

STRUCTURAL REINFORCEMENT OF FRAGILE ARCHAEOLOGICAL GLASS BY LOSS COMPENSATION USING ACRYLIC SHEETS.

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Abstract

The current paper discusses a new technique for restoring glass artifacts with gaps and loss of fragility. Acrylic slides of Paraloid B-44 are the best in terms of use because the degree of glass transition (T_g) is about 60, which suits the temperature in Egypt. The slides were made with 40% Paraloid B-44 w/v in acetone and used to fill the lost parts for structural reinforcement. The paper also aims to study the glass vessel that was discovered in the excavations of the American Research Centre in Fustat in 1966 and kept in the stores of the Museum of Islamic Art in Cairo. The glass vessel was examined by optical digital microscope (ODM) and ultraviolet imaging. After that, it was analysed by the scanning electron microscope (SEM) with an EDX unit and Fourier transformed infrared spectroscopy (FT-IR). Then, the conservation processes were performed. The conservation of the glass vessel included disassembling the previous restoration, cleaning, reassembling, filling and supporting the lost parts, and consolidation. The study concluded important results about the composition of the glass, deterioration factors, and identifying the materials of the previous restoration that were found inappropriate and required removal and conservation. It also clarified the method and importance of using acrylic (paraloid B-44) slides in strengthening the fragility and thinness of glass artifacts.

Keywords: Paraloid B-44; Gap fills; Compensation; Archaeological glass; Reinforcement.

Introduction

Gap filling of glass artifacts is a major challenge in the field of conservation, especially for constituent glass that has degraded and is structurally weakened, which may be extremely thin. Glass artifacts discovered in excavation locations often suffer from smashing and breaking, causing the loss of some parts [1-3]. Filling the gaps aims to give the vessels structural stability in order to keep them in a reconstructed state, which is intended to improve their aesthetic and informative value [4-7].

Filling materials were selected based on several criteria, e.g., transparency, color stability, viscosity, and reversibility [8-14]. They were mainly synthetic resins. Epoxy and polyester resins have been widely used over the past years for glass fills, and their characteristics are well documented. However, it has been observed that they become yellow and degrade over time. Furthermore, their long-term chemical instability causes severe physical stress on glass artifacts because of their high tensile strength. Thus, they are not suitable for

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weak and fragile glass artifacts [15-21]. Moreover, they are either directly cast or moulded for indirect casting, requiring extensive interaction with the object that can cause further damage.

Acrylic resins have proven successful as adhesives, consolidates, and coatings for glass artifacts because of their reversibility, mechanical resistance, and relative stability [22–25]. They are known as paraloid and acryloid. Several types are available for use, including Paraloid B-44, 48, 66, 72 and 82. However, the most common in restoration and conservation are Paraloid B-72 and Paraloid B-44 [26, 27].

For some glasses, particularly fragile archaeological glasses, a different method using cast Paraloid™ B-72 sheets or films was devised to accomplish loss compensation. Several advantages of this technique have been observed, such as reversibility, long-term chemical stability, and easy future removal and preparation away from the glass object while preventing excessive handling, making it particularly helpful for extremely fragile glass [28, 29]. Gap filling with B-72 enables manipulation of the appearance to fit the glass, such as coloration, texture addition, and retouching [30].

Selecting the resin depends on the environmental conditions of the glass artifact, especially the temperature [31]. Some studies reported that Paraloid B-72 gave poor results in high temperatures and hot climates [32] because of the low degree of glass transition. Moreover, the company's manufacturer illustrated that the glass transition degree of Paraloid B-72 is 40, which is close to the temperature in summer in the Middle East. Determining the glass transition degree of the polymer is very important. It is described as the degree to which thermal energy is less than the forces linking molecules [25]. At a lower temperature, the least molecular bonding can be achieved. While the polymer is fragile and hard below the transition degree (T_g), it is soft and soluble above T_g because T_g determines the properties of the resin in terms of hardness and flexibility.

