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Photographic Activity Tests of Various Adhesives Suggested for Use on Water-Sensitive Photographs

Jane Down, Joe Iraci. and Greg Hill

Abstract
Photographic Activity Tests (PATs) were run on various adhesives of interest to conservators for conserving silver-image water-sensitive photographs where aqueous adhesives might cause planar distortions. The results suggest that the ever popular Lascaux 360 HV and Lascaux 498 HV adhesives should not be used. Other products that were tested passed the PAT and are possible substitutes.

Keywords: photographic activity test, PAT, adhesive, Lascaux 360 HV, Lascaux 498 HV, Lascaux 303 HV, Paraloid B-72, BEVA 371, Plextol B500

Introduction

For more than 25 years, conservators have been using various acrylic adhesives (e.g. Lascaux 360 HV and Lascaux 498 HV) either in a pressure-sensitive, heat-set or solvent reactivation application for conserving silver-image photographs especially for water-sensitive photographs where aqueous wheat starch paste or methylcellulose solutions might cause planar distortions. Some of these acrylic adhesives were tested as part of a project on tapes and heat-set tissues carried out at the Canadian Conservation Institute (CCI) (Down et al., 2011) and were found to fail the Photographic Activity Test (PAT). This threw these adhesives into question so it was decided to look at them in more detail to see what could be recommended for silver-image photographic materials and to see if possible alternatives could be found.

Photographic Activity Test

The PAT (ISO 18916 2007) is a test used to detect possible chemical interactions between a material-in-question (e.g. paper, adhesive, etc.) and the photographic image (such as in silver-gelatin prints).

The test consists of constructing two different types of sandwich stacks consisting of the material to be evaluated and one of two detectors. The first sandwich is used to measure the propensity of the material being tested to stain gelatin and makes use of a Stain Detector. This detector is a piece of conventional fibre-based black-and-white photographic paper unexposed and processed so that the end product is a clear piece of gelatin on a white background. The second sandwich utilizes a detector that is unprocessed colloidal silver in gelatin on a polyester plastic base and is called the Image Interaction Detector. Colloidal silver is sensitive to oxidative or reductive processes initiated by the paper, adhesive, etc. The material being evaluated is not in direct contact with the detectors, but instead, Whatman #1 filter paper is used as a separator in order to prevent any physical interaction (such as adhesion) between the detectors and the material.

The whole assembly of detector, sample material, and filter paper is placed between two pieces of glass and under a small amount of weight to ensure the sandwich remains flat. The sandwiches are then placed in a temperature and humidity controlled chamber for 15 days at a

temperature of 70°C and relative humidity of 86%. Corresponding controls are also incubated alongside the test samples. For the controls, Whatman #1 filter paper is substituted for the adhesive samples.

The samples are evaluated using densitometry and via a visual test. Densitometry measures the yellowing of the Stain Detector by comparing the density readings before and after incubation. For the Image Interaction Detector, densitometry measures the fading or darkening of the colloidal silver film by once again comparing before and after readings. The Image Interaction detector is evaluated visually after incubation for what is called "mottling" or in other words patchy fading, darkening, or spotting which may not be picked up by the densitometry readings. Any mottling that is easily recognizable and any density changes in the detectors beyond a certain amount, when compared to controls, determines whether a material passes or fails the PAT. If the material-in-question fails any one of the three tests (i.e. image interaction, staining, or mottling) then it fails the PAT test.

Depending on the situation, the adhesive test samples placed in the sandwich were prepare by either heat-setting them to Whatman #1 filter paper, solvent reactivating them to the filter paper or simply placing them next to the filter paper without using either of the above processes.

PAT Results from the Tapes and Heat-set Tissues Project (2009)

The tapes and heat-set tissues project was initiated at CCI to help understand the nature and stability of different tapes and heat-set tissues and their impact on paper (Down et al., 2011). Besides running a PAT on the adhesive side and carrier side of all 42 tapes and heat-set tissues included in the project, the following tests were also performed: extracted pH, colour change of the carrier side of the product and of the paper substrates to which they were attached (reverse side), and mechanical and solvent removal of the products from 1870s commercial printing paper and resin-coated photographic paper before and after aging in the dark (some aging tests were done in ovens).

