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Treatment and conservation of broken glass photographic negatives

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Abstract:

Archaeology has received the attention of many scientists; Civilization is the fabric of human thought. Photography is the new element of this cultural fabric. Photographs consist of negative images and positive images; it is one of the most important archival materials that abound in Egypt and the world, in which there are thousands or hundreds of thousands of negatives about Egyptian antiquities from the 19th and 20th centuries, which became a record of important records and documents.

The work consists of manufacturing and preparing trial samples of photographic negatives on a glass support, accelerated industrial obsolescence operations were also performed on laboratory-manufactured samples to simulate historical negatives; a set of suggested adhesives was selected for gluing broken negatives and applied to samples of previously manufactured negatives. Two slides of experimental photographic glass negatives were collected; It has been subjected to the conditions of accelerated obsolescence to apply the chosen adhesives, Four materials were selected (Araldite 1092 – Cyanoacrylate - Epoxy Epobond Transparent – Clear Silicon Transparent C 901). The transparent epoxy epobond was proven by comparison in terms of technical specifications and color change as well as resistance to mono-tensioning. The process was applied to a historic B0901 negative from the collection of the Bella Photography Studio.

Keywords: photographic negatives – Glass support – fractures – restoration – adhesive

Introduction

Historic glass mount photographic negatives consist of a glass support and a photographic layer that is very sensitive to deterioration as a result of the binder (gelatin) and silver halides (bromide, iodide and silver nitrate). The glass photographic negative is easy to break and the process of gluing the parts and assembling the broken glass can be called the process of building the piece. In the beginning, natural materials were used to glue the broken parts of the glass traces such as animal glue, gum arabic, natural candles (beeswax) and others[(1)]. However, over time it has been subjected to many issues (brittleness, discoloration, dryness, shrinkage, shrinkage, dustiness, biological deterioration). The association between the sensitivity of the sensitive photographic layer material and the adhesive of the glass holder and its vulnerability to damage in the case of using an improper adhesive mod may cause destruction of negativity.

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Materials and Methods

1- Materials :

- Photographic glass negatives for experimental study consisting of (glass slides cm3*cm3*cm3*3mm - photographic sensitive layer consisting of gelatin, halogenated alkali (potassium iodide + potassium bromide), silver nitrate, gold salt and Ammonia.
- By researching the previous studies of several theses and experimental studies of gluing glass artifacts, which studies have proven their ability to be colorless and resistant to various aging factors and have been used in various fields [(2)]. To various aging factors and have been used in many fields, so they are being tested to know their success in the field of vitreous photographic negatives, namely (cyanoacrylate - epoxy epoxy epoxy). Transparent epoxy epoxy transparent epoxy - Clear Silicone C-901 transparent).
- The process was applied to a historic B0901 negative from the collection of the Bella Photography Studio.[1]

2- Methods- :

2-1- Documentation: -

The oldest photography studios in Egypt were searched to acquire a negative on a glass stand that is actually applied to it and contacted the Bella Studio, which is managed by Mr. Ashraf Bella, a photographer - Fig. 1- , Bella's supervisor still works with the old camera that Bella used in 1890, the German Lehnhoff camera, which is a wooden box mounted on a four-wheel stand- Fig. 2-, The photos and negatives kept by Mr. Ashraf are characterized by the presence of silver nitrate on them as a result of the method of acidification as well as the knowledge and visibility of the storage location - Fig. 3-,. The image has become a clear picture of the most important factors of damage, as well as the manifestations resulting from these factors, as shown in Fig, 4, 5, 6

		
Fig. 1 Bella Studios in the old downtown area	Fig.2: Lehnhoff's old German camera	Fig. 3 shows the warehouse where the photographic glass negatives are stored.

[1] A Cartier Bresson :- " Syntheses deas travaux reccuillis dans la lilterature sur la restoration des photographies en noir et blanc " – Etude bibliographies sur. La restoration des photography's- imprimerie Louis – jean 1981 , p 114 يمكن الاطلاع علية https://www.tcd.ie/French/assets/doc/Counter-Revolution%20Poly%202011.pdf

[2] Hill, G. and J. Keister. 2011. "An unexpected learning opportunity: making the most of a problematic treatment" Topics in Photographic Preservation, Vol. 14. American Institute for Conservation Photographic Materials Group. Washington, D.C.: AIC. 93-102.

