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PRELIMINARY TESTING OF ADHESIVES USED IN PHOTOGRAPH CONSERVATION

Kimberly Schenck* and Constance McCabe**

INTRODUCTION

When choosing an adhesive, a conservator takes many things into consideration, including the materials and construction of the object, the strength of adhesion required, and the stability, reversibility, and working properties of the adhesive. To make an informed choice, the conservator should be aware of the aging characteristics of the adhesive and its effect on the substrate.

To better understand the effects of adhesives on photographic materials, a series of accelerated aging studies were undertaken using a range of photographic detectors in contact with various adhesives used or encountered during conservation treatment. These adhesives were selected because they have been used for mending, mounting, and consolidation and as inpainting media or coating materials. In addition to gaining insight on the effects of these adhesives on photographs, another goal of the research was to formulate ideas and procedures for further experiments.

EXPERIMENTAL

ADHESIVES

The aging studies examined the following adhesives:

- Wheat starch paste (Atex-P, milky thin consistency)
- Methyl cellulose (Process Materials, 2% aqueous solution)[1]
- Polyvinyl acetate AYAA/AYAC (2:3, 3% solution in toluene)[2]
- Elvace 1874 vinyl acetate dispersion:deionized water (3:2)[3]
- Seal MT5 Dry Mounting Tissue[4]
- Acryloid B72 acrylic resin (3% in toluene or xylene)[5]
- Rhoplex AC73 acrylic dispersion[6]
- Plextol B500 acrylic dispersion[7]
- Rhoplex AC73:Plextol B500:deionized water (1½:1:1)[8]
- Rhoplex AC234 acrylic dispersion
- Rhoplex AC234:methyl cellulose
- Liquitex Acrylic Gloss Medium
- Rubber Cement (Carter's Rubber Cement and Best-Test Paper Cement)

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Several dispersions and mixtures were tested because they offer numerous application possibilities. Dispersions, or emulsions, can be applied directly as water-based adhesives or they can be activated after drying by using solvents or heat. They can be diluted with water and some organic solvents. Methyl cellulose can be added to alter viscosity and to theoretically increase reversibility. The stability of dispersions has been questioned due to the uncertain reliability of numerous unspecified ingredients in the solutions. In addition to the resins present, secondary ingredients may include polymer initiators, surfactants, plasticizers, buffers, modifiers, and preservatives. Manufacturers do not provide specific information on the secondary ingredients incorporated in the dispersions and may vary the ingredients from batch to batch.

DETECTORS

Tests were carried out using different photographic detectors. These detectors were used to indicate how the various adhesive samples might effect image silver, common binder layers, and paper supports. Presence of staining, fading, mottling, spotting, darkening, or change in surface appearance were factors used in determining the effects of the adhesives on the detectors.

The detectors included albumen step tablets[9], printed-out silver gelatin step tablets[10], developed-out silver gelatin step tablets[11], colloidal silver in gelatin on polyester film (detectors of oxidative attack), and fixed-out developed-out silver gelatin photographic paper (stain detectors). Step tablets incorporate a progression of high to low image desities. Colloidal silver detectors appear a transparent yellow-orange color due to the size, shape, and distribution of the silver particles. DOP stain detectors appear white in order to detect any staining of the gelatin binder.

TESTS CONDUCTED

TEST GROUP 1: SAMPLES MOUNTED ONTO FILTER PAPER

Detectors: Albumen step tablets.

Sample Preparation: Recently-made albumen photographic paper was mounted onto Whatman Chromatography Paper #1 by applying the adhesives onto both substrates. The mounted samples were dried between polyester web and blotters under glass. Seal MT5 Dry Mounting Tissue was heated with a tacking iron (116°-120° C) to adhere the albumen paper to the filter paper. The acrylic dispersions were coated onto filter paper, air-dried for eight days, and sprayed with acetone:xylene (1:1) to reactivate the adhesives before mounting the albumen paper onto the filter paper. Rhoplex AC234 and Rhoplex AC73:Plextol B500 were also applied directly as stated above.

The samples were dried for one month to allow for proper film formation of the dispersion adhesives before aging. Dispersions form proper films at room conditions in one to two months.[12] During the film formation period, drastic changes in temperature or humidity, like those used in accelerated aging tests, may cause inadequate films possibly skewing any test results.

Aging Conditions: Thermal Aging: One set of samples were suspended from glass rods between interleaving polyester strips inside glass dessicators and were incubated at 50°±1° C and 86%±2% relative humidity (RH) for 30 days as described in the ANSI PH1.53 Photographic Activity Test. A saturated solution of potassium nitrate was used to maintain the desired relative humidity.