For the PATs, the tapes and heat-set tissues were adhered to Whatman #1 filter paper as they would have been applied in normal conservation practice. For the heat-set tissues, the products were heat-set onto Whatman #1 filter paper at temperatures recommended by the manufacturer using a dry mount press held at the recommended temperatures for 3 minutes. For the pressure-sensitive products, they were lightly tacked at either end of the strip onto the test papers and then pressed using a Roll Down Machine that delivered the same pressure to each strip. Water-activated and solvent reactivated products were also applied to the filter paper and then pressed using the Roll Down Machine to ensure that the same pressure was used for every sample.

The results of the PATs for the tapes and heat-set tissues can be seen in Table 1 (see Appendix) which is organized according to adhesive chemistry of the products. Only 43% of the products passed the PAT. Of particular interest to photographic conservation will be the following results:

- homemade wheat starch paste passed the PAT
- Library of Congress Heat-set Tissue (containing Plextol B500 and Rhoplex AC-73) passed the PAT
- Lascaux 360 HV, Lascaux 498 HV and the mixture of 360 and 498 (all heat-set) failed the PAT

- Seal Fusion 4000 Film and Colormount Tissue for dry mounting (both heat-set) both passed the PAT
- BEVA 371 Film which was heat-set passed the PAT.

The results obtained from the Lascaux products were particularly disturbing as these products have been used frequently in conservation as heat-set or solvent-reactivated adhesives for silver-image photographic materials when aqueous applications (e.g. wheat starch paste and methylcellulose solutions) cannot be used because they would cause planar distortions.

Additional PAT Results (2013)

Because the Lascaux products failed the PAT in 2009, it was decided to retest these products, look at them in more detail and test a few alternative ones that have been suggested for possible use in photographic conservation in hopes of finding a heat-set or solvent-reactivated type that might pass the PAT and be recommended for use on silver-image water-sensitive photographic materials. Thus, the following adhesives were tested without being heat-set onto the Whatman #1 filter paper as they were done in 2009. Instead, these adhesives were painted onto Whatman #1 filter paper full strength, dried for 72 hours and then sandwiched with another piece of Whatman #1 filter paper for the PAT.

- Rhoplex N-580
- Lascaux 360 HV (2009 and 2012 batches)
- Lascaux 498 HV (2009 and 2012-1 batches)
- Lascaux 303 HV
- Plextol B500
- Paraloid B-72 (in toluene)
- BEVA 371 Original Formula solution
- BEVA 371b solution (the new substitute for BEVA 371).

Also PATs were run at the Image Permanence Institute (IPI) on the following samples: Lascaux 498 HV and Plextol B500 both prepared on Hollytex as a backing[1]. A PAT of the Lascaux 498 HV on Hollytex was also run at CCI to see if the same results would be obtained as that from IPI.

The results of these PATs are given in Table 2 (see Appendix). It can be seen that the Rhoplex N-580, Lascaux 498 HV (samples done at CCI) and the Plextol B500 (samples done at CCI) all failed the PAT. On the other hand, the IPI samples for Lascaux 498 HV and Plextol B500 on Hollytex both passed the PAT which contradicts the CCI results for these samples. There are three possible reasons why the CCI and IPI results might differ for the Lascaux 498 HV on Hollytex. First, it should be noted that the same sample batch was tested at both locations. Table 2 shows that the reason for the Lascaux 498 HV on Hollytex failing the PAT at CCI was due to the mottling test. The mottling evaluation is subjective and it is possible that what was considered mottling in the CCI PAT Image Interaction Detector was not considered mottling at IPI. The second reason is inherent laboratory to laboratory variations. Even though the PAT is a standardized test and variations between laboratories, for the most part, are taken into consideration in the PAT results evaluation, borderline samples may still either fail or pass depending on where the test was performed. The third possible reason for a difference is that the

testing at CCI was performed one year later than at IPI. Sample storage, handling or aging might have caused these differences.

As for the Plextol B500 sample, the test samples were not exactly the same. The IPI sample was on Hollytex and the CCI sample had no backing. It is not suspected that this would be the cause for the different results. The more likely cause would be the subjective evaluation of the mottling test and variations between the laboratories causing the sample to narrowly fail the stain portion of the PAT.

