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Conservation of Broken Dry Plate Negatives from Francis Amin's Private Collection – A Scientific Assessment of Selected Adhesives

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Abstract

Institutions housing photographs and documents (e.g. archives) usually include large collections of glass plate negatives of significant historical value among their collection. Gelatin dry plates were the most common negative process in the years between 1880s and the 1920s. Gelatin dry plate negatives consist of a layered structure. This structure can be divided into three components: the primary support, glass; the binder layer, gelatin; and the final image material, metallic silver grains. As a result, dry plate negatives have a complex physical and chemical nature that must be taken into consideration if they are to be preserved into the future. One common preservation issue presenting a true challenge to photograph conservators is the treatment of broken glass negatives, mainly caused by improper handling and misuse, but also as a result of disasters (e.g. earthquakes). There are two different approaches for assembling broken glass negative: i) by preparation of a non-adhesive housing mat, and ii) by using an adhesive such as Paraloid B72 and epoxies. This study aims at assessing three different types of adhesives for use in the assembly of broken fragments of dry plate negatives. Tested adhesives were exposed to humid heat artificial ageing at a temperature of 80°C and 65% RH for 5 day. Evaluation was carried out using several techniques including visual inspection, colorimetric measurements, bursting test and Fourier transform infrared spectroscopy (FT-IR). The adhesive with the best results was used for further testing to evaluate its long- term efficiency when used to repair dry glass negatives by means of visual inspection, microscopic inspection, colorimetric measurements, FTIR and burst strength test. The second part of the study includes the treatment of six dry glass plate negatives, from Dr. Francis Amin's private collection, a famous Egyptian photo collector. The negatives mainly suffered from surface dirt, silver mirroring and breakage.

Keywords

- *Gelatin Dry Plate Negatives.*
- *Broken.*
- *Adhesives.*
- *Epoxy.*
- *Visual Inspection.*
- *Microscopic Inspection.*
- *Colorimetric Measurement.*
- *FTIR.*
- *Adhesion Strength.*
- *Conservation.*

1. Introduction

Museums, libraries, research institutes and archives in particular hold some form of photographic collection. Due to their artistic and documentary value, the interest in photographic materials is growing and many institutions

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are making immense efforts to expedite access to photographic collections, as well as preserve originals for future generations **[1]**. Since the birth of photography in the late 1830s, many photographic techniques and materials have been used to produce positive and negative images **[2]**. Each type of these photographic processes is subject to deterioration and/or degradation by slightly different factors; resulting in distinctive deterioration characteristics. Anyhow, institutions housing photographs and documents (e.g. archives) usually include large collections of glass plate negatives of significant historical value among their collection **[3]**. A negative is photographer's implement, a stepping stone on the way to make a positive photo **[4]**.

The use of glass plate negatives began around 1851. The first glass plate negatives, known as wet plate negatives used collodion, and were in use from 1851. Later in 1880, wet plate negatives were replaced by dry plate negatives, which used a silver gelatin emulsion that was applied not by the photographer, as in the case of wet plate process, but by a manufacturer **[5]**. It was in 1971 when Richard Leach Maddox successfully developed a workable gelatin dry plate negative in 1971. However, this process was slow compared to the collodion wet plate process **[6]**, **[7]**. Many significant steps were made to improve Maddox's process **[8]**. However, it was not until 1878 that Charles Bennett produced gelatin dry plates with his ripening process **[9]**. These were about ten times as sensitive as the collodion wet plates and gave images of great clarity **[8]**. The process was commercially viable in 1878 by the Liverpool Dry Plate Company, Wratten & Wainwright and many others followed **[7]**. Gelatin dry plates were the most common negative process in the years between 1880s and the 1920s when they were gradually replacedby cellulose acetate and cellulose nitrate negatives **[10]**. The dry plate process is based on the light sensitivity of silver halides, in

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most cases silver bromide, which are suspended in a gelatin binder on a glass support **[9]**, **[11]**, **[12]**, **[13]**. They have glass plates of different dimensions and of thickness of the order of 1-2 mm as a support. The gelatin emulsion is of thickness of the order of 50 micrometers **[10]**. During exposure a latent image is formed, which is chemically developed to produce a visible image. The image is then fixed and washed to obtain a permanent image **[13]**.

