





USING FIBERGLASS TO CONSOLIDATE COMPLEMENTING MATERIALS IN ARCHAEOLOGICAL GLASS "EXPERIMENTAL STUDY"

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ABSTRACT

Due to the breakable nature of glass, archaeological objects made of that material lose some of their parts and need to be completed.

A new technology using fiberglass is presented in the consolidation and improvement of the properties of complementing materials, which are used in the conservation of archaeological glass, especially the addition of Araldite 2020. This combination is characterised by the high adhesive strength of fiberglass. The study has dealt with the physical and mechanical properties of the samples before and after adding the fiberglass. Samples were exposed to different aging processes (Thermal aging, moisture aging, UV aging). The colour change of the samples was recognized by colorimeter and FTIR-ATR analysis to track the change of functional groups for the samples.

Microscopic images by USB digital microscope have shown interconnected mesh within samples containing Araldite 2020 and fiberglass. The study demonstrated the improvement of the properties of Araldite 2020 when added to fiberglass to consolidate archaeological glass.

KEYWORDS

Fiberglass; Archaeological glass; Consolidation; Araldite 2020; Completion; ageing.

الملخص

نظرا لطبيعة مادة الزجاج سهلة الكسر فإن الآثار الزجاجية تفقد بعض أجزائها وتحتاج إلى إجراء عملية الإستكمال. ويتناول البحث تقنية جديدة من خلال إستخدام الألياف الزجاجية في تقوية وتحسين خواص مواد الإستكمال المستخدمة في ترميم الزجاج الآثري. تستند فكرة البحث إلى دراسة إستخدام الألياف الزجاجية المضافة إلى مادة الأرالديت 2020 لعلاج واستكمال المواد الأثرية وتتميز بقوة لصق عالية بالألياف الزجاجية، حيث تناول البحث دراسة الخصائص الفنزيائية والميكانيكية للعينات قبل إضافة الألياف الزجاجية وبعد الإضافة، تم تعريض العينات لعمليات التقادم المختلفة (التقادم الحراري - التقادم الرطوبي -التقادم الأشعة فوق البنفسجيةU.V). تم التعرف على مقدار التغير اللوني للعينات من خلال جهاز الcolorimeter إلى جانب التحليل FTIR-ATR للتعرف على التغير في المجموعات الوظيفية للعينات. وتظهر الصور المجهرية بواسطة المجهر الرقمي USB للعينات شبكة مترابطة تكونت بالألباف الزجاجية داخل مادة الإستكال المستخدمة. أظهرت الدراسة تحسن خواص مادة الأرالديت Araldite 2020 المستخدمة عند إضافة الألباف الزجاجية. الكليات الدالة الألياف الزجاجية، الزجاج الآثري، تدعيم، أرالديت 2020، الإستكمال، التقادم.

1. INTRODUCTION

Archaeological glass is subjected to breaks and fragmentation, due to the breakable nature of glass material. This causes it to lose some of its parts forever, which are restored through the completion process.¹ The completion process aims to preserve the antiques by supporting the object structurally and completing its technical shape. The completion process is carried out in accordance with international conventions.²

Natural materials, such as animal glue, gum arabic, natural waxes (beeswax) and others, were used to strengthen and supplement the archaeological glass. Over time these were excluded by the scientific community due to many problems (fragility, colour change, drought, shrinkage, soil attraction).^{3&4&5} The discovery of manufactured materials, resins, and industrial adhesives^{6&7&8}, such as Epoxy, Acrylic and Polyester resins, ^{9&10&11&12} revealed that some provided good results in the collating and completing of the archaeological glass.¹³

In the glass conservation field, epoxy resins are mainly used as adhesives or as filling materials, thanks to the strength of their adhesion, their little shrinkage during drought, and their glass-like appearance.¹⁴ A common epoxy resin is Araldite 2020, as it has the ability to form strong and permanent bonds with glass.¹⁵ By adding an epoxy monomer, the molecules are connected together, forming hydroxyl groups that interact with other epoxy groups that form ether bonds; this results to a remarkable resin structure in three dimensions turning the material from this reaction, hard and strongly cohesive.¹⁶ However, the loss of mechanical properties of the epoxy resin occurs with ageing caused by photochemical reactions.¹⁷ Araldite 2020 is specifically designed for glass bonding, though it may also bind metal, ceramics, rubber, or rigid plastics¹⁸. This epoxy resin has a refractive index similar to that of glass. It is suitable for clear casts and laminates.¹⁹

¹Carmen et al., "Making 3D implants for conservation and restoration of archaeological glass", pp. 103-109

² Abd-Allah, "Study of the effective factor on deterioration of buried glass objects and its recent techniques of treatment and conservation" pp. 176.