It is also concerning that the new BEVA 371b did not pass the PAT while the old BEVA, which is no longer available, did pass. This is likely due to the formula change (Chludzinski 2010) in the new product which now is causing it to fail the PAT.

The results for the Lascaux 360 HV were somewhat confusing. The samples that were heat-set onto the Whatman #1 filter paper in 2009 failed the PAT while the samples run in 2013 (i.e. same batch used in 2009 and new batch from 2012) both passed the PAT. One difference between the samples was that no heat-setting was used for the samples run in 2013. Another difference was that the 2009 heat-set sample was treated as a label or tape and therefore the filter paper backing also acted as the separator between the adhesive and the detector. For the non-heat-set samples that were run in 2013 there was no backing and therefore a filter paper separator was required between the adhesive sample and the detector. This could be the reason why the samples in 2013 passed the PAT. It is likely that the heat-setting used in 2009 pushed the adhesive into the filter paper with the result that it was physically closer to the detector compared to the 2013 samples which were separated from the detector by the full thickness of filter paper (see Figure 1). Ultimately, the adhesive in 2009 was closer to the detector and more likely to cause a detrimental effect when compared to the non-heat set samples. It is suggested that the 2013 samples are the more accurate PAT results for this adhesive product.

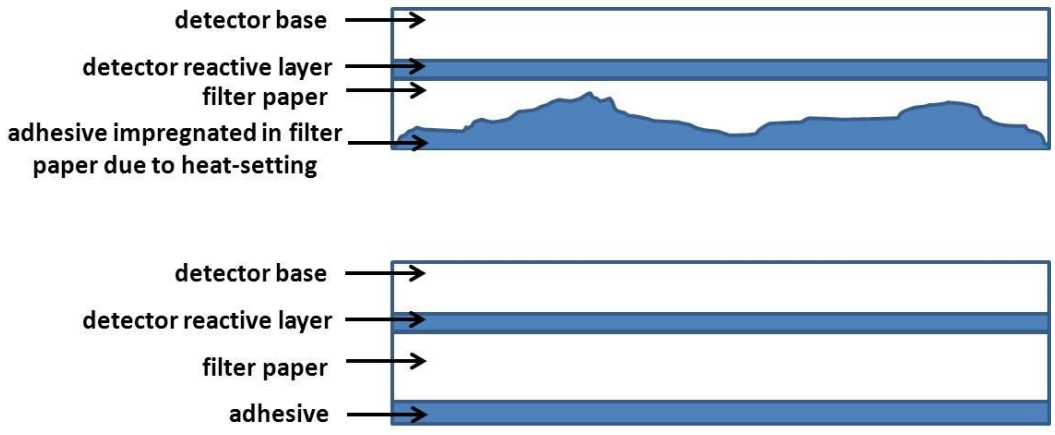

Figure 1. The top image shows the possible migration of adhesive into the filter paper caused by the heat-setting procedure. The bottom image shows the adhesive with no heat-setting and a filter paper barrier between it and the detector reactive layer. It is clearly evident that with heat-setting and no additional filter paper separator being used, the adhesive is much closer to the reactive layer of the detector.

The Lascaux 498 HV samples all failed the PAT except for the IPI sample as explained above. Because of the widespread use of this product with photographic materials, a further examination was required to see if a concentration of the adhesive could be found that would pass the PAT. PATs were performed at four different concentrations with and without heat-setting. The results are summarized in Table 3 (see Appendix). In either case, heat set or not, the trend is clear. As the concentration is lowered, the PAT result progresses closer to passing grade. Eventually, at a concentration of 25 percent for no heat-setting and 50 percent for the heat-set sample, the adhesive passed the PAT. However, at these low concentrations they are not very effective adhesives and do not adhere well making them essentially useless as for use in conjunction with photographic materials.