The T_g of paraloid B-44 is high, about 60 [33]. That is, it withstands high temperatures and does not soften easily. Therefore, it was used in the conservation of the glass case study because the temperature can be 40°C in Egypt in the summer. Paraloid B-44 (40% w/v in acetone) was utilized in making slides to fill the gaps in the glass vessel case study. The paper also aims to register, examine, and analyse the glass vessel. The glass vessel was subject to corrosion and previous harmful restoration. Therefore, the authors decided to dismantle the previous restoration and carry out a conservation plan, starting with cleaning, reassembly, filling the gaps, and consolidation.

Experimental part

Materials

Preparation of paraloid B-44

Paraloid B-44 is a copolymer of methyl methacrylate and ethyl acrylate obtained from CTS. It was used (40% w/v in acetone) to form slides to fill gaps. Moreover, it was used as a solution (30% w/v in acetone) in the assembly of glass fragments. It was added between the lines of shards with a pin tip.

Description of glass vessel

The glass vessel under study was discovered in the excavations of the American Research Centre in Fustat in 1966. It has been kept in storage at the Museum of Islamic Art in Cairo under the identification number 24290. It dates to the early Islamic era. It has suffered weakness because glass is fragile and subject to corrosion and rust, iridescence, pitting, and surface calcification, and previous restoration has missing parts (Fig. 1).

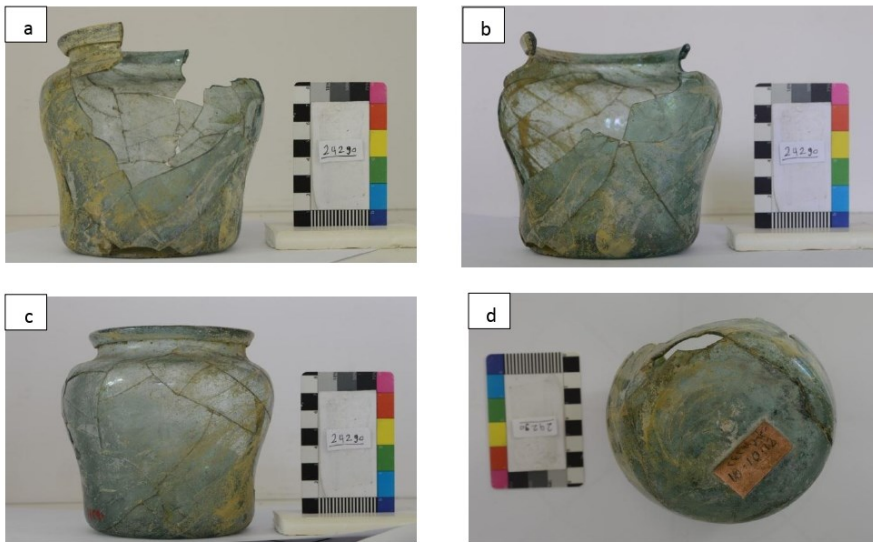


Fig. 1. Photographs show glass vessel from different sides and its condition: a and b - the front side, c - the reverse side, d - the base of vessel.

AutoCad documentation

Its condition was documented using AutoCAD to show the deterioration aspects and create a deterioration map (Fig. 2). The rim of the vessel is wide, with a thick, rounded lip and subtly curved pyriform body, a bigger upper part, and a narrow circular base. The vessel measures 11.5cm by 9cm. It is broken into different parts, some of which are assembled, but large parts are lost. At least 35% of the original material was lost on our vessel. As a result, reconstructed archaeological glass vessels will almost always necessitate structural support in order to be preserved. The circumstances were made worse due to the glass body's natural fragility and thinness. At their thinnest points, the glass measured 0.2 to 0.35mm.