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Fig. 4 shows the passivity in the studio during delivery	Fig. 5 shows the appearance and location of handprints resulting from damage during preparation	Fig. 6 shows the phenomenon of peeling and loss of the sensory layer

Fig. 7 shows the documentation of the programs and the placement of the negative damage manifestations (B0901)

2-1-1 Diagnostic process

The first diagnostic steps were the historical photographic negatives on a glass holder number B 0901 from inside the Bella Studio storage room on the roof of the property and the previous memory was preserved inside a carton underneath a stack on top of each other and not separated by anything. A visual inspection was carried out to make a preliminary study of the damage to the historical photographic negative on the glass holder B 0901, the subject of the study, using a magnifying glass to identify cracks, dents, fingerprints, scratches and other damage that cannot be seen with the naked eye.

The historical photographic negative on glass holder number B 0901 was found to be the most significant form of deterioration: -

Dust and dirt: - One of the most important manifestations of damage that can be observed is the presence of blue adhesive tape to mark the image, which may lead to tearing the sensitive photographic layer and thus distort the negative, in addition to the deposition of dust on the photographic layer - Fig. (6)

Cracking and broken: - As a result of improper storage and preservation of the negative made of glass, it is easy to break and shatter due to the shocks to which it was exposed, the glass photographic negative No. B 0901 was separated into 6 parts of different sizes – Fig. No. (6).

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Silver Reversal :-.When silver is oxidised, silver ions are formed, which become highly reactive and able to migrate through the gelatin emulsion - Fig. 5 This chemical change of the silver image is due to exposure to strong oxidising agents, such as peroxide bleach from the poor quality paper box it is stored in and sulphur dioxide from air pollution, in the presence of moisture where silver ions migrate to the gelatin surface and combine with other silver ions to form a layer of visible metallic silver.

Some Parts are Missing: - The historical photographic negative on glass holder No. B 0901 was found to be missing after assembling the broken parts - - Fig. (1)

Scratches: - Negative B 0901 was found to have numerous scratches - Fig. 4 This appearance is caused by improper handling and exposure of the photographic film to a sharp object.

Peeling: - Negative B 0901 was found to have many small pieces of the sensitive layer separating from the glass holder. This appearance is due to the long drying time required for the dense sensitive layer, poor adhesion and poor preparation of the glass holder. Fig. 4

Fingerprints: - Negative Number B 0901 was found to have - Fig. (5) - handprints in alphabetic characters, which is evidence that the fingers that touched the negative were wet with water, or wet with the appearance at the time of presentation.

2-2 Experimental: -

2-2-1 Experimental Samples and Artificial Aging: -

The experimental aspect included the fabrication and preparation of experimental samples of photographic negatives on a glass substrate. The sensitive photographic layer was deposited on a glass substrate at a temperature of 49 °C with a layer thickness of 1. 200 microns in the glass panels: 200 microns in glass Plates-Fig. 8 -, After pouring the solution, we have manufactured samples of photographic negatives that have been exposed to light to give the same effect as light on photographic film - Fig. 9

Fig. 8 shows the ripening process to produce a photographic emulsion

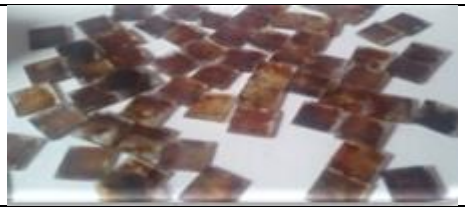

Fig. 9 shows the result of photographic negative samples after exposure to illumination

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2-2-2 Results and discussion: -

2-2-2-1 Operational properties of selected epoxies:

The selected materials were compared through their respective technical specifications at 25°C according to the producing company through the following table1