Since Lascaux 360 HV is no longer available and the Lascaux 303 HV is its substitute, PATs were performed on the Lascaux 303 HV. The adhesive was applied to the Whatman #1 filter paper using solvent reactivation, heat-setting, and without either one of these processes. The results are also given in Table 3 (see Appendix). Fortunately, it passed the PAT and it appears that there is no significant difference in the results, regardless of the method of application. Aging data on this adhesive is not available at this time. Analysis showed that it is 2-ethylhexyl acrylate/ethyl acrylate copolymer which is definitely different than the Lascaux 360 HV (Williams 2013).

Recommendations

Recommendations for adhesives for silver-image water-sensitive photographs might encompass the following products since Lascaux 360 HV, which has been used in the past, has been discontinued. If more aging and handling data on the Lascaux 303 HV becomes available in the future and shows it to be a stable useful product, it could be considered for use as it passed the PAT. Paraloid B-72 and the old BEVA 371 (film and solution) also passed the PAT and if they have the correct handling properties, they also could be considered. On the other hand, the mixed PAT results obtained from CCI and IPI for Lascaux 498 HV and Plextol B500 suggest that they should not be used on such photographs.

Notes

1. These samples were made and sent to IPI by Christophe Vischi, Assistant Conservator of Photographs, National Gallery of Canada.

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Table 1: Results of the PAT from the Tapes and Heat-set Tissues Project, Arranged According to Adhesive Type