Light Aging: A second set of samples were incubated in a Sunlighter apparatus which produced visible and ultraviolet radiation. The following adhesive samples were light aged: wheat starch paste, methyl cellulose, and acrylic dispersions. The samples were exposed to over 1000 footcandles for 20 days (480,000 footcandle-hours) at 41°±2° C and 20%±5% RH.

Controls: Unmounted samples were thermally aged and light aged as stated above. One set of mounted samples were not aged.

TEST GROUP 2: DETECTORS IN CONTACT WITH FILTER PAPER/ADHESIVE SANDWICHES

Detectors: Colloidal silver detectors for oxidative attack, fixed-out DOP stain detectors, developed-out silver gelatin step tablets, and printed-out silver gelatin step tablets.

Sample Preparation: To avoid adhesion of the samples onto the detectors, the adhesives were applied between two sheets of Whatman Chromatography Paper #1 to form "sandwiches". These adhesive sandwiches were then dried between blotters for one month.

Aging Conditions: The sandwiches were placed into contact with the detectors and were incubated at 70°±1° C and 86%±2% RH for 15 days using glass dessicators. The incubation conditions and the stacking of test materials followed guidelines in the ANSI IT9.2 Photographic Activity Test. A saturated solution of potassium nitrate was used to maintain the desired relative humidity.

Controls: Filter paper strips without adhesives were placed into contact with the detectors during aging.

TEST GROUP 3: DIRECT APPLICATION OF ADHESIVES ONTO DETECTORS

Detectors: Colloidal silver detectors, fixed-out DOP stain detectors, and gelatin DOP step tablets.

Sample Preparation: Adhesives were applied directly onto the detectors using cotton swabs. The colloidal silver detectors changed in color saturation where the water-based adhesives were applied. Even though the changes were permanent, it was hoped that results from the accelerated aging conditions could still be evaluated.

Aging Conditions: After air-drying for one week, the samples were suspended from glass rods between interleaving polyester strips and were incubated at 70°±1°C and 86%±2% RH for 15 days.

After aging, the adhesives were partially removed from one sample in each group using organic solvents or water in order to better evaluate any changes without visual interference from the adhesives.

Controls: Uncoated samples were aged as stated above. One set of unaged coated detectors were retained to compare with the aged samples.

TEST GROUP 4: ADHESIVES APPLIED ONTO FILTER PAPER

Sample Preparation: Adhesives were coated onto filter paper and air-dried for one month.

Aging Conditions: Thermal aging: The samples were suspended from glass rods between interleaving polyester strips inside glass dessicators and were incubated at 50°±1° C and 86%±2% RH for 30 days.

Light aging: Another set of samples were exposed in the Sunlighter apparatus to over 1000 footcandles for 20 days (480,000 fc-hrs.) at 41°±1° C and 20%±5% RH.

Controls: Uncoated filter paper strips were aged as stated above. One set of coated samples were not aged.

OBSERVATIONS
(See chart for overview of results.)

TEST GROUP 1: SAMPLES MOUNTED ONTO FILTER PAPER

Thermal aging: All albumen samples suffered slight yellowing and fading with little difference noted among the various samples. Samples mounted with Seal MT5 appeared slightly yellower than other samples due to the yellow color of the tissue itself. After the tissue was partially removed from one sample, the sample appeared the same color as the other adhesive samples.

All samples adhered with PVA AYAA/AYAC developed faded spots or mottling. Two samples mounted with Rhoplex AC234:methyl cellulose developed slightly faded spots. A few faded spots developed on only one sample of the following adhesives: wheat starch paste, methyl cellulose, Seal MT5, and Elvace. Seal MT5 samples exhibited a few

yellowed areas which could have been due to longer heat exposure in those areas during application.

No differences were noted between the albumen samples mounted with the acrylic dispersions which were applied as water-based adhesives and those dispersion samples which were dried and reactivated with solvents.

Light aging: All albumen samples yellowed and faded dramatically, but little difference was noted between adhesive samples.

TEST GROUP 2: DETECTORS IN CONTACT WITH FILTER PAPER/ADHESIVE SANDWICHES

Colloidal Silver Detectors: All colloidal silver samples faded after aging except for samples in contact with the Acryloid B72 sandwiches which were similar in color to the unaged sample. The dry mounting tissue samples and possibly the rubber cement samples were also slightly darker when compared with the other samples.

Some samples exhibited faded or orange spots. All colloidal silver samples in contact with the acrylic dispersions exhibited faded spots or mottling. Spotting was most frequently observed in the Rhoplex AC73:Plextol B500 samples and was least frequently observed in the Rhoplex AC234 and Liquitex Gloss Medium samples. At least one sample in contact with the following materials exhibited a few orange spots: filter paper, Elvace 1874, Plextol B500, Rhoplex AC73:Plextol B500, Rhoplex AC234:methyl cellulose, and Liquitex Gloss Medium.