	Araldite 1092	CA (N 2 C 5 H 7(	Epoxy Epobond	Silicone C-901
Color	Transparent colorless	Transparent colorless	Translucent to yellowish	Transparent colorless
The mixing ratio of compounds A B by weight	2 resin: 1 hardener	-	1 resin: 1 hardener	-
Percentage of solids	100%	100%	100%	100%
Operating Period	25:40 min	10 min	30-60 min	25-50min
Final Dry Time	18-24 hr.	3 hr.	1 hr.	18-24 hr.
Retrievability and Removal	Acetone	Acetone	Acetone	Acetone, alcohol or benzene
Prepare samples by gluing two films with adhesive				

Table 1 shows the standard specifications of the selected adhesive

2-2-2-2 The effect of different aging processes on the selected epoxies:

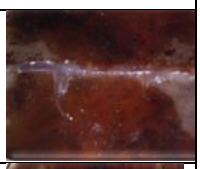
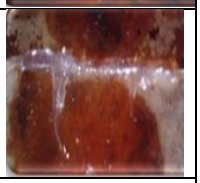
	Araldite 1092	CA (N 2 C 5 H 7(	Epoxy Epobond	Silicone C-901
Before obsole scence				
After obsole scence				
Notes	Yellowing that may reach dark areas at the splice line	It hasn't changed color	The blackening and darkening evident at the gluing line	No changes have occurred

Table 2 shows the evaluation by appearance before and after treatment (photography)

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2-2-2-3 Single-Column Materials Tensile Tester

When all subjects were evaluated, the following results were given- :

Material	Samples N.,	Tensile Strength (Mpa)
Araldite 1092	A1	31
	A2	30
	A3	30.5
Epobond Epoxy Clear	B1	44.5
	B2	45
	B3	43.8
Material	Samples N.,	Tensile Strength (Mpa)
Ca (N2c5h7(	C1	33.5
	C2	32.1
	C3	33.3
Clear Silicone C-901	D1	15.9
	D2	16.4
	D3	15.9

Table (3) shows the technical specifications of transparent epoxy epoxy epibond

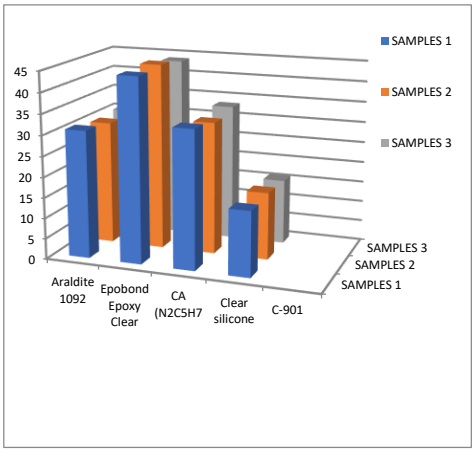

Fig. 22 shows the comparison of tensile values for adhesive materials

It is clear from the test that Epobond Epoxy Clear epoxy adhesive has the highest average tensile strength, followed by the average tensile strength CA cyanoacrylate (N 2 C 5 H 7), then comes Araldite 1092, and in the last rank of the best and weakest in terms of tensile strength is C - silicone adhesive. 901 Clear silicone- Fig. 22

From here, after comparing all the points in terms of operating time, final drying time, density, chemical resistance, tensile strength, retrievability, color and surface change of the adhesive after curing, we can say that the epoxy epoxy transparent epoxy adhesive gave satisfactory results in all the comparisons. We can say that transparent epoxy epoxy adhesive gave satisfactory results in all comparisons, followed by Araldite 1092, but it can be said that each glass photographic negative has its own case.