³ Davison, "Conservation and restoration of glass" pp 199-226

⁴Hamad, "Study of factors affecting deterioration of archaeological glass" pp. 101

⁵ Coutinho, "Studies of the degradation of epoxy resins used for the conservation of glass" pp. 127-133

⁶ Henderson, "The Science and Archaeology of Materials: An Investigation of Inorganic Materials", pp. 24-108.

⁷ Davison, "Conservation and restoration of glass" pp. 107-112

⁸ Davison, "A History of Joining Glass Fragments." pp. 107–112.

⁹Roemich, "Glass and Ceramics Conservation"

¹⁰ Barton et al, "Examination and Experimentation: Conservation of an Archaeological Glass Unguentarium for Display." PP 69-78

¹¹ Down, "The Evaluation of Selected Poly(Vinyl Acetate) and Acrylic Adhesives" 33–54.

¹² Hana et al,"Archaeological Glass Conservation "PP188-206

¹³ Davison, "A History of Joining Glass Fragments." pp. 107–112

¹⁴ Roemich, "Glass and Ceramics Conserv ation"

¹⁵ Barton et al, "Examination and Experimentation: Conser vation of an Archaeological Glass Unguentarium for Display." PP 69-78

¹⁶ Down, "The Evaluation of Selected Poly(Vinyl Acetate) and Acrylic Adhesives" 33–54.

¹⁷ Hana et al,"Archaeological Glass Conservation " PP188-206

¹⁸ Esmeralda, "FT-IR spectroscopy of ageing of adhesives used in restoration of archaeological glass objects" pp.525:531

¹⁹ Mena et al., "Study the effect of thermal aging and its relationship with color change on adhesives used in collecting archaeological glass" pp 143:157

Araldite 2020 is a low-viscosity and transparent resin, which has a high mechanical durability. It is workable, resistant to discoloration, and has appropriate setting time.²⁰ It uses a system of two components which cure at room temperature. On glass, a degreasing agent, such as Leksol or acetone, should be used prior to its application to remove all traces of grease, grime, or dirt. Araldite 2020 is mixed by weight (100 parts of Part A to 30 parts of Part B) or by volume (100:35) and blended until the two components form a homogenous mix before applying to a clean dry surface. Be assembled, then clamp, and apply constant and even pressure for best results.²¹ When large areas are missing, more has to be done to support the object.

This was especially true for one of the glass balls "They are placed on top of the complaints, to keep them balanced" in the Museum of Islamic Art in Cairo, which was crushed during the recent bombing of 2014, which took place in the vicinity of this museum. As seen on Fig. 1, a large area of the ball went missing, which rendered difficult the completion process. The added support was provided by completing the shape with Araldite 2020.²² The same situation arises in the case of glass panels of archaeological windows,²³ which have lost large parts, as well as in the case of structural parts missing, such as handles or bases.



Figure 1 Archaeological glass ball with large missed area completed with Araldite 2020 from Islamic Art Museum in Cairo.

This study examines a new technique to support and improve the properties of complementing materials which are used for the conservation of archaeological glass, especially in the cases where large parts are needed to be complete or to be stronger. For instance, it may be found useful for the completion of bases of antique glass, large missing parts in archaeological glass, especially stained glass.

²⁰ Abd-Allah ,"In Situ Glass Conservation: A Case Study from the Archaeological Site of Barsinia / Jordan" pp 25-36

²¹ Mena et al., "Study the effect of thermal aging and its relationship with color change on adhesives used in collecting archaeological glass" pp 143:157

²² Mohamed, "Conservation and protection of the Damaged Archaeological Glass by the Explosion"pp178

²³ Pradell et al., "Techniques and Conservation of Historic Stained Glass" PP41-58.