Tape or Heat-set Tissue	WA PS HS	Adhesive Component Chemistry	Image (%) (pass is -20 to +20 %)	Stain (depending on run, pass is <19-22)	Mottling	Adhesive Overall PAT Rating
PROTEIN Containing Products						
Gummed Paper Hinging Tape	WA	Protein	pass 1.79	pass 0.10	fail	**FAIL**
Repa Tex G5	WA	Protein + MC	pass -4.02	pass 0.11	pass	**PASS**
Gummed Linen Hinging Tape	WA	Protein + Starch (>8:1)	pass -8.93	pass 0.10	fail	**FAIL**
Hinged Cambric Cloth Tape	WA	Protein + Starch (>8:1)	fail -40.18	pass 0.10	fail	**FAIL**
Perforated White Linen Tape	WA	Protein + Starch (>8:1)	fail -49.86	pass 0.09	fail	**FAIL**
STARCH Containing Products						
Homemade WSP (w Kurotani tissue)	WA	Starch	pass -2.59	pass 0.12	pass	**PASS**
Gummed Linen Tape	WA	Modified Starch	pass 12.71	pass 0.14	fail	**FAIL**
ACRYLIC - PnBA Containing Products						
filmoplast P	PS	PnBA + $CaCO_3$ + UN	pass -4.14	pass 0.12	pass	**PASS**
Frame Sealing Tape FST 1000	PS	PnBA	pass 0.75	pass 0.13	pass	**PASS**
Self-adhesive Linen Hinging Tape	PS	PnBA	fail 21.80	pass 0.11	fail	**FAIL**
Framer's Tape II #S2000	PS	PnBA	pass 19.08	pass 0.11	fail	**FAIL**
ACRYLIC - PnDA Containing Products						
Scotch Magic Tape #810	PS	PnDA	pass 6.24	pass 0.14	pass	**PASS**
Scotch Magic Removable Tape #811	PS	PnDA	pass 6.09	pass 0.15	pass	**PASS**
Photo & Document Repair Tape #001	PS	PnDA + PIB ++	pass -3.79	pass 0.12	fail	**FAIL**
Acid-Free Db-Stick Adhesive Pen #007	PS	PnDA + PIB ++	pass -6.67	pass 0.14	fail	**FAIL**
Scotch Adhesive Transfer Taper #924	PS	PnDA + small PAA	fail 30.68	pass 0.12	fail	**FAIL**
Double-coated Film Tape #415	PS	PnDA (+PAA)	fail 21.96	pass 0.12	fail	**FAIL**
ACRYLIC - PEHA Containing Products						
filmoplast P90	PS	PEHA + $CaCO_3$	pass -5.32	pass 0.12	pass	**PASS**
gudy 871	PS	PEHA + PVAC + soap	pass -1.78	pass 0.10	pass	**PASS**
gudy 831	PS	PEHA + PVAC + soap	pass -8.61	pass 0.12	fail	**FAIL**
filmoplast T Tape (black)	PS	PEHA + PVAC + talc/mica	pass 5.94	pass 0.12	fail	**FAIL**
filmolux 609	PS	PEHA + PVAC + PH	pass 14.81	pass 0.12	fail	**FAIL**
filmoplast SH	PS	PEHA + PVAC	pass 2.81	pass 0.18	fail	**FAIL**
filmomatt libre	PS	PEHA + PVAC	pass 8.40	pass 0.11	fail	**FAIL**
PH7-70 Conservation ATG Tape Perm	PS	PEHA + UN	pass 15.25	pass 0.12	fail	**FAIL**
Self-adhesive Frame Sealing Tape	PS	PEHA	pass -3.44	pass 0.15	fail	**FAIL**
ACRYLIC – PODA Containing Product						
filmoplast R	HS	PODA + UN	pass -1.85	pass 0.11	pass	**PASS**
ACRYLIC – PEA/PMMA Containing Product						
Library of Congress Heat-set Tissue	HS	PEA/PMMA	pass -5.11	pass 0.13	pass	**PASS**
ACRYLIC - PBA/PMMA Containing Products						
Lascaux 498 HV	HS	PBA/PMMA	fail -51.99	pass 0.12	pass	**FAIL**
Lascaux 360 HV	HS*	PBA/PMMA	fail -41.34	pass 0.12	pass	**FAIL**
Lascaux 498 HV/360 HV	HS	PBA/PMMA	fail -33.81	pass 0.11	pass	**FAIL**
ACRYLIC - PMA/PEMA Containing Products						
Unsupported Archibond	HS	PMA/PEMA	pass -1.85	pass 0.12	pass	**PASS**
Crompton Tissue	HS	PMA/PEMA	pass 5.97	pass 0.12	fail	**FAIL**
PVAC or VAE Containing Products						
Hayaku Gummed Japanese	WA	PVOH/PVAC	pass -1.38	pass 0.12	pass	**PASS**
Document Repair Tape	PS	PVAC-vinyl maleate	pass -1.68	pass 0.13	pass	**PASS**
Vinamul 3252	HS	VAE + VAL/VAC + NaCMC	fail -73.30	pass 0.16	fail	**FAIL**
EVA Containing Products						
Seal Fusion 4000 Dry Mounting Film	HS	EVA	pass -4.83	pass 0.13	pass	**PASS**
Seal Colormount Dry Mounting Tissue	HS	EVA + UN	pass -1.70	pass 0.13	pass	**PASS**
BEVA 371 Film	HS	EVA + KRN ++	pass 2.70	pass 0.13	pass	**PASS**
Rubber Containing Products						
Duck General Purpose Masking Tape	PS	Rubber + tackifier	pass -4.22	pass 0.13	pass	**PASS**
Scotch 893	PS	Rubber + PP	pass 0.84	pass 0.10	pass	**PASS**
SBR Containing Product						
Spray Adhesive Super 77	PS	SBR + rosin?	pass 5.96	pass 0.14	fail	**FAIL**

Abbreviations: $CaCO_3$ = calcium carbonate; EVA = ethylene/vinyl acetate copolymer; HS = heat-set product; KRN = ketone resin N; MC = methylcellulose; NaCMC = sodium carboxymethylcellulose; PAA = poly(acrylic acid); PEA = poly(ethyl acrylate); PEHA = poly(ethylhexyl acrylate); PEMA = poly(ethyl methacrylate); PH = phthalate; PIB = polyisobutylene; PMA = poly(methyl acrylate); PMMA = poly(methyl methacrylate); PnBA = poly(butyl acrylate); PnDA = poly(decyl acrylate); PODA = poly(octadecyl acrylate); PP = polypropylene; PS = pressure-sensitive product; PVAC = poly(vinyl acetate); PVOH = poly(vinyl alcohol); SBR = styrene butadiene; UN = unknown; VAC = vinyl acetate; VAE = vinyl acetate/ethylene copolymer; VAL = vinyl alcohol; WA = water-activated products; WSP = wheat starch paste.
* Is also used wet, as a PS product or solvent-reactivated.