2-3: Examining and analyzing the components of (negative B0901)

2-3-1 Transmission Electron Microscope (TEM):-

Assayed in the laboratories of the Graduate School of Nanotechnology at Cairo University, through a high-resolution transmission electron microscope (HRTEM, JEOL TEM-2100, Japan) with a voltage of 250 kV and 20X magnification. where he showed the damage to silver halide granules and the flattening of silver, as well as the damage and wrinkling of gelatin on the glass crystals - Fig. 23,24,25

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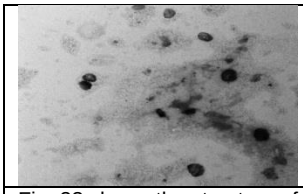

Fig. 23 shows the structure of the TEM grains at 100NM magnification

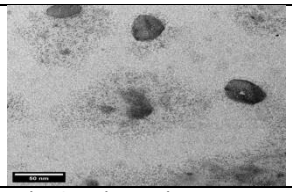

Fig. 24 shows the structure of the TEM-photographed granules at 50NM magnification

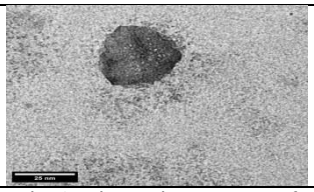

Fig. 25 shows the structure of the TEM-photographed grains at 25NM magnification

2-3-2 Infra-Red (IR) Examination:-

The sample was examined and compared with several standard samples of varnish, gelatin, and gelatin to find that the fluorescence pattern of the historical sample of the photographic negative corresponded to a standard sample of gelatin to confirm the quality of the negative as a gelatinous glass photographic negative. Fig. 26

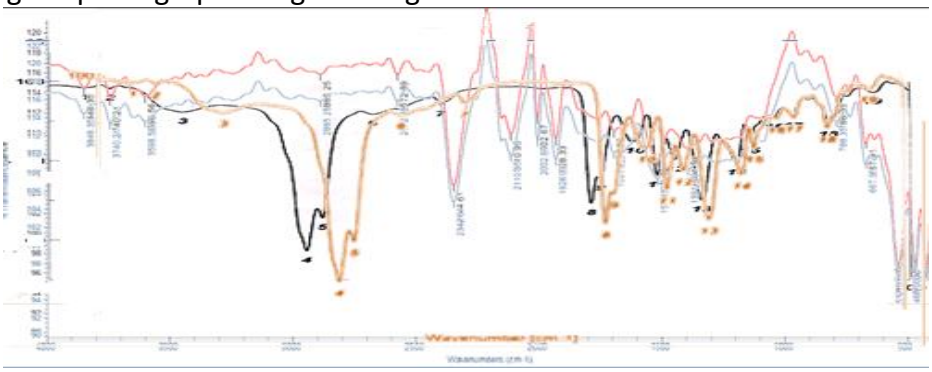

Fig. (26) shows the fluorescence patterns of the negative sample in red, the standard gelatin sample in blue, the varnish sample in brown, and the truss sample in black

2-3-3 SEM with EDX inspection and analysis: -

The first sample was tested in the laboratories of the Graduate School of Nanotechnology at Cairo University, and the results of the test and analysis of the first sample are shown below.

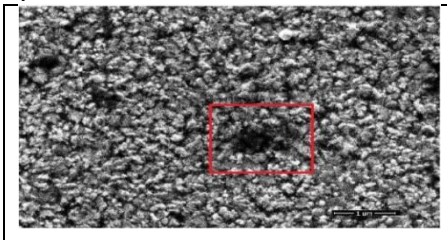

Fig. 27 shows the position of a decomposition pattern of region A with a missing photographic layer at 30,000 X magnification

Element	Wt%	At%
Ag	11.76	**15.04**
Si	71.88	**44.16**
Ca	12.25	**35.48**
Fe	3.01	**3.72**
O	1.1	**1.6**
Total	100	**100**

Table 4 shows the EDX elemental ratios for point A

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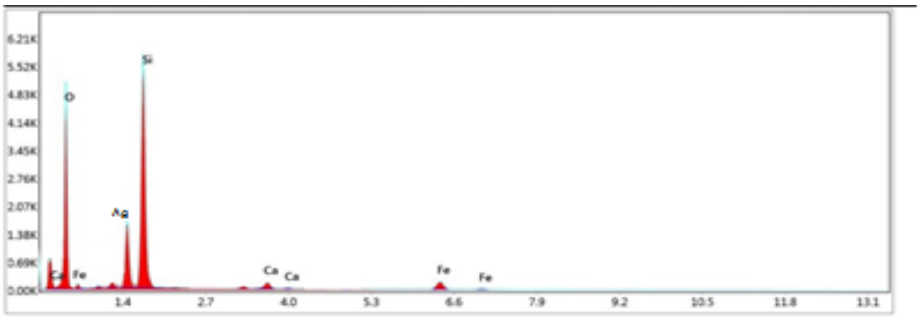