Similar to most photographic materials, gelatin dry plate negatives consist of a layered structure. This structure can be divided into three components: the primary support, glass; the binder layer, gelatin; and the final image material, metallic silver grains **[14]**. As a result, dry plate negatives have a complex physical and chemical nature that must be taken into consideration if they are to be preserved into the future. The most likely source of glass for gelatin dry plates was soda lime cylinder glass and crown glass **[11]**, **[15]**, **[16]**. This is consistentwith the fact that early glasses in the western world were almost all soda– lime–silica compositions that varied depending upon the availability of raw materials **[17]**. However, the basic ingredients are: amorphous silicon dioxide (i.e. SiO_2), soda (i.e. sodium carbonate, Na_2CO_3) or potash, a compound or flux to lower the melting point, and a stabilizer such as lime (i.e. calcium oxide, CaO) to restore insolubility **[18]**. Cylinder glass was produced by blowing a bubble of molten glass to form a cylinder that was cut along its length and flattened while the glass was still sufficiently soft. On the other hand, crown glass was produced by blowing a bubble which was then opened and spun round until it formed a disc of glass **[19]**. These two types of glass had many imperfections since both procedures physically altered the molten glass into one shape then flattened **[11]**. The binder layer holding the silver grains is composed of gelatin, an organic material extracted from collagen which is the abundant protein present in bones and skins **[20]**; in the case of photographic

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materials, gelatin is obtained from hides and ears of calves and pork **[21]**. Metallic silver particles are suspended in the binder layer as twisted strains known as filamentary silver **[22]**.

The threats affecting the permanence of dry plate negatives are many, including: improper temperature and relative humidity levels, light, air pollution, improper handling and misuse, poorstorage and display materials, biological threats and disasters **[23]**, **[24]**. Additionally, one must not overlook the inherent instabilities of such photographs as most of the stability problems associated with glass negatives result from their physical and chemical nature. Nevertheless, preservation priority of glass negatives is often underestimated because of the misconception thatglass is durable; and therefore less vulnerable compared to other archival materials **[11]**.

One common preservation issue presenting a true challenge to photograph conservators is the treatment of broken glass negatives, mainly caused by improper handling and misuse, butalso as a result of disasters (e.g. earthquakes) **[25]**. There are two different approaches for assembling broken glass negative: i) by preparation of a non-adhesive housing mat **[26]**, **[27]**, **[28]**, **[29]**, **[30]** and ii) by using an adhesive such as Paraloid B72, which has been used in severalcases **[25]**, **[31]**; and epoxies **[32]**.

This study aims at assessing three different types of adhesives for use in the assembly of broken fragments of dry plate negatives. Tested adhesives were exposed to humid heat artificial ageing at a temperature of 80°C and 65% RH for 5 days **[33].** Evaluation was carried out using several techniques including visual inspection, microscopic inspection, colorimetric measurements, and Fourier transform infrared spectroscopy (FT-IR). The adhesive with the best results was used for further testing to evaluate its long-term efficiency when used to repair dry glass negatives by means of visual inspection, microscopic inspection, colorimetric measurements, FT-

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IR spectroscopy and adhesion strength test. The second part of the study includes the treatment of six dry glass plate negatives, from Dr. Francis Amin's private collection, a famous Egyptian photo collector from Luxor, Egypt. The negatives mainly suffered from surface dirt, silver mirroring and breakage.

1. Scientific evaluation of glass adhesives for assembling broken glass negatives.

1.1. Materials and Methods

2.1. 1. Test materials

Two sets of samples were prepared for this study: glass slides and naturally aged broken glass transparencies. The latter was not selected as the main test material to eliminate the effectof potential variables. However, they were still included to give a more practical, rather than theoretical focus to this study **(as shown in Figure 1)**.

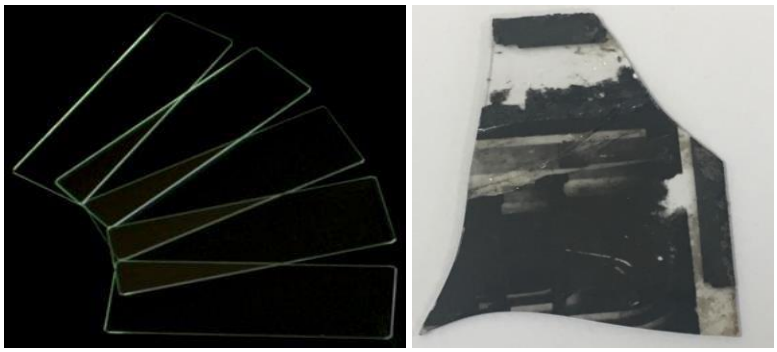

Figure 1. Test samples. Glass slides (left) and aged transparency (right)

2.1. 2. Adhesives

Four adhesives were selected: Epoxy euxit 50, four minute epoxy, Super glue and EH glue (as shown in Figure 2). All tested adhesives are commercially available for a considerable price.

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Figure 1. Selected adhesives for scientific testing.

2.1. 2. Application method

Three of the adhesives required mixing the monomer and hardener in the recommended proportions. Each adhesive was then applied on four glass slides using a brush. For the naturally aged samples, the adhesives were applied using a needle. Selected adhesives were applied on the edges of broken fragments to secure them together **(as shown in Figure 3)**. Table 1 shows the sample numbers for glass slides according to used adhesive type.

Figure 3. Applying adhesives to glass slides by brush.

Table 1. Sample numbers.

Sample Number	Adhesive type
E1, E2, E3, E4	Epoxy euxit 50
EH1, EH2, EH3, EH4	EH glue
EP1, EP2, EP3, EP4	Four minute epoxy
S1, S2, S3, S4	Super glue

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2.1. 3. Artificial aging

Ageing was carried out in a Binder Dry Oven with Digital Indicator. Samples were aged at80°C and 65% RH for 5 days at the National Institute of Standards.