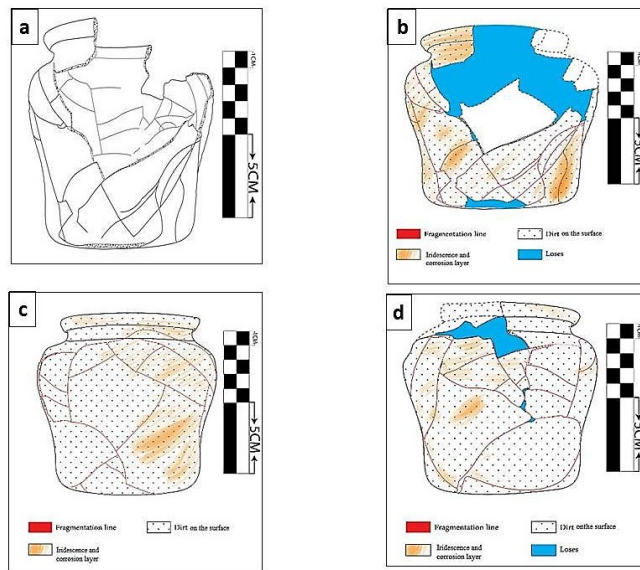


Fig. 2. AutoCAD documentation of the glass vessel from different sides: a - front side, b, c and d - illustrate the deterioration aspects map (losses, fragmentation line, dirt layers, iridescence and corrosion layer)

Glass samples used in study

The samples used included all deterioration products on the surface of the glass vessel (previous restoration materials, i.e., materials of assembly and coating), and the corrosion layer.

Methods*Visual examination*

Visual examination is the first stage of examination to identify the deterioration, damage, and corrosion forms of the glass vessel using different lenses.

Imaging with ultra-violet light

It is used to illustrate the deterioration aspects by studying the surface and different deterioration manifestations, including cracks, fissures, and previous restoration. Ultraviolet photography was done with a 35mm SLR camera with a lens Ft4 capable of receiving light between 11 and 511nm and plain colorless film (3200 ASA). This procedure was carried out in the Restoration Laboratory of the Museum of Islamic Art in Cairo.

Examination with a portable light microscope

A Leuchtturm USB digital microscope (China) with a 20–500X zoom and 8 LED lights with measurement software was used to examine the glass surface features, especially corroded and prone to rust, to illustrate the deterioration as well as the form, color, and rate of corrosion.

Examination and analysis using a scanning electron microscope with an EDX unit

The form, texture, and modifications in the corrosion layer, including new crystallisations, of the glass surface are identified using a Scanning Electron Microscope (SEM) with an EDX unit. Additionally, it aids in identifying the components of the glass surface. Using an EDX unit (Energy Dispersive X-ray Analyzers) linked to a SEM Model Quanta 250 FEG (Field Emission Gun), with an accelerating voltage of 30kV, FEI Company, Netherlands. This method was performed in the SEM lab of the Housing and Building National Research Center in Cairo, Egypt.

Fourier transformed infrared spectroscopy (FT-IR)

It is used to characterise the functional categories of organic and inorganic materials. In the present study, it was used to identify the assembly and coating materials in the previous restoration. In the laboratory restoration of the Museum of Islamic Art, the instrument is a Bruker ATR-Platinum 64 Scan with serial number 12382310.

Results and Discussion*Visual examination*

Visual examination revealed many deterioration manifestations in the vessel, such as iridescence layers, corrosion, and expulsion in the assembly locations of the previous conservation processes, separation of shards, yellowing, irregular scratches, as well as great loss of the rim, body, and base (Fig. 3).



Fig. 3. Deterioration manifestations of the glass vessel (a) Fracture and loss in the body (b) Corrosion on the surface (c) Yellowing of the glass surface and the presence of expulsion between the glass fractures.

Ultraviolet imaging

Ultraviolet imaging showed the locations of assembly in the previous restoration. It illustrated uneven shards in the assembly. Some pieces were outside the correct locations. The color of the corrosion glass in these images was a light violet, which helped determine the corrosion locations accurately. Moreover, the piece number is written in red at the base (Fig. 4).



Fig. 4. Ultraviolet images of glass vessel show the areas of corrosion layers and assembly places between the glass pieces

Optical digital microscope

Microscopic examination revealed glass surface deterioration such as dust, corrosion layers, weathered crust (Fig.5a), iridescence (Fig.5b), flaking, pits, and air bubbles (Fig.5c, d, e). These manifestations demonstrate the fragility and weakness of the glass. In addition, yellowing that occurred in some areas of the glass surface may be due to the discoloration of coating materials used in previous restorations (Fig.5f).