Fig. 28: An analysis pattern of the missing photographic layer in region A at 30,000 X magnification with EDX.

As can be seen in the previous microscopic image, parts of the negative-sensitive photographic layer have fallen as a result of various damage factors, which This is evident in the results of the EDX analysis- Fig. 27, where in the analysis of zone A we observe a high silicon content of up to 71. 88% and the presence of calcium elements 12.25%, which is one of the main components of glass, while the percentage of silver was only 11.76%, although it is the main component of the photographic layer, which is evidence of the fall of flakes of the sensitive photographic layer to historical negativity- Table.4 and Figure 28.

Analyzing another point using EDX

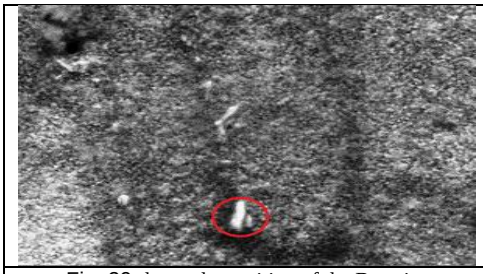

Fig. 29 shows the position of the B-region decomposition pattern for a portion of the photographic layer at 60000 X magnification

Element	Wt%	At%
N	1.25	**3.06**
Ag	79.14	**74.36**
Si	2.05	**2.57**
C	1.10	**1.37**
Ca	0.40	**1.24**
Na	1.04	**3.72**
O	15.02	**16.30**
Total	100	**100**

Table 5 shows the EDX elemental ratios for point B

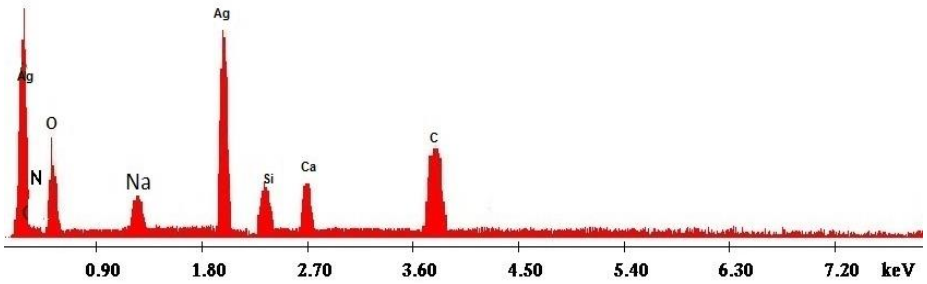

Fig.30 Analysis pattern of the B-zone of the photographic layer at 60,000 X magnification with the EDX system

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As can be seen in the previous microscopic image, the formation of fine silver particles away from their original positions in the sample and the presence of a split in silver ions is evident in the EDX analysis results- Fig. 29, where in the analysis of zone B we observe a high silver content of 79%. 14% with the formation of various silver salts, as well as the presence of carbon, which is the transformation of gelatin as a result of exposure to high temperature, at 1. 10 %, as well as the appearance of silicon, which indicates the presence of cleavage in the photographic layer, the appearance of glass with photographic negativity but little cleavage, and the presence of sodium carbonate due to the formation of salts due to an oscillation in relative humidity Table.5 and Figure 30 .