2.1.4. Assessment methods

2.1.4. 1. Visual inspection

Visual observation is included to monitor visual changes. A photographic survey wascarried out before and after treatment and artificial aging in ambient light.

2.1.4.2. Colorimetric Measurements

The change in color was measured using a MiniScan Model No. EZ MSEZ0693. All samples were measured in a visible region, with an interval of 10 nm using D65 light source and an observed angle of 10 degrees.

2.1.4.3. Fourier Transform Infrared Spectroscopy

FT-IR spectroscopy was used to study the chemical changes which may have occurred after treatment and artificial aging. The FTIR instrument used is Nicolet 380 FT-IR Spectrometer.

2.1.4. 4. Burst testing

Burst test was carried out using a Dynamometer SDL ATLAS, H5K T at the National Institute of Standards (NIS) in Cairo, Egypt.

2.2. Results and Discussion

2.2.1. Visual inspection

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Visually speaking, all tested adhesive showed no apparent change after artificial aging, excluding super glue which showed slight yellowing **(as shown in Figure 4)**. Accordingly, the three tested adhesives (E, EH and EP) were applied on naturally aged glass transparency **(as shown in Figure 5)**. No apparent change was detected in all three samples.

all three samples.

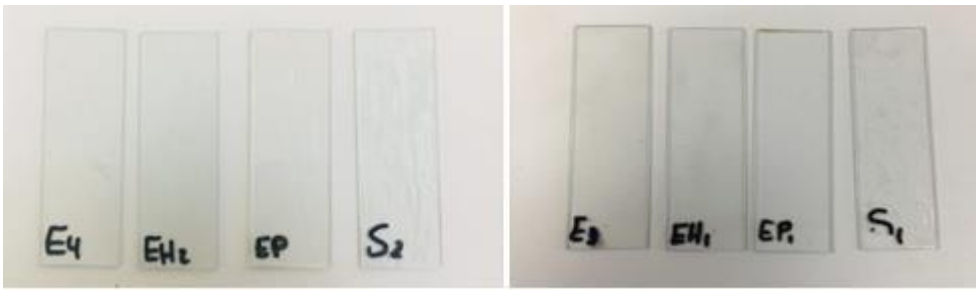

Figure 4. Coated slides before aging (left) and after aging (right).

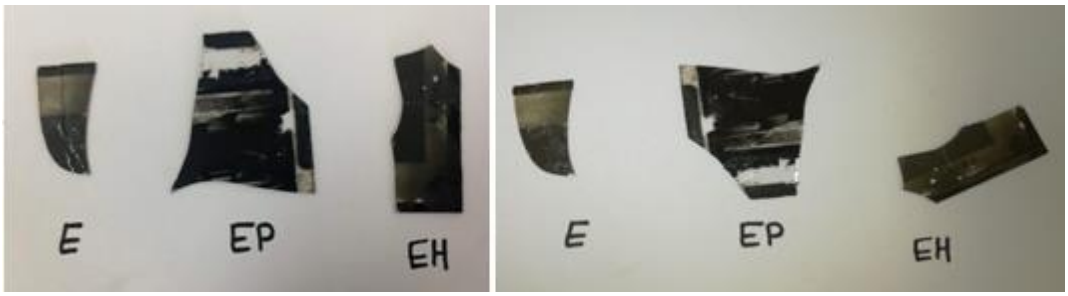

Figure 5. Restored glass transparencies before aging (left) and after aging (right).

2.1.1. Colorimetric measurements

The CIELAB color parameters (L*, a*, b*) were used to express color change, where L* defines lightness and varies from 0 (black) to 100 (white); a* represents the red/green axis, where +a means red and –a means green; b* represents the yellow/blue axis, where +b means yellow and –a means blue. All values of L*, a*, and b* were obtained before treatment and after treatment and artificial aging. Each reading was the average of three measurements. The total color difference ΔE* was also calculated from the

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following formula: ΔE* = (ΔL*2 + Δa*2 + Δb*2)½ **[34]**, **[35]**, **[36]**. According to DIN EN ISO (super ceded by BS EN ISO 4628-1:2004), evaluation of ΔE* is as follows:

- 0 – 1: color difference is not visible.
- 1-3: few people can recognize the difference.
- 3- 5: 66 % of people can recognize the difference.
- >5: everyone can recognize the difference **[34]**, **[37]**, **[38]**.

In literature, ΔE* values of 2-3 are thought to be observable and unacceptable color difference **[39]**; however, it is clearly lower than the threshold limit (ΔE* = 5) required for the maintenance and restoration of historical surfaces **[40]**. Another study also mentions that a ΔE* <<4 is a value normally accepted as limit for the visual impact of surface treatments **[41]**.