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The main objective is to investigate the use of fiberglass to complement other materials (Araldite 2020) in the restoration of glass. This is done to know the extent of its effect on the physical and mechanical properties of the complementing materials. The basis of the fiberglass textile is silica SiO₂ in its pure form. Silica is found as a polymer, which does not have a melting point but dilutes up to 1200° C, as it begins to deteriorate. At 1713°C, most particles move freely. If the glass is cooled quickly at this temperature, it will not be able to form a tidy structure.²⁴ In the polymer, the SiO₄ groups formed in tetrahedron with a silicon atom are formed in the centre, with four oxygen atoms on the corners. These atoms then form a network attached to the corners by sharing oxygen atoms. The vitreous and crystalline states of silica (glass and quartz) have similar energy levels on a molecular basis, also implying that the glassy form is extremely stable.²⁵s²⁶

					-		<u> </u>				
Туре	Percentage Composition (%)										
of	SiO ₂	Al_2O_3	Fe ₂ O ₃	TiO ₂	B_2O_3	CaO	MgO	Na ₂ O	K ₂ O	ZnO	ZrO_2
GF							U				
Α	67.5	3.5	-	-	1.5	6.5	4.5	13.5	3	-	-
С	64.3	4.1	-	-	5	13.4	3.3	9.6	0.5	-	-
D	74	-	-	-	22.5	-	-	1.5	2.0	-	-
E	55	14	-	0.2	7	22	1	.05	0.3	-	-
R	60	24	0.1	-	0.3	9	6	.05	0.1	-	-
S	65	25	-	-	-	-	10	-	-	-	-
ECR	58.2	11.6	0.1	2.5	-	21.7	2.0	1.0	0.2	2.7	-
AR	61	1.0	-	-	-	5	1	14	3	-	15

Table 1 Chemical composition of fiberglass kinds²⁷

Although silica in pure form is a perfect glass fibre, it must be handled at very high temperatures, which is a defect unless specific chemical properties are needed. Usually impurities are introduced into glass in the form of other materials to reduce the working temperature. These materials also transfer various other properties to glass that may be useful in different applications. The type of glass used for fibres is E-glass, which is alumina borosilicate glass.²⁸ Most glass fibres have limited solubility in water.²⁹

2. MATERIALS AND METHODS 2.1 Materials

2.1.1 Araldite 2020

Araldite 2020 is a transparent epoxy material that is characterised by a high viscosity and a high luminosity. It adheres to surfaces of complementing materials and fiberglass, and benefits from a high chemical resistance. Araldite as most epoxy

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²⁴ Gupta et al., "Manufactured Fibre Technology" .London, pp. 544–546

²⁵ Hamad, "Restoration and conservation of a model of archaeological glass objects used for medical purposes. (2014) pp 51-58.

²⁶ Loewenstein (K)" The Manufacturing Technology of Continuous Glass Fibers" pp. 294

²⁷ Amer et al., "Performance and properties of glass fiber and its utilization in concrete - A review" pp296-1:296-10

²⁸ Milos et al., "Technical Approach to Glass" New York, (1990) pp 22:28

²⁹ Amer et al., "Performance and properties of glass fiber and its utilization in concrete - A review" pp296-1:296-10

resins, is based on reacting epichlorohydrin (ECH) with bisphenol A, which results in a chemical substance known as bisphenol A diglycidyl $(C_{15}H_{16}O_2)$.³⁰

2.1.2 CMB Fiberglass[®]

CMB³¹ fiberglass is short fiberglass that are mixed with epoxy materials for excellent mechanical properties. Glass type: E Fibres diameter: 13 microns The length of fibres: 12-16 mm

Combined with epoxy mixtures 0.5 - 1% by weight of epoxy materials. In the case of epoxy mixtures, the compounds of the substance are mechanically mixed for 3 minutes, and then glass fibres are added with constant mixing until homogeneity.

2.1.3 Samples

8 samples were prepared in 5cm length, 2cm high; 4 samples from Araldite 2020, and 4 others with Araldite 2020 combined with CMB fiberglass of 1% of the weight of Araldite 2020.

Table 2 Operating properties for Aradite 2020 and Aradite 2020 with fiber glass									
Samples	Operating time	Time of final hardening	Parts by weight						
Araldite2020	25:40 minutes	14 hours	3 resin: 1 hardener						
Araldite 2020 + fiberglass	25:35 minutes	12 hours	3 resin: 1 hardener + 0.5 - 1% by weight of Resin						

Table 2 Operating properties for Araldite 2020 and Araldite 2020 with fiberglass

2.2 Methods

2.2.1 Physical properties

Colour and degree of transparency of the samples were visually examined. The volumetric weight of the samples was measured at the Housing and Building National Research Centre in Cairo (HBRC).