2-4 Application: -

2-4-1 Cleaning Processes

The mechanical cleaning process started with soft brushes to get rid of the dust and dirt on the sensitive surface- Fig. 31, which was holding some paper labels- Fig. 42, and was cleaned with a water and alcohol solution using cotton wool and removed with fine scalpels with blunt blades- Fig. 34, The layers were difficult to remove and were handled with patience, great care and under a magnifying glass so as not to scratch the delicate surface- Fig. 33, Chemical cleaning was then resorted to in order to get rid of the residual dirt and deposits that cause negative opacity. Care was taken not to exaggerate and overuse chemical solutions and solvents. Therefore, in most cases, cleaning with a solution of iodine in alcohol was limited to 5%, depending on the location and size of the soiling, and the immersion process was carried out in a glass basin for no more than 5 minutes. Putting the piece on top of a net with slow stirring did not take a long time during the process and in this case it was found that the immersion process is better than compresses due to the rapid spread of the solution and the loosening of dirt and deposits- Fig. 35. Without contacting the sensitive surface or exposing it to any mechanical friction forces with the use of chemical solutions, it was considered to dry the parts immediately after cleaning using indirect air- Fig. 36.

Fig.31 shows how to remove dirt with a soft mattress

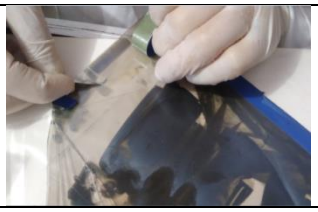

Fig. 32 shows the removal of adhesive and cellulose

Fig. 33 shows the removal of a candle from the other side

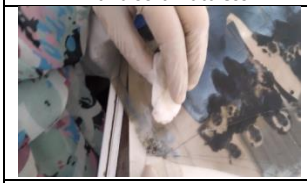

Fig. 34 shows the removal of blue solitape with acetone

Fig. 35 shows the cleaning of the negative by the immersion method

Fig. 36 shows the process of drying the passive after the cleaning process

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2-4-2 Joining and Adehsion Processes

After the initial assembly process was completed- Fig. 37, the final assembly was carried out using Epoxy Epo Bend adhesive, which proved to be successful in the experimental aspect and was applied to the fracture line using a thin pin from the glass carrier face and in appropriate quantities, taking into account the placement of free paper. It was applied to the fracture line using a thin pin from the glass carrier face and in appropriate quantities, taking into account the placement of acid-free paper on the sensitive surface for photographic negativity and the lack of penetration of the adhesive to the sensitive surface Fig. 38, After a week, the celluloid strips were removed and the fracture line itself was cleaned of excess adhesive residue, mechanically using a scalpel. taking care not to scratch the surface, followed by a quick chemical cleaning process using acetone to make sure there are no adhesive residues on the surface Fig. 39.

- Fig. 37 shows the fractional parts of the negative

- Fig. 38 shows the assembly and gluing process

- Fig. 39 The final shape after assembly

2-4-3 Completion of Missing Parts Processes

Then, a chassis was made from the wood cells after encapsulating them with cellulose, then the insulation process was carried out using Vaseline in preparation for the application of layers of complementary material (transparent epoxy epobond, which has been experimentally proven successful and is viscous and not liquid). The application should be done from the main negative surface of the sensitive photographic surface, with extreme care to ensure that the filling level is 1 to 2 mm below the surface. After the drying and hardening process is complete, the chassis and chains are removed and cleaned with acetone Fig. 40.

2-4-4 Labelling process

The negative numbering was done from the side of the glass support from
the bottom at the far left side using Japanese paper saturated with Paraloid B72, then added to the surface using brushes, and then the number was written Fig. 41.

2-4-5 Consolidation process

The strengthening and insulation process was carried out using silver nitrate nanocomposite soft brushes Fig. 42

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- Fig. 40 shows the shape of the chassis used to complete	- Fig. 41 shows the negative numbering	- Fig. 42 during the strengthening process with the use of a mattress

2-4-6 Packaging Processes

The passive storage process has two stages: -

The first stage was done after receiving the negative number B0901 from the studio and keeping it, by separating each part separately to preserve the fracture edges to prevent double damage from corrosion of the letters and thus when assembling, a difference occurs in the fingerprint of the fracture edges and the stages of packaging appear through the tools used as well as knowing the dimensions of the parts to divide the preservation box - Fig. 43. Ethafome is divided by the same shape as the edges inside the box and reinforced well to prevent movement, vibration and friction - Fig. 45. The negative is placed in a temporary preservation box until the restoration begins - Fig. 46.

