BUILDING A BETTER CLAMSHELL BOX: AN EVALUATION AND STUDY OF ADHESIVES FOR TRADITIONAL RARE BOOK BOXES

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I) Background

The cloth covered box for rare books, commonly referred to as a clamshell or drop spine box, is often considered the gold standard for housing library materials. Since the 1970s, when the Conservation Division at the Library of Congress (LC) was established, clamshell boxes have been constructed onsite and until recently were routinely made for each rare book that received conservation treatment. Each box is constructed by hand to the exact dimensions of the book. It's a labor-intensive process that requires skill and significant amounts of book board, cloth, and adhesive.

The adhesive most used for clamshell box making at LC, as well as many other institutions in the United States, has been PVAc (polyvinyl acetate). Its popularity is due in large part due to excellent working properties. It has very good flow and spreads easily on cloth, paper, or board, has ample "open time," i.e. the amount of time to work with the adhesive after it has been applied but before it sets, and creates a strong bond that is also flexible in the box joints where the lid opens and closes. While the working properties of PVAc have made various PVAc formulations an obvious choice for box making, there have also been concerns about its stability. For many decades the conventional wisdom that was passed from conservator to conservator was to "let your box air for two weeks" before putting the book inside.

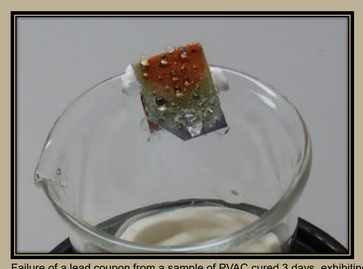
II) Previous evaluation of adhesives by the Library of Congress

The Library began testing adhesives for constructing housings as early as 2004. The results of 2004 Oddy testing of a PVAc adhesive widely used by the preservation community indicated that the adhesive was suitable only for temporary use, i.e. less than 6 months[1].

Several years later the Preservation Research & Testing Division (PRTD) tested a larger group of adhesives to identify one suitable for permanent use. Samples of PVAc, ethylene-vinyl acetate (EVA), and thermoplastic resin (hotmelt) adhesives were prepared and cured for 3, 7, 14, 21, or 30 days before Oddy testing. All PVAc and EVA adhesive samples tested as unsuitable. Interestingly, odors associated with those adhesives had diminished noticeably after a few days curing at ambient conditions, but no improvement in off-gassing after a longer cure time was observed. The hot-melt adhesive was recommended for permanent use after off-gassing for 3 or more days, but its working properties aren't appropriate for construction of cloth-covered clamshell boxes.

Oddy test jar modified with glass beads and ultra-pure metal coupons and center folds.

Photo Kelli Stoneburner



Failure of a lead coupon from a sample of PVAC cured 3 days, exhibiting layers of black, red and green corrosion products covering the entire surface, liquid droplets, surface delamination, and dense white extrusions adhering the coupon to the rim of the jar.

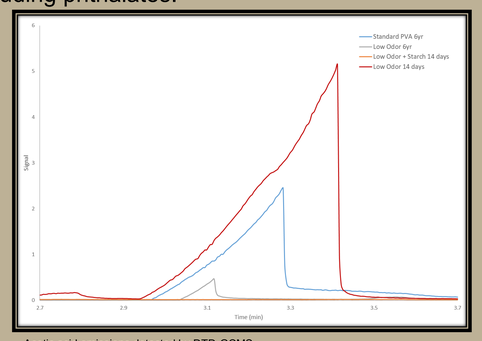
Photo Cindy Connelly Ryan

Boxes were air sampled after three to eight months. Similar classes of compounds were detected in all the boxes at varying ratios. Aldehydes, alcohols, hydrocarbons were the most common types of compounds but some ketones, carboxylic acids, esters, ethers, glycols were also detected.

The liquid and prepared hide glues were tested by Oddy test and DTD-GCMS (see table 2 for Oddy results). The prepared hide glue box air sample did not identify any compounds of concern. In contrast, liquid hide glue 1 there was a concerning level of carboxylic acids. For liquid hide glue 2, increased levels of chlorinated compounds were detected, which is not ideal. Similarly, the fish glue and PVAc both had elevated levels of carboxylic acids including acetic acid. An odor was also noted to be emitting from the fish glue box at the time of air sampling, eight months after construction.

In 2017, Oddy testing was augmented by direct thermal desorption-gas chromatography mass spectrometry [DTD-GCMS] to identify volatile organic compounds [VOC]. Among the five adhesives tested was a 1:9 mixture of low-odor PVAc and wheat starch paste, as well as two PVAc samples previously tested in 2011, which were re-tested after aging naturally for the intervening 6 years. All samples failed the Oddy test. Extensive corrosion covered and partially consumed the lead coupons. The copper coupons were rated Temporary/less than 6 months (T) or Unsuitable for display or storage case use (U). The silver coupons for the 14-day-cured adhesives were rated U. The 6-year-aged sample of low-odor PVAc passed only on the silver coupon, as it did in 2011 testing.[2]

A significant amount of acetic acid, 140 ppm relative to toluene, was released from 14-day-cured low-odor PVAc. Surprisingly, the two re-examined 2011 PVAc adhesives also released 5 and 50 ppm acetic acid, despite 6 years of natural aging. Dilution of PVAc with 9 parts wheat starch paste did not reduce its impact on the Oddy test coupons, although the amount of acetic acid detected in VOC analysis was very low, only 0.4ppm.[4] VOC analysis detected a range of compounds besides acetic acid in the samples, such as sulfur and plasticizers, including phthalates.[2]



Acetic acid emissions detected by DTD-GCMS

III) Alternative adhesives and their working properties

Four of the boxes made using the adhesives mentioned below.
Photo Kelli Stoneburner