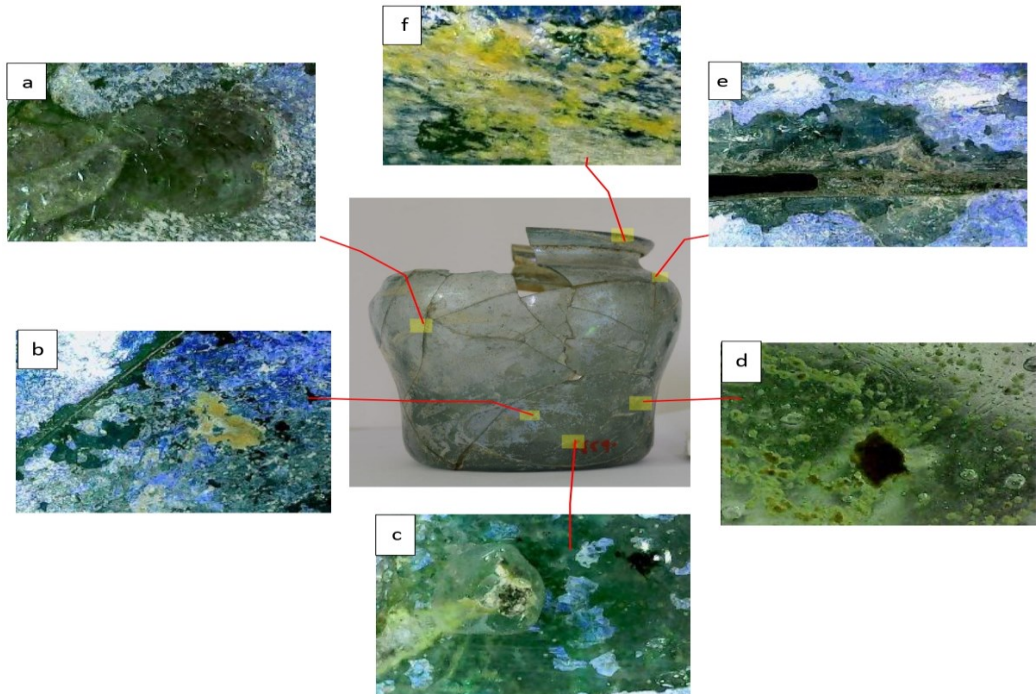


Fig. 5. Optical microscope images of the deteriorated surface of the glass vessel: (a) Corrosion of the glass and formation of weathered crust; (b) Appearance of a blue iridescent layer on the surface; (c) Formation of black pits; (d) Bubbles on the glass surface; (e) Cracks in the corrosion layer; (F) Yellowing of the glass surface

Examination and analysis using a scanning electron microscope with an EDX unit

A glass surface sample (Figs. 6a and b) and a corroded layer sample (Figs. 6c and d) were examined. SEM examination of the glass surface sample showed pits, cracks, air bubbles, and fissures, indicating fragile glass. Furthermore, SEM examination of the corrosion sample showed crystals, crusts, cracks, fissures, and a heterogeneous distribution of corrosion on the surface.