Results showed ΔE* values below two for all tested adhesives, with Super glue having the highest color difference (ΔE* value = 1.1) and Epoxy euxit 50 having the lowest color difference (ΔE* value = 0.39), after artificial aging (as shown in Figure 6).

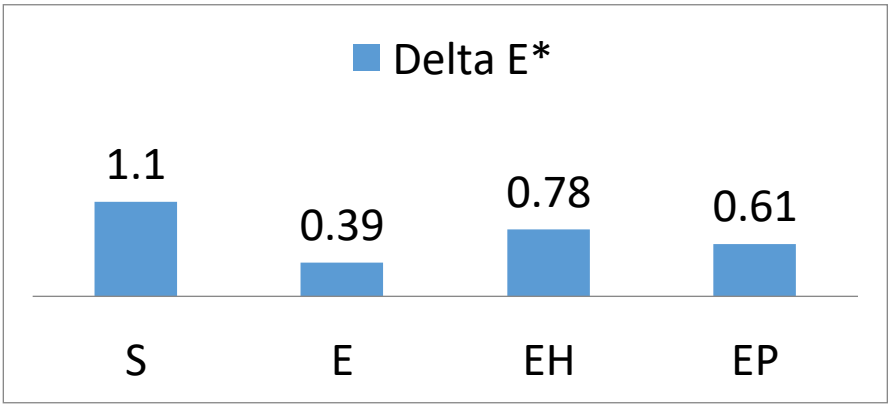

Figure 6. Total color difference (ΔE*) for all tested adhesives on glass slides.

In breaking down the data to L*, a*, b* values, for the Super glue sample, it mainly showed a decrease in L* value. Very slight change in L*

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values were detected for the other adhesives **(as shown in Figure 7)**.

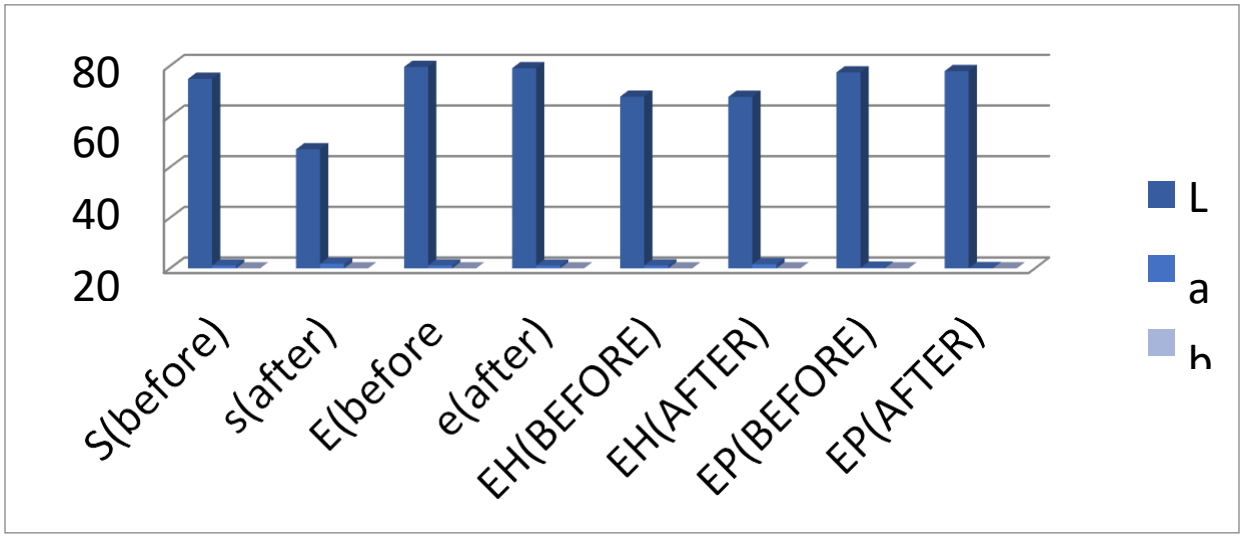

Figure 7. L*, a* and b* values for tested adhesives before and after artificial aging.

For naturally aged samples, both Super glue and Epoxy euxit 50 were excluded fromtesting; the former because it showed the highest color change and the latter because it is time consuming, given the fact that archives or other institutions usually house thousands of glass negatives. The four minute epoxy (EP sample) showed the least color change with a ΔE* value of 4.76 **(as shown in Figure 8)**.