2.2.2 Mechanical properties

The objective of identifying the mechanical properties of the epoxy Araldite 2020 combined with CMB fiberglass is to use materials whose hardness is proportional to the hardness of glass. In the case of a fragmented artefact, the material used must be able to withstand the same factors pressures than the glass and to identify the mechanical properties of Araldite 2020 individually, then after adding the fiberglass, it was measured.

2.2.1 Hardness

Hardness was measured for selected samples at the National Research Centre (NRC).

2.2.2 Compressive resistance

Compressive resistance was measured for the samples of Araldite 2020 and those of Araldite 2020 with CMB fiberglass, at Housing and Building National Research Centre (HBRC).

³⁰ Abd-Allah, "Hard and Tough Epoxy Resins Modified with Thermosetting Materials" pp 78-88.

^{*} CMB "Chemicals For Modern Building" company, cmb@cmbegypt.com

2.2.3 Accelerated ageing

For the separate thermal, UV and moisture ageing experiment, two replicate samples of Araldite 2020 and Araldite 2020 mixed with CMB fiberglass were used for each accelerated ageing. Samples in the thermal accelerated ageing were exposed to 60 °C for twenty cycles every cycle is 8-hour, while moisture ageing samples were exposed at 100% humidity for twenty cycles of 8 hours each.

The samples were then subjected to UV for 200 hours, corresponding to ASTM standards. One test piece of each resin was kept in darkness to act as a reference sample. Ageing tests were conducted in the laboratories of the Faculty of Archaeology, Fayoum University.

2.2.4 USB digital microscope

The samples were examined by Leuchtturm USB Digital Microscope (China) with 20 to 500 x zoom, using 8 LED lights with Measurement Software.

2.2.5 Colorimetry

Colorimetric measurements were performed in accordance with the Commission Internationale de l'Éclairage (CIE), lab colour system "1976" using Spector densitometer "Exact X-Rite, Switzerland".

2.2.6 Attenuated total reflectance-Fourier transform infrared (ATR – FTIR) spectroscopy

Analyses through the infrared spectrum with the ATR were done to identify the change in the functional groups of each sample after the different ageing processes. These were processed in the Restoration laboratory of the Islamic Art Museum. Device specifications (ATR – Platinum - serial number 12382310, Bruker, 64 Scan).

3. RESULTS

3.1 Physical properties

						8 8					
	Colour and transparency degree					Volumetric weight (gm/cm ³)					
Samples	Before ageing	After moisture ageing	After thermal ageing	After U.V ageing	Before ageing	After moisture ageing	After thermal ageing	After U.V ageing	(gm/cm ³)		
Araldite 2020	High transparent	High transparent	Yellow	Yellow	1.18	1.18	1.17	1.18	2.58		
Araldite 2020 + Fiberglass	Transparent	Transparent	yellowish	yellowish	1.21	1.21	1.20	1.21	2.46		

Table 3 Physical properties for samples before and after ageing



3.2 Mechanical Properties

Table 4 Mechanical properties for samples before and after ageing

		Hard	ness		Compressive resistance (kg/cm ²)			
Samples	Before ageing	After moisture ageing	After thermal ageing	After UV ageing	Before ageing	After moisture ageing	After thermal ageing	After UV ageing
Araldite2020	4.5	4.5	4.4	4.5	308.6	308.6	302.2	308.5
Araldite2020 + Fiberglass	4.7	4.7	4.7	4.8	312.4	312.2	312.1	312.6



3.3 Accelerated ageing

There was no noticeable colour change in the fiberglass except during thermal ageing, while the Araldite 2020 changed to yellow with UV exposure ageing (Fig. 5). Cracks appeared with Araldite 2020 during thermal ageing after the ninth cycle, while they did not appear in samples with added fiberglass (Fig. 6).



Fig.5, Samples of Araldite 2020 and Araldite 2020 + Fiberglass before and after moisture, thermal and UV ageing.



Fig. 6, "A" shows no cracks in sample of Araldite 2020 + Fiberglass, shows cracks in Araldite 2020 after thermal ageing.

3.4 USB digital microscope

Microscopic images clearly showed interconnected lines of fiberglass inside the samples consisting of Araldite 2020 and Fiberglass (Fig. 7).



Fig. 7, USB digital microscope photos for Araldite 2020 + Fiberglass.