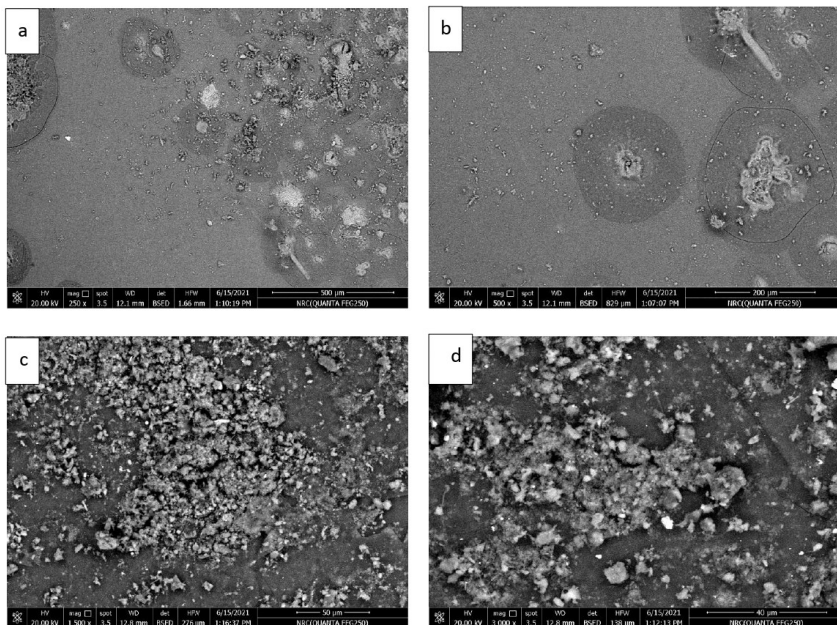


Fig. 6. SEM micrographs (a and b) show the glass surface of the vessel; The formation of pitting, air bubbles, and cracks ; and (c and d) show the corrosion layer, indicating crystallisation phases and dissolution voids of the corroded surface, which loses its glassy nature

The results of the EDX analysis for the glass surface and corrosion layer are presented in Table 1. The analysis of glass sample reveals that the glass primarily comprises silica (52.96%), sodium oxide (4.31%), and calcium oxide (6.96%). It was observed that the glass is sodium-calcium glass, which has been dominant for more than 3000 years [34–36]. The percentage of silica in the glass sample was less than typical. Furthermore, sodium was less than the natural ratio because of the migration of a large amount of alkali outside the glass [37, 38], demonstrating the weakness and fragility of the glass vessel. Furthermore, the high presence of carbon dioxide is observed, attributed to the utilization of a consolidating or coating material during the previous restoration processes.

Table 1. EDX data with chemical composition for glass surface and corrosion layer

Oxides	Glass sample	Corrosion layer
CO ₂	29.69	16.82
Na ₂ O	4.31	2.73
Al ₂ O ₃	3.00	7.80
SiO ₂	52.96	58.72
CaO	6.96	4.60
MnO	0.28	2.44
Fe ₂ O ₃	1.02	3.88
K ₂ O	1.78	1.56
Cl ₂ O	-----	1.45

Iron oxide (1.02% Fe_2O_3) in the glass sample is responsible for the light green and yellowish green colors of the glass vessel. Furthermore, manganese oxide (0.28% MnO) is most likely present in glass as an intentionally added decolorizing agent. It is an oxidising agent that transfers (Fe) to the oxidised state by giving a yellow or light green color to glass [39].

The corrosion layer exhibits a notable presence of silica (58.72%), indicating its migration and subsequent deposition on the glass surface. Additionally, chlorine (1.45%) was observed, likely attributed to salt accumulation resulting from burial and subsequent deposition on the corrosion layer [40]. Chemically, the glass vessel has undergone complete decomposition due to the relentless impact of severe deterioration factors, particularly prolonged exposure to water while buried in moist soil and inadequate storage conditions within the museum. A discernible change in composition is noticeable between the corrosion layer and the underlying interior glass surface. Specifically, there is a decrease in sodium and calcium content, accompanied by an increase in silica content.

Fourier transformed infrared spectroscopy (FT-IR)

A sample of the adhesive material used to assemble the shards and a sample of the material used to insulate the glass in the previous restoration process were analysed using infrared spectrometry. It was determined that both samples consisted of polyvinyl acetate. A sample was taken from the standard polyvinyl acetate and analyzed by ATR. The two results were compared and were identical (Fig. 7).