The rubber cement adhesive inside the sandwiches was discolored.

Fixed-Out DOP Stain Detectors: All samples yellowed slightly after aging. The most severe yellowing occurred in samples in contact with the Rhoplex AC73, Plextol B500, and Rhoplex AC73:Plextol B500 sandwiches. The Elvace 1874, Rhoplex AC234:methyl cellulose, and rubber cement sandwiches also caused some yellowing. Samples in contact with the Acryloid B72 sandwiches may have yellowed slightly more than the aged controls.

Gelatin DOP Step Tablets: All samples yellowed slightly after aging. However, no difference was noted between samples in contact with the adhesive sandwiches and the uncoated filter paper.

Gelatin POP Step Tablets: All samples yellowed, faded, and mottled in the highlights to varying extents. Samples in contact with methyl cellulose and Plextol B500 sandwiches faded and yellowed less than the other samples. Samples in contact with the dry mounting tissue, Rhoplex AC73, and Rhoplex AC234 sandwiches faded slightly more than the other samples. Tiny faded spots were most prevalent on the samples in contact with the Elvace 1874 sandwiches. The results were inconsistent in the samples in contact with the Acryloid B72, Rhoplex AC234:methyl cellulose, and rubber cement sandwiches.

TEST GROUP 3: DIRECT APPLICATION OF ADHESIVES ONTO DETECTORS

Colloidal Silver Detectors: Most colloidal silver samples faded after aging, but some samples exhibited orange areas. The rubber cement samples were a mottled orange color overall. The colloidal silver samples darkened where the Elvace dispersion had been applied particularly heavily. The Seal MT5 samples exhibited an orange halo around the adhered tissue, but faded where the tissue was applied directly. Also, the Liquitex Medium exhibited areas of slight darkening where the coating was heavily applied.

As the water-based adhesives caused a lightening, or loss in density, of the colloidal silver strips during application, the samples could not be reviewed discriminately, but some comparisons were made. The PVA AYAA/AYAC samples and possibly all the acrylic dispersion samples, except perhaps the Rhoplex AC73, faded slightly. The Acryloid B72 samples were ambiquous as one sample faded where the adhesive was applied whereas the other sample faded more around the adhesive.

A few tiny orange spots were noted on at least one colloidal silver sample of the following adhesives: PVA AYAA/AYAC, Elvace 1874, Rhoplex AC234:methyl cellulose, and rubber cement.

Changes in the adhesives themselves were noted in three sample groups. Rhoplex AC73 and Rhoplex AC73:Plextol B500 adhesive films cracked slightly after aging. The rubber cement adhesive yellowed.

Gelatin DOP Stain Detectors: All stain detectors yellowed slightly, but no difference was noted between the adhesive samples.

Gelatin DOP Step Tablets: All DOP step tablets coated with acrylic dispersions faded in the areas where the adhesives had been applied. The rubber cement samples also appeared to have suffered slight fading Samples coated with Elvace 1874 faded with a color shift to blue-grey.

Partial Removal of Adhesives: Reversibility tests were not undertaken, but some quick observations were made when the aged adhesives were partially removed from the samples. Most adhesives remained soluble in water or organic solvents (acetone, xylene, or toluene). However, the dry mounting tissue was very difficult to remove with organic solvents or with heat. Rhoplex AC73 was not completely soluble in organic solvents, but the solubility increased when mixed with Plextol B500. Liquitex was only partially soluble in organic solvents.

TEST GROUP 4: ADHESIVES APPLIED ONTO FILTER PAPER

When initially removed from the aging ovens, little difference between the unaged samples and the thermal or light aged samples was apparent. However, after six months, the dispersion adhesive samples which had been light aged had yellowed slightly. The most yellowing occurred in the Rhoplex AC73:Plextol B500 sample. The least yellowed of the dispersions was the Elvace 1874.

DISCUSSION

The results were not dramatic, but overall the testing did allow limited conclusions to be drawn concerning the reliability of some adhesives used in photograph conservation. The traditional adhesives of wheat starch paste and methyl cellulose consistently produced the best results in all tests as they did not appear to damage the photographic substrates. Some adhesives caused deterioration problems in most tests while others produced inconsistent results from test to test.

The acrylic dispersions and to a lesser degree the Elvace 1874 dispersion gave unfavorable results in most tests. This could be due to ingredient instability or could be related to numerous changes occurring in the dispersion films during aging. Secondary ingredients in the dispersions may volatilize only at elevated temperatures or they may become instable as the films soften. A change of pH can occur during aging which could also effect the photographic substrates. It is uncertain what caused the deterioration of the photographic detectors and whether this deterioration would occur under room conditions.