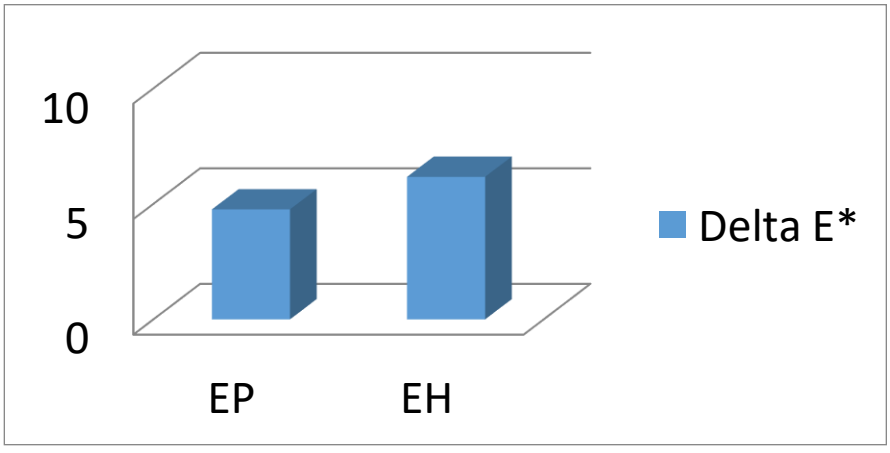

Figure 8. Total color difference (ΔE*) for tested adhesives on naturally aged photographic samples.

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2.1.1. Burst testing

Bursting Strength is a reliable index of the strength and performance of materials **[42]**. For the burst strength test, a different set of samples were prepared. Each sample was prepared by adhering two glass slides with one of the tested adhesives in an overlapping manner. Results showed the highest bursting strength value for the four minute epoxy, with a value of 1143 **(as shown in Figure 9)**.

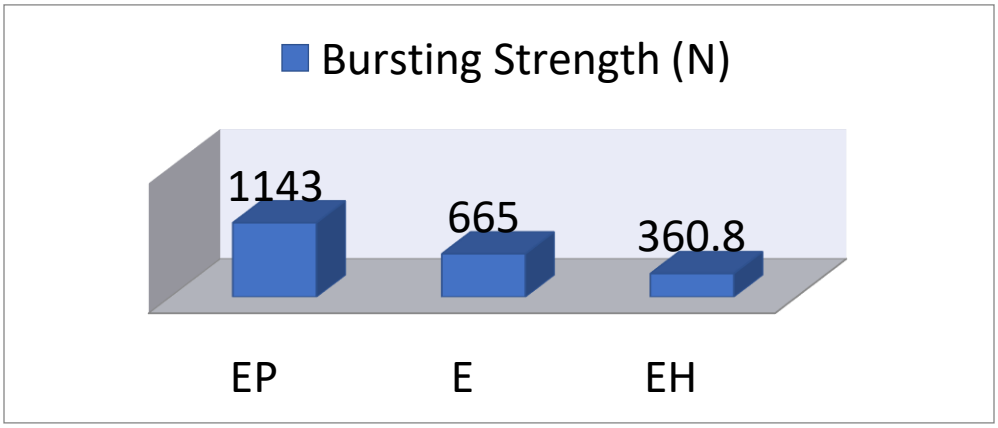

Figure 9. Results for the bursting strength test.

2.2.3. Fourier Transform Infra Red (FT-IR)

Based on previous results, four minute epoxy (EP) was selected for further testing with Fourier transform infrared spectroscopy. This analysis was only applied to the naturally aged photographic sample. Characteristic bands from glass and epoxy have been clearly observed. The broad band at 3448 cm^{-1} shows the stretching vibration of O-H bond. The band at 2931 cm^{-1} indicates the stretching vibration of C-H bond, the band at 1636 cm^{-1} indicates the stretching vibration of C=C bond, and the band at 1047 cm^{-1} indicates the stretching vibration of Si-O bond **[43]**, **[44]**. Results showed

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that there is a slight increase in the O-H band and insignificant changes have been recorded for the other identified bands **(as shown in Figure 10)**.