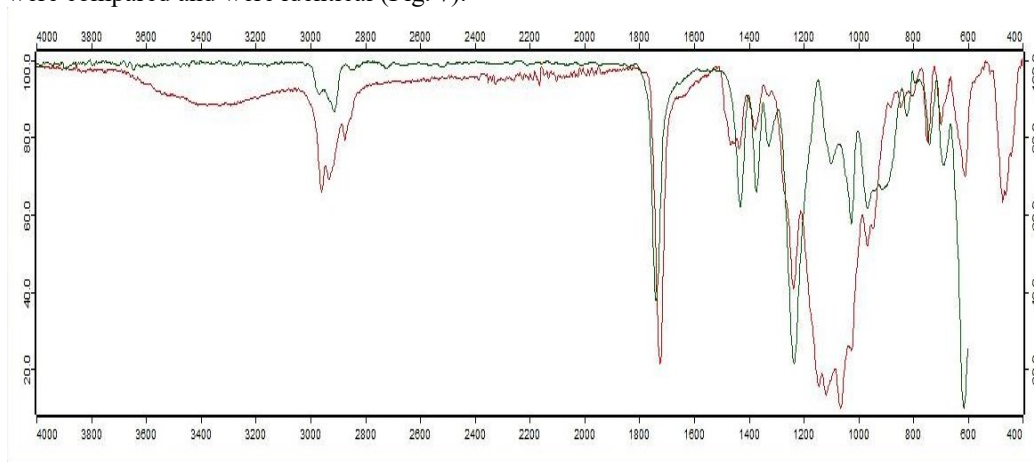


Fig. 7. Illustrates the infrared (IR) patterns of the adhesive and coating materials used in previous restoration processes and the standard polyvinyl acetate (green curve)

Methods of treatment

Disassembling the previous restoration

The glass vessel suffers from solid and cohesive residues because of the wrong application of the material in assembly and insulation, as well as the presence of greasy stains, yellowing of the material, inner and outer dirt, thin layers of corrosion and weathered products, and uneven pieces and spaces, causing stress. Therefore, the previous conservation was disassembled, and the vessel was restored.

Cleaning

Mechanical cleaning was carried out with soft brushes. The glass vessel was immersed in distilled water followed by warm water (Fig. 8a and b). Then, an acetone solvent was used in the assembly locations. The glass vessel has been disassembled into 40 pieces, as shown in Fig. 8c.

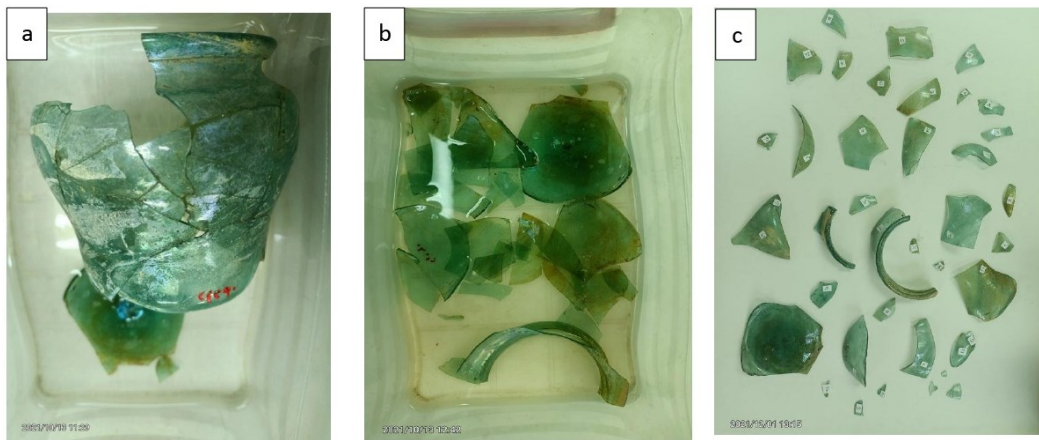


Fig.8. Illustrates the process of disassembling the previous restoration:
 a and b - during the disassembly process, c - after the disassembly into 40 pieces

Assembly process

Assembly was a two-stage process because the number of shards was large; the initial assembly was carried out using transparent adhesive strips (Fig. 9a and b). The second stage was the final assembly using Paraloid B-44 30% w/v in acetone (Fig. 9c), which is characterised by strong adhesion and low shrinkage during dryness. Moreover, its refractive index, expansion, and contraction are close to those of glass, causing no color change after aging like epoxy resins [33].