In addition to stability problems of some of the adhesives, two questions concerning practical application should be considered. They include the reversibility of dry mounting tissue and the possible environmental barrier effects of Acryloid B72. These issues need to be explored further as they could effect treatment decisions.

As stated in the introduction, one goal of the research was to formulate ideas and procedures for further experiments. A few observations were made concerning testing procedures which will be incorporated in future testing:

1. Tests will be carried out using various photographic detectors as each test and detector offer valuable information.

2. More samples per adhesive need to be tested to avoid ambiguous results and to provide thorough statistical analysis.

3. Samples will be evaluated immediately after accelerated aging and several months or even years later to gain more information concerning the adhesives and their aging properties. Some deterioration may not be visible until long after the initial aging tests. However, changes in the detectors were sometimes apparent within days, especially in the case of the colloidal silver detectors. This is important in order to standardize interpretation of test results.

4. Natural aging studies will be carried out to compare with the accelerated aging studies. Problems exist with the accelerated aging tests. Some adhesives may perform poorly under elevated

temperatures and relative humidities due to rheological changes or to volatilization of ingredients. Accelerated aging tests do provide valuable information which should be considered with other research results.

5. Rubber cement, although it did not perform well in these tests, did not fail as dramatically as expected. The sensitivity of these testing procedures may not be adequate for determining the suitability of some adhesive types for use with photographs. It is important when specifying adhesives for use with photographic materials, that certain "known bad actors" are disallowed when purchasing storage enclosure materials or supplies to be used in close proximity to photographs.

CONCLUSION

The testing of materials used in conservation often results in the formation of more questions than in the providing of answers. Further studies of adhesives and other related materials must be carried out to enable conservators to make informed treatment decisions. Reliability is just one factor, albeit a major one, to consider in deciding treatment materials. However, other properties of the adhesives may play an equally important role in the decision-making process.

ACKNOWLEDGEMENTS

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REFERENCES

1 AYAA and AYAC are two grades of polyvinyl acetate with different melting points and viscosities.
2 Elvace 1874 is a vinyl acetate/ethylene copolymer dispersion.
3 Seal MT5 Dry Mounting Tissue may be composed of a glassine paper base with a rubber/wax/vinyl acetate adhesive coating.
4 Acryloid B72 is an ethyl methacrylate/methyl acrylate copolymer resin. (C.V. Horie, Materials for Conservation, Butterworths, 1987.)
5 Published information concerning the resin component(s) of Rhoplex AC73 unavailable at this time.
6 Plextol B500 is an ethyl acrylate/methyl methacrylate/ethyl methacrylate dispersion. (C.V. Horie, Materials for Conservation, Butterworths, 1987.)
7 Library of Congress adhesive recipe for heat-set tissue.
8 Published information unavailable at this time. Rhoplex AC234 may be an ethyl acrylate, but other resins may be present.

9 Albumen paper was purchased from the Chicago Albumen Works, and was printed and processed by the authors.
10 Kodak Studio Proof printed-out silver gelatin paper.
11 Azo F (glossy) Grade 2 developed-out silver gelatin paper.
12 R.L. Feller, "Polymer Emulsions", On Picture Varnishes and Their Solvents, Feller, Stolow, and Jones, Washington, National Gallery of Art, 1985 (revised edition), p. 221.

TESTS AND DETECTORS

ADHESIVES	MOUNTED ALBUMEN		SANDWICHES				DIRECT APPLICATION			FILTER PAPER	
	Thermal Aging	Light Aging	Col Ag	Gel Stain	DOP Step	POP Step	Col Ag	Gel Stain	DOP Step	Thermal Aging	Light Aging
Wheat Starch Paste											
Methyl Cellulose											
PVA AYAA/AYAC	◇						● ▽				
Elvace 1874			▽	■		◇	▲ ▽		● ○		■
Seal MT5			▲			●	● ▲halo		●?		
Acryloid B72			▲▲	■?							
Rhoplex AC73			◇	■■		●	●?		●●		■
Plextol B500			◇ ▽	■■			●		●●		■
AC73:B500			◇◇ ▽	■■			●		●●		■■
Rhoplex AC234			◇			●	●		●●		■
AC234:Methyl Cel	◇		◇ ▽	■			● ▽		●●		■
Liquitex Medium			◇ ▽				● ▲		●●		■
Rubber Cement			▲?	■			▲ ▽		●		

KEY

■ yellowed
● faded
▲ darkened
◇ mottling
▽ orange spots
○ color change

Designations on chart point out the most obvious cases of deterioration, even though many or all samples exhibited changes.