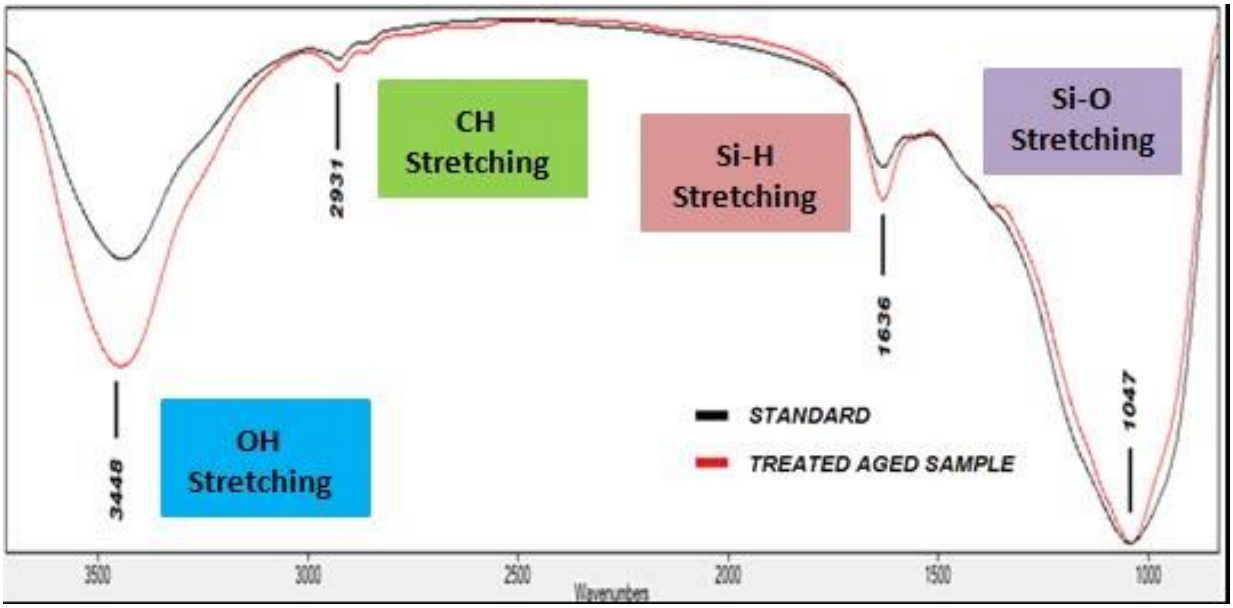

Figure 10. FTIR spectra for glass fragments from naturally aged photographic sample after restoration with four minute epoxy and after artificial aging

2.2. Conclusion

The tested four minute epoxy gave very good results in terms of optical and chemicalproperties. It has the following advantages:

- Ease of application.
- Dries fast.
- Dissolves very well in acetone.
- Inexpensive.

When necessary it can be used to assemble gelatin dry plates. However,

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a sink mat is a more safe option.

2. Conservation of Broken Dry Plate Negatives from Francis Amin's Private Collection

3.2. Collection Description

The collection consists of three portrait black and white negatives, three large fragments anda number of small fragments **(as shown in Figure 11)**. The collection belongs to Francis Amin, photograph collector from Luxor, Egypt.

Figure 11. Dry glass plate negatives from Francis Amin's collection.

3.3. Condition Assessment

The photographic process used was identified as dry gelatin process as the following common characteristics were verified: glass is used as a primary support, the image has a neutral black and white tone, the glass surface has a glossy surface sheen while the image surface ismatt, it has

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high details, and the presence of silver mirroring **[45]**.

Three glass negatives were broken into fragments of various sizes **(as shown in Figure 12)**. The image side of the glass negatives suffers from silver mirroring. Silver mirroring is an image decay form which appears as a bluish metallic sheen giving the shadow areas an iridescent appearance **[46]**. The collection also suffers from surface dust and finger prints due to improper housing and inappropriate handling **(as shown in Figure 13)**. Forms of decay found in the collection are listed in Table 2.

Table 1. Damage forms found in Francis Amin's dry plate negatives.

Location	Damage Form
Image damage	▪ Silver mirroring. ▪ Fading.
Binder damage	▪ Peeling. ▪ Flaking. ▪ Surface dirt.
Support damage	▪ Breakage. ▪ Scratches.

Figure 12. Broken fragments from the Francis Amin's dry plate negative collection.

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Figure 13. Forms of damage found in the dry plate negative collection.

A SUPEREYES PZ01 500X USB Digital Microscope was used to document damage forms such as surface dirt, scratches **(as shown in Figure 14)**, silver mirroring and glass damage **(as shown in Figure 15)**.

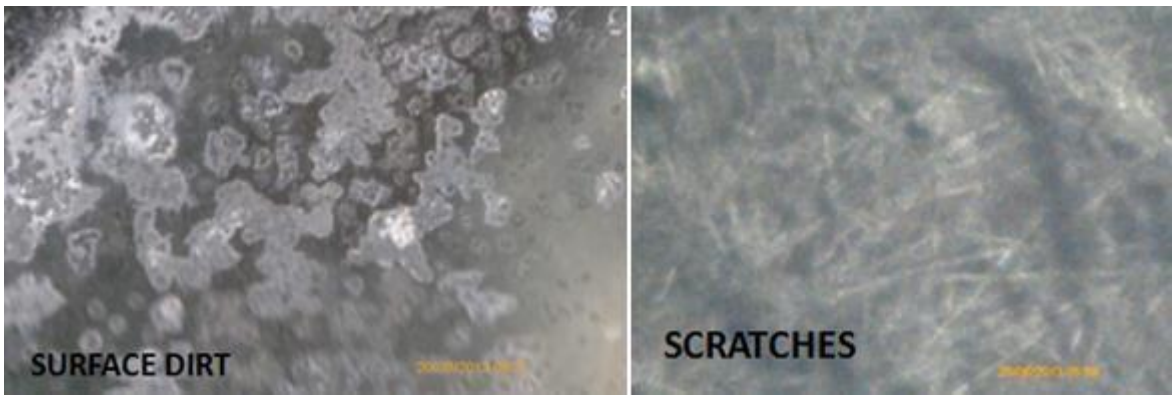

Figure 14. Surface dirt (left) and scratches (right).

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Figure 14. Glass damage (left) and silver mirroring (right).

3.4. Intervention Conservation

Based on the examination and analysis results, we decided that the conservation treatments would involve disinfection, sorting out and initial assembling, mechanical cleaning, assembling using adhesives and housing.