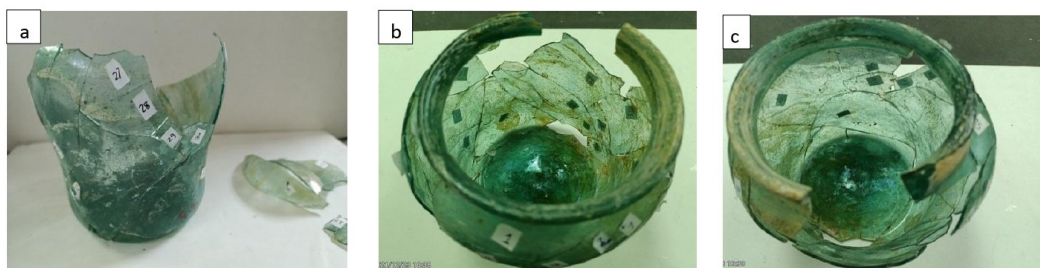


Fig. 9. Shows the stages of assembly of the glass vessel during treatment:
 a and b - during the assembly of glass pieces, c - after the assembly of glass vessel.

Gap filling with B-44 resin

Paraloid B-44 (40% w/v) solution was prepared in acetone. To slow the evaporation rate of acetone and reduce the formation of bubbles, a little amount of ethanol was added [30]. The mixture was mixed and cast in a prepared tray resistant to solvents and lined with silicon paper to facilitate the removal of the resulting film (Figs. 10a and b). Then, it was placed in a closed space and left for 4 days until complete hardening.

A sheet (50×50cm) was made, and as shown in Fig. 10c, it was taken into account that the volume of the resulting film shrinks during the drying and evaporation of the solvent. Koob and others report that volume shrinkage (70%) occurs for B-72 at 30% w/v concentration [29]. Controlling the required film thickness was also considered.

There are three large areas of loss on the glass vessel, namely the rim, the body, and the base. The outline of the required fill has been traced on the Paraloid B-44 sheet with a

permanent marker (Fig. 10e). The fills were cut out with a scalpel to the desired shape (Fig. 10f). It is advised to leave a small margin around the edges to support the missing parts from inside with 1mm from all sides. At this stage, any curvature must be considered. Therefore, the resin sheet can be softened by briefly heating it with a hairdryer to take the curve of the glass surface easily (Fig. 10d). The application of heat exploits the thermoplasticity of the resin, making it adaptable enough to curve into the desired shape before hardening as it cools. There is the advantage that the resin film can be repeatedly heated and cooled to take the curves accurately [28].