Table 2: Comparison of Results of the PATs for Various Adhesives

Adhesive					Results of the PAT			
Batch	**Heat-set**	**Backing**	**Run Date**	**Polymer Chemistry**	**Image (%) (pass is -20 to +20)**	**Stain (pass is <0.20; Control 0.11)**	**Mottling**	**Overall**
Rhoplex N-580								
2012	no	none	2013	BA	fail -25.96	pass 0.13	fail	**FAIL**
Lascaux 360 HV								
2009	51.5°C	tissue	2009	>>50% BA/MMA	fail -41.34	pass 0.12	pass	**FAIL**
2009	no	none	2013		pass -14.94	pass 0.11	pass	**PASS**
2012	no	none	2013		pass -4.43	pass 0.13	pass	**PASS**
Lascaux 498 HV								
2009	75.5°C	tissue	2009	56% BA/MMA	fail -51.99	pass 0.12	pass	**FAIL**
2009	no	none	2013		fail -23.76	pass 0.11	pass	**FAIL**
2012-1	no	none	2013		fail -26.74	pass 0.11	pass	**FAIL**
2012-2	no	Hollytex	2013		pass 9.83	pass 0.13	fail	**FAIL**
2012-2	no	Hollytex	2012 IPI		pass -1.26	pass 0.13	pass	**PASS**
Lascaux 303 HV								
2013	no	none	2013	EHA/EA	pass -7.68	pass 0.12	pass	**PASS**
Plextol B500								
2012-1	no	none	2013	66% EA/34% MMA	pass 14.63	fail 0.20	fail	**FAIL**
2012-2	no	Hollytex	2012 IPI		pass -1.31	pass 0.12	pass	**PASS**
LOC HST	88.4°C	tissue	2009	56% EA/44% MMA	pass -5.11	pass 0.13	pass	**PASS**
Paraloid B-72								
in toluene	no	none	2013	70% EMA/30% MA	pass 2.31	pass 0.12	pass	**PASS**
BEVA								
371Film	60-62°C	tissue	2009	EVA + KRN ++	pass 2.70	pass 0.13	pass	**PASS**
371Solution	no	none	2013		pass 5.42	pass 0.19	pass	**PASS**
371b Solution	no	none	2013	EVA + AKR ++	fail 23.31	fail 0.20	fail	**FAIL**

Abbreviations and Notes: AKR = aldehyde ketone resin; BA = butyl acrylate; EA = ethyl acrylate; EHA = 2-ethylhexyl acrylate; EMA = ethyl methacrylate; EVA = ethylene vinyl acetate copolymer; HS = heat-set; KRN = ketone resin N; LOC HST = Library of Congress Heat-set Tissue which contains Plextol B500 and Rhoplex AC-73; MA = methyl acrylate; MMA = methyl methacrylate; ++ means there are more components present. All samples were run at CCI except those labeled IPI which were run at the Image Performance Institute.

Table 3: Results of the PATs for Lascaux 498 HV Prepared at Different Concentrations With and Without Heat-setting and the Results of Lascaux 303 HV With and Without Heat-setting and Acetone Reactivating

Adhesive			Results of PAT			
Batch	**Concentration of Adhesive (%)**	**Heat-set or Solvent Reactivated**	**Image (%) (pass is -20 to +20 %)**	**Stain (pass is <0.23; Control is 0.14)**	**Mottling**	**Overall**
Lascaux 498 HV						
2012-1	100	no	fail -29.13%	pass 0.13	pass	**FAIL**
	75		fail -25.71%	pass 0.12	pass	**FAIL**
	50		fail -23.26%	pass 0.13	pass	**FAIL**
	25		pass -14.10%	pass 0.12	pass	**PASS**
	100	75°C	fail -27.42%	pass 0.12	fail	**FAIL**
	75		fail -24.61%	pass 0.13	pass	**FAIL**
	50		pass -19.84%	pass 0.12	pass	**PASS**
	25		pass -13.73%	pass 0.12	pass	**PASS**
Lascaux 303 HV						
2013	100	no	pass -7.38%	pass 0.14	pass	**PASS**
		50°C	pass -14.10%	pass 0.14	pass	**PASS**
		acetone activated	pass -13.00%	pass 0.13	pass	**PASS**