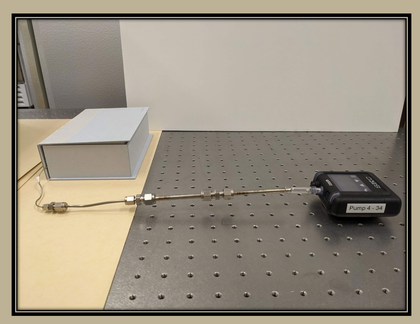
Beginning in 2022, boxes composed of book board and book cloth commonly used by the conservation lab were constructed for testing. Board from a single source and cloth from a single roll were chosen for uniformity and boxes were made to a standard size. A control box was constructed of book board and wheat starch paste without book cloth. The spine piece and case boards were attached with thin strips of long-fibered East Asian tissue to hold the box together. Adhesives included in this round of testing were determined by what was already on-hand in the conservation lab and what could be easily attained through usual supply vendors. Each conservator recorded notes on the working properties and length of working time before bonding (open time) for each adhesive. The results are presented in Table 1.

Table 1. Observations on adhesives used in box preparation

Adhesive	Preparation	Working Properties	Open times ranked 1-5 (least to most)
Wheat starch paste (WSP) (Control box – no cloth)	Hydrated in deionized water for minimum 30 minutes then cooked at high heat, stirring, until thick and translucent. Stirred while cooling. Double strained and slightly thinned with deionized water.	Wet, minimal tack.	5
PVAc	None	Easy to apply. Nice tack. Easy to reposition pieces while working.	4
Liquid hide glue 1	None	Viscous. Difficult to spread. Some strikethrough visible on off-white cloth.	3
Liquid hide glue 2	None	Viscous. Difficult to spread. Very tacky.	2
Liquid fish glue	None	Viscous. Strong odor. Very tacky.	2
Prepared hide glue	Swelled with water overnight. Heated gently in a bain-marie while working	Not viscous. Easy to spread, very wet, very tacky. Stains book cloth.	2
Wheat starch paste	Prepared as above	Very wet, minimal tack. Easy to reposition pieces. Very long drying time.	5
Wheat Starch Paste/Hide Glue	WSP and prepared hide glue prepared as above and mixed 1:1	Easy to spread like WSP but tacky from the hide glue. Faster drying time than straight wheat starch paste.	4

Table 2. Summarized DTD-GCMS air sampling results & Oddy testing

Sample	# Peaks	TVOC (µg/m³)	Acetic acid (µg/m³)	Oddy rating
Control box, no cloth with WSP	40	52	0.4	
PVAc	63	183	22	U
Liquid Hide Glue 1	71	366	11	U
Liquid Hide Glue 2	72	179	2	U
Prepared fish glue	59	607	71	
Prepared hide glue	63	156	6	P
WSP	62	169	3	
WSP/Hide glue	65	196	2	



Air sampling control box.
Photo Kelli Stoneburner

Of the other three boxes (WSP, WSP/hide glue, and control box), all but the control had very similar total VOC concentrations (TVOC) and emitted compounds that were very similar with only slight variations. One variation was in the WSP/hide glue box where a late eluting sulfur compound was detected in a small quantity. The largest variation of all eight boxes was the control WSP box as it emitted the least number of compounds and its TVOC was three times less than the next smallest TVOC.

IV) Testing Process/Methodology

Oddy

PVAc adhesive samples were prepared by brushing out a uniform thickness onto polyester film. Hide glues were poured out or melted, into aluminum weigh boats to cure for over a week. Tests were completed following the LOC modified version of the method outlined in *Bamberger et al. 1999*[3].

DTD-GCMS

Hide glues were poured out or melted, then poured into aluminum weigh boats to cure for over a week. PVAc samples were prepared by brushing a uniform thickness onto polyester film. Samples of the glues ranging from 14-19mg were analyzed using DTD-GCMS and the standard protocol GMW15634 / VDA278. Peak areas were integrated and used to calculate the concentration of each compound by comparing the signal to a toluene standard.

Boxes were air sampled by inserting a 1/16" metal tube through one of the small gaps in the box. The tube was attached to two Tenax TA tubes connected sequentially to a battery powered air pump with a constant flow rate of 50 mL/min for 157 min. The samples were then analyzed via DTD-GCMS utilizing the same protocol as above.

V) Conclusion

The current box making protocols at the Library of Congress reflect the experimentation and analysis results of the above adhesives. Decisions are based on several factors, including the type of material to be boxed, curatorial input, and time/cost of housing. In practical terms this means more custom-made corrugated board boxes and fewer clamshells made which now require more drying time and specialized adhesives. Experimentation continues. While Library of Congress conservators have a comfort level with the current approach to box making, we are still looking for adhesive(s) that will provide long term stability and ease of use.

The authors wish to stress that the adhesives considered unsuitable for box making as a result of testing have other, unrelated applications. In most cases they are conservation supply materials that were designed for other purposes. In casting a wide net for possible PVAc replacements many adhesives not designed for application to paper-based materials were considered and tried. Failure of one or more tests by these adhesives when used for the unique purpose of clamshell box making does not imply unsuitability for their other, intended purposes.

References

[1] Hengemihle, F. and Dekle, C. Draft [Library of Congress PRTD] internal report. 2004

[2] Connelly Ryan, C., Stoneburner, K., Monroe, E. [Library of Congress PRTD] Internal Report 0817 0241. September 8, 2017

[3] Bamberger, J., Howe, E., Wheeler, G. A variant Oddy test procedure for evaluating materials used in storage and display cases.*Studies in Conservation* 44 (1999) 86-90.

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