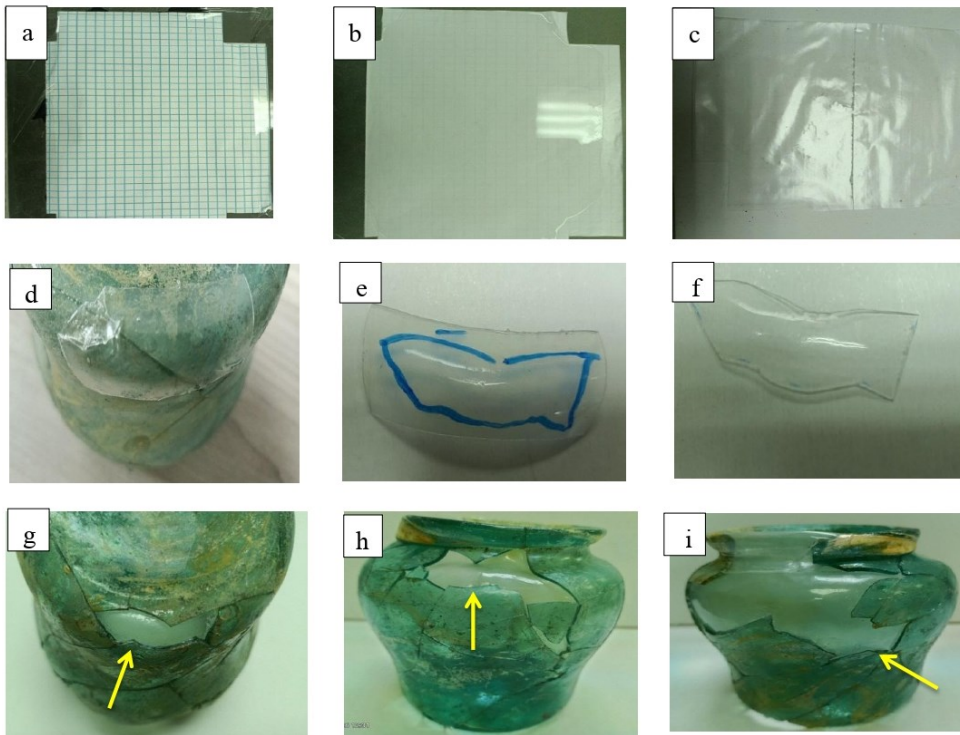


Fig. 10. Gab filling with resin slide (a) Showing the tray lined with silicon paper (b) Pouring paraloid 44 (40%) (c) The slide after drying (d) Shape of the loss was traced onto the film (e) Defining the area of loss compensation (f) Cutting the gap fill to size (g) Adhering the gap fill to the base of the glass vessel (h) Adhering the gap fill to body of the vessel (i) inserting the gab fill to the body and rim of vessel. (g, h, i) the glass vessel after treatment and conservation

After that, before adhering the gap fill to the glass vessel, carefully insert it into the area of loss to ensure a tight fit. For instance, wavy vessel walls meant that each edge of the filling needed to be angled slightly differently to properly align with the surrounding break edges. Patience was required at this stage as edges were cautiously trimmed away from the vessel and repetitively inspected against the area of loss. It is observed that the rim has a wide lip, so the fill edges were manipulated to form a lip with a modest overhang (Fig. 10i), which will provide additional support in these areas that are extremely vulnerable. Extreme caution should be taken not to apply pressure to any part of the vessel. Finally, the fills were placed in the areas of loss and adhered with a solution of 30% w/v Paraloid B-44 in acetone (Fig. 10g, h, and i).

Consolidation process

The mixture of nanoalumina and nanosilica 2% with Silres BS-290 at 7% was used to consolidate the glass surface because it has proven its efficiency and success in protecting the antique corroded glass [41].

Conclusion

The study showed that the acrylic resin (Paraloid B-44 40% w/v in acetone) proved efficient and successful as slides in filling and supporting the lost parts of the glass vessel under study. Casting a B-44 film has many advantages: It saves time and effort compared to other casting techniques using epoxy and polyester resins; it is prepared without touching the object and without complex moulding and casting processes; its slides can be formed and modified because they are flexible after hardening; and its fillings can be easily removed in case of necessary restoration.

Examinations and analyses showed that the glass vessel suffered from many deterioration manifestations, such as iridescence and corrosion layers. Microscopic examination showed pits, cracks, air bubbles, and fissures, indicating fragile glass. EDX analysis illustrated that this glass belonged to silica, sodium, and lime with a lower percentage of silica than the typical and low sodium and calcium, indicating that the vessel was corroded because a large amount of alkali (sodium oxide) moved outside the glass. Furthermore, there was a percentage of iron oxide, giving a green color to the glass vessel. Also, carbon oxide was found in a high percentage because of the previous restoration using a coating material. FTIR showed that polyvinyl acetate was used in the assembly and coating of the previous restoration, illustrating the yellow color on the surface of the glass because this substance turned yellow with aging.

The conservation of the glass vessel included disassembling the previous restoration, cleaning, reassembling, filling the lost parts, and consolidation. Paraloid B-44 was used in the final assembly and in the form of slides to fill and support the lost parts, but in different proportions so that there were no differences in the expansion and contraction between the material used in the adhesive and the material used for filling the lost parts.

It is hoped that sharing this experience will serve as a supplementary guide for conservation professionals who wish to perform similar treatments. It is also recommended to consider the condition of each artifact, as well as the temperature conditions, when selecting the appropriate resin to fill gaps and reinforce missing parts for better preservation.

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