3.4.1. Disinfection

Natural products obtained from plants with biocidal activity represent an alternative and useful source in the control of biodeterioration of documentary heritage, without negative environmental and human impacts. Therefore, clove oil was used since it has antimicrobial antiseptic, and disinfecting action, given by its content in terpenes, aromatic aldehydes, terpenicaldehydes and phenolic compounds, among other components **[47]**.

3.4.2. Sorting Out and Initial Assembly

Fragments were sorted out and initially assembled to decide which fragments belong to which negative. This process produced three broken glass negatives **(as shown in Figure 15)**.

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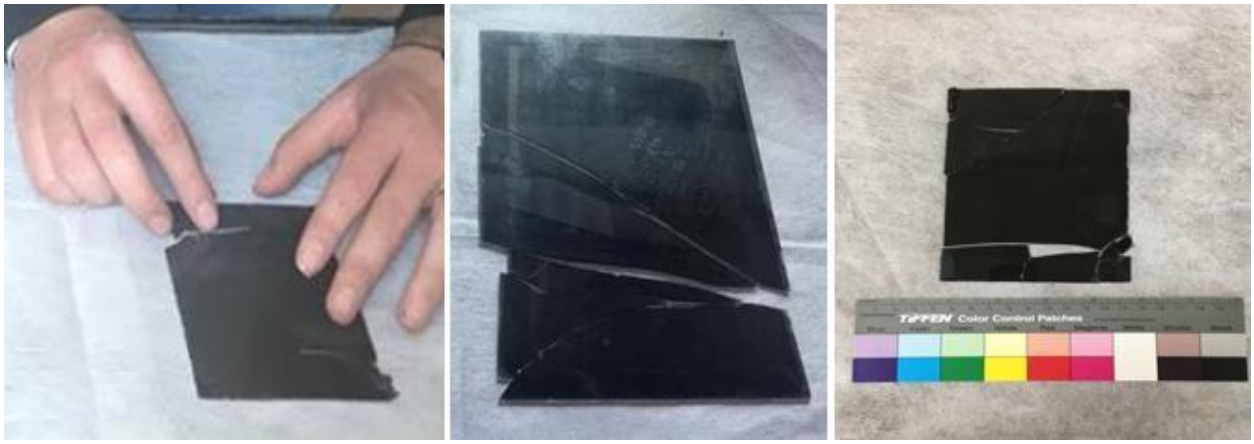

Figure 15. Initial assembly phase.

3.4.3. Surface Cleaning

Dry cleaning was carried out on the recto and verso of the negatives using a very fine brush. This was followed by solvent cleaning using a mixture of 80:20% ethyl alcohol in distilled water. The solution was applied by cotton swabs **(as shown in Figure 16)**. This helped remove surface dirt and reduce the mirroring appearance **(as shown in Figure 17 and Figure 18)**. Magic Rub eraser was also used and gave efficient results in mirroring reduction **(as shown in Figure 19)**.

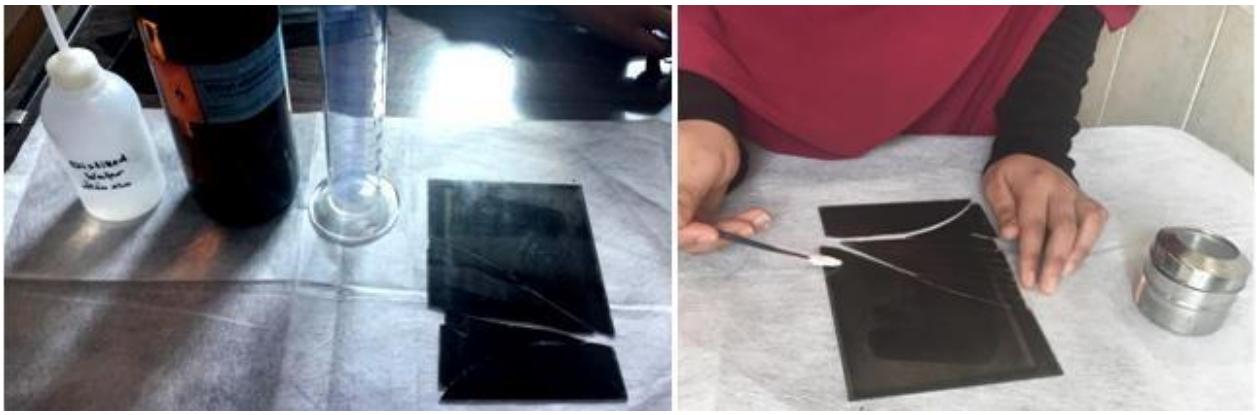

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Figure 16. Surface cleaning using ethyl alcohol and distilled water mixture (80:20%).

Figure 17. Silver mirroring reduction using solvent cleaning.

