Different types of lithologies can have varying responses to treatments with the solvents used in adhesives or consolidants. For example, in laminated shale, water will react with the clay content to cause swelling, whereas acetone causes instant desiccation and severe deformation when applied directly. Toluene and 190-proof (95%) ethanol have proven to be the best solvents in terms of reactivity with the shale, though toluene is a carcinogen that requires more personal protection (Falkner 2016).

The results of the drying study confirm that a slower, more gradual drying period reduces the occurrence of damage compared to a faster, more uncontrolled drying cycle. All drying methods used in this study caused damage to some of the untreated samples. Untreated samples require more controlled and gradual drying cycles to prevent damage from occurring. Further testing to identify appropriate lab and field protocols, such as introducing desiccated silica gel over a controlled period into a sealed environment with hydrated samples, is an area for future research.

Samples treated with TEOS, where the TEOS was given time to condense under humid and stable conditions before RH was decreased, were protected from damage during initial drying. The treatment also appears to have stabilized samples during subsequent fluctuations after initial drying, which is based on the fewer instances of damage of all TEOS-treated samples compared to the untreated counterparts. The results indicate that once TEOS gel is formed, the paper shale is more resistant to mechanical damage caused from environmental fluctuations; however, treating samples without providing time for TEOS to fully react is detrimental, causing surficial cracking, delamination, and warping. This may be caused by the shrinkage of TEOS as it condenses during drying, though additional analyses are required to determine the cause. Mitigating these effects by applying different concentrations of TEOS in silicon oil and covering the samples to control evaporation after TEOS application is an area for future research. Similarly, testing with different dilutions and protocols may help identify a treatment route with less risk during the TEOS drying process. Similar to consolidants, there is also a need to test how TEOS ages.

Although fossils can occur within all lithologic layers, the more delicate specimens are typically found in the thinner laminae. Thin samples (defined in this study as less than 1.31 mm thickness) are prone to warping, particularly if they are composed of a single lamina or few laminae. However, when thinner laminae are still attached to thicker beds, they are prone to damage in the form of cracking and delamination. If more delicate layers are exposed on the exterior of a slab, they may be more prone to cracking, delamination, and detachment compared to slabs that have more robust layers exposed on their outer surfaces. Some samples may not consist of thin laminae, which could make them less prone to damage. Variation of interbedded laminated shales and siltstones across the samples was an uncontrolled variable that may have impacted study results. Future testing of individual lithologies will provide a better understanding of impacts by the drying process. Additional work is still needed to determine the feasibility of freeze-drying techniques, optimal conditions for environmental control for long-term storage of collections, and compatibility of TEOS with consolidants, specifically Butvars and Nanolime.

CONCLUSIONS

Specimens from the Florissant Formation are unique as a fossil record of the late Eocene ecosystem and face conservation challenges due to their composition and laminar structure. This research has initiated new developments of best practices for two aspects of conservation: drying shale following field collection, and the application of consolidants. Both of these aspects are critical for ensuring the stability of specimens.

Prevention of deformation is a major concern with Florissant shale, beginning at the moment of collection but also continuing throughout the duration of long-term storage. This study indicates that the best procedure for initial drying of the shale is accomplished by slow and stable drying protocols exceeding 21 days. Treatment with TEOS a few weeks prior to drying stabilized the samples during environmental fluctuations and prevented damage from occurring compared to untreated samples. This study only examined the thickness of a sample, whereas future research is still needed to examine drying effects on lithologic variation within the shale units.

The choice of consolidants is influenced by the effect of these substances on the color, gloss, and durability of the fossil. The Butvar resins performed the best in regard to overall appearance and did not result in any loss of material, although they did exhibit the most consistent chemical change over the course of weathering.

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