The second stage took place after the completion of the restoration work and full documentation of the negative. Both stages of packaging were done using acid-free cardboard (free acide board) and a reinforcing method for the negative to preserve it during the storage process to prevent shaking or breakage. This method was achieved as it is suitable for display and storage at the same time as the dimensions of the negative were raised after the completion of its restoration - Fig. 47- 3 cm was added on each side and a thickness of a name was made to prepare the preservation box, then acid-free paper was placed and then the Ethafome material was divided to be around and around the negative space in all directions to support it and prevent shaking and movement Fig. 48 A silica gel moisture absorbent was applied and the negative was covered with acid-free transparent paper . Then all the data was written with the registration form - Fig.49 . And a photo showing the negative before restoration and a photo of the negative after restoration - Fig. 50. And the negative was converted to a positive photograph to know the shape of the final image and become a complete record of the negative to prevent its frequent circulation in order to preserve it - Fig. 51.

		
Fig. 43 shows a negative split to illustrate the dimensions of	Fig. 44 shows the shape of the preservation box after Ethafome has been correctly	Fig. 45 shows the final shape of the broken negative and its

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the Ethafome material for each separate part	positioned to separate the broken parts	parts are securely separated to prevent further damage
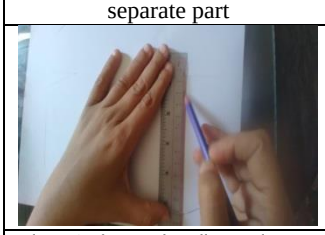		
Fig. 46 shows the dimensions of the packaging box	Fig. 47 shows how to apply acid-free foam.	Fig. 48 shows after placing the negative and covering it with acid-free Japanese paper
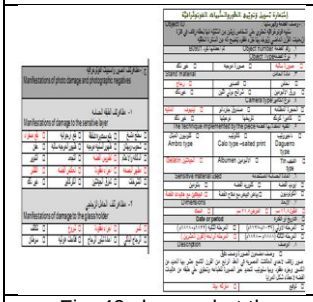		
Fig. 49 shows what the attached registration form looks like before it is written down	Fig. 50 shows what the negative looks like after restoration	Fig. 51 shows the positive and negative image after restoration

Results and discussion: -

As a result of the process of moisture, heat and chemical aging of laboratory samples, we find that the transmittance value of the manufactured samples before aging was (56%), while after heat and moisture aging it reached (10%), where the lower the transmittance value of the negative, the less the opacity for him and vice versa, and after aging with HCL acid, it became (61%) where hydrochloric acid caused an increase in the transmittance of the samples, this is due to the fact that it causes the cleaning of the surface layer of the glass negative and the next layer appears more transmissive to light and it is noticed after alkaline aging using NaOH, the transmittance became (40%) and this is due to the fact that alkaline substances cause opacity and this is the reason for the decrease in transmittance.

- Transparent epoxy adhesive has been given many advantages, including excellent adhesive strength, and it has resisted the aging conditions without changing color or even its bond strength, in addition to its chemical stability, so it did not occur any kind of reaction with the sensitive layer as well as with the glass, and therefore it is recommended for use in gluing historical photographic negatives executed on a glass support.

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- Double and single molding should not be restricted in the process of restoring missing parts of the glass, but creative solutions that fit the historical negativity and the location of the missing parts should be considered.

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The Recommendation

1. The need to pay attention to increasing publicity in international forums, museums, major libraries and universities about the importance of preserving historical photographic negatives, especially glass, because of the documentation they carry in addition to the weakness and complexity of the material they are made of.
2. Photographic negatives executed on glass mounts kept in stores must be protected by creating suitable preservation conditions of temperature and relative humidity, which should not exceed 42%, and the atmosphere of the stores should be characterized by purity and continuous ventilation with regular maintenance operations for these collections.

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