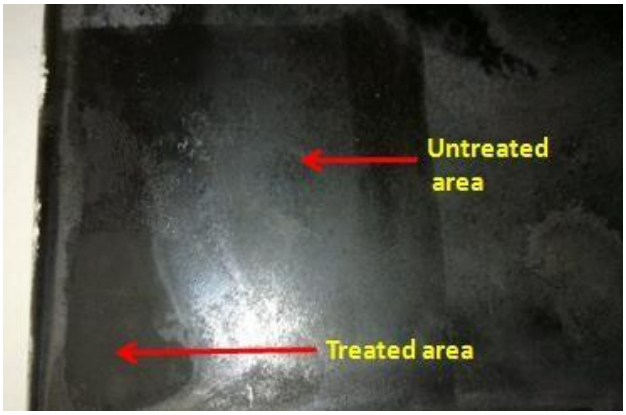

Figure 18. Silver mirroring reduction using solvent cleaning.

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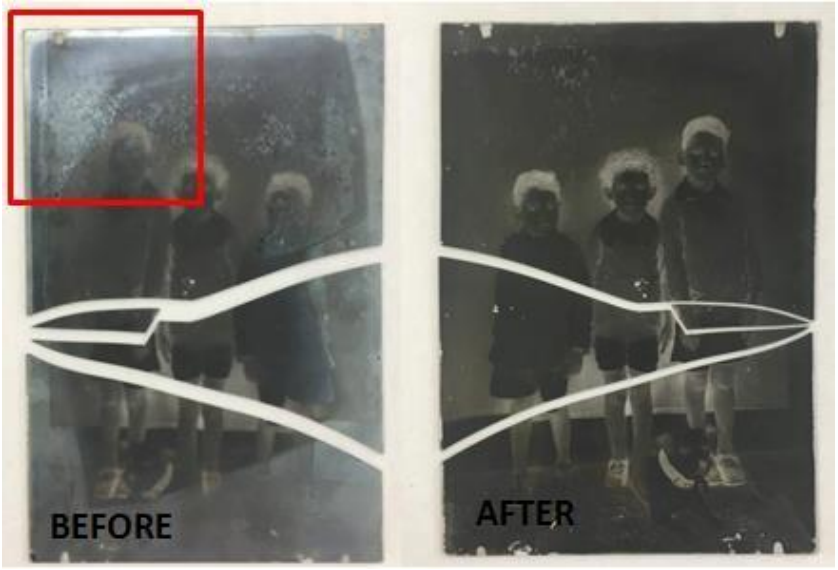

Figure 19. A glass negative before and after cleaning.

3.4.5. Assembly of Broken Fragments

This process was done using four minute epoxy. The broken fragments were first assembledusing self adhesive tape. This was followed by applying the selected epoxy with a needle in the gaps between the fragments **(as shown in Figure 20)**.

Figure 20. Before assembly (left), after initial assembly (center) and after assembly with four minute epoxy (right)

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3.4.6. Housing

Sink mats were designed to house the negatives as well as a storage box from acid-free cardboard paper **(as shown in Figure 21)**.

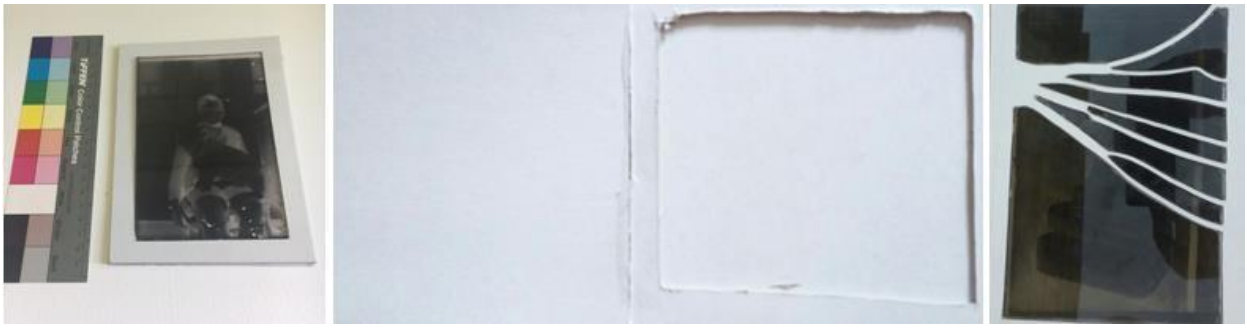

Figure 21. Sink mats for housing glass negatives.

3.5. Conclusion

The preservation of glass negatives is extremely challenging and requires multiple measuresto ensure its survival for future generations including:

- Proper storage conditions.
- Proper handling.
- Adequate understanding of preservation needs.
- Trained staff.

Based on our study we recommend the following:

- Institutes holding glass negatives are strictly advised to include digitization as part of theirpreservation strategy.
- Glass negatives should be housed individually in loosely-fitting, buffered paper sleeves withside seams.

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- Sink mats are advisable for storing broken glass negative.
- Only when necessary certain epoxies can be used for assembling broken fragments only ifcarried out by professional.
- Glass negative should be stored in a controlled environment at a temperature of less than18◦C and 30 % RH.

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