3.5 Colorimeter

The colour change within samples was evaluated after the ageing processes thanks to a colorimeter. It must be mentioned that the colorimeter is not usually used with transparent objects, but the objective was to identify the amount of colour change. As a result, a white background for the samples was developed, and the measurements were taken using a colorimeter in the original image, as well as in the samples after the different ageing processes.

Fiberglass was not significantly affected by the ageing processes except in thermal ageing. The colour change decreased in moisture ageing and was minimal in UV and thermal ageing. On the other hand, the colour change was more noticeable in Araldite 2020 (Table 4).

The total amount of colour change (ΔE) is ^{32&33} $\Delta E^* = \{(\Delta L^*) 2 + (\Delta a^*) 2 + (\Delta b^*) 2\} 1/2$

3.6 ATR-FTIR analysis

There was no noticeable change in the functional groups of the Araldite 2020 sample with the fiberglass after moisture ageing (Figs. 8-9), while there was an increase in the OH (3300 -3400), C=O (1650-1750) and CH (2800-3000) groups in Araldite 2020 samples (Figs. 12-13). After thermal ageing, there was an increase in the extension of all functional groups for Araldite 2020 samples (Figs. 8, 10), which decreased in samples of Araldite 2020 with Fiberglass (Figs. 12, 14). UV ageing produced the same results in samples containing solely Araldite 2020 and those with fiberglass, where one observed a decrease in the extension of all functional groups (Figs. 11, 15). In all graphs blue represents the standard sample (before ageing), while red is after

ageing.

³² Wyszecki and Stiles, "Color Science Concepts and Methods. Quantitative Data and Formulae"

³³ Atodiresei et al., Chromatic Characterization in Cielab System for Natural Dyed Materials, Prior Activation in Atmospheric Plasma Type DBD"



Table 4 Results of colour	change of samples	after ageing
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Ŵŵ Fig. 10, (ATR-FTIR) spectroscopy for Araldite 2020 before and after thermal ageing.

2000

1000

0.3



Fig. 11, (ATR-FTIR) spectroscopy for Araldite 2020 before and after UV ageing.



Fig. 12, (ATR-FTIR) spectroscopy for Araldite 2020 + Fiberglass before ageing.



Fig.13,(ATR-FTIR) spectroscopy for Araldite 2020 + Fiberglass before and after



Fig.14, (ATR-FTIR) spectroscopy for Araldite 2020 + Fiberglass before and after thermal ageing.



Fig. 15, (ATR-FTIR) spectroscopy for Araldite 2020 + Fiberglass before and after UV ageing.

4. **DISCUSSION**

The physical properties of Araldite 2020 were improved when adding fiberglass, as these fibres stabilized the properties of volumetric weight and transparency. Araldite 2020 changed colour as evidenced by the study (Figs. 2-3, Table 3).^{34&35}

The density of Araldite 2020 and Araldite 2020 with CMB fiberglass is 2.58 - 2.46 gm/cm³.

Both are suitable for the density of the mother glass which ranges from 2.4 to 5.9 gm/cm³.³⁶ The mechanical properties of Araldite 2020 improved after the addition of fiberglass, as the hardness of Araldite 2020 mixed with CMB fiberglass ranges from 4.7 to 4.8. It is thus close to that of the mother glass, which ranges from 4.5 to 6.5, according to the Mohs scale of hardness.³⁷

The resistance to pressure of the samples increased (Fig. 4, Table 4), which is an important property in the completing materials for archaeological glass. Araldite 2020 mixed with CMB fiberglass is more similar to the mother glass than Araldite 2020 alone. 38&39

Fiberglass is highly resistant to thermal, moisture and UV ageing (Fig. 5), improving the efficiency of Araldite 2020 against all factors that affect epoxy resins, especially thermal and UV ageing.^{40&41}

A bonded network of fiberglass formed inside Araldite 2020 resin (Fig. 7), which strengthened the adhesive properties of the completing material. This in turn improved its properties and increased resistance to affecting weathering factors.⁴²

Xiaoyu et al., "Effect of Epoxy Resin on the Actuating Performance of Piezoelectric Fiber Composite" pp 1:10

Mena, "Experimental study of adhesives used for conservation of archaeological glass"

³⁶ Davsion, "Conservation and restoration of glass" PP 14

³⁷ Davsion, "Conservation and restoration of glass", PP 14

³⁸Xiaoyu et al., "Effect of Epoxy Resin on the Actuating Performance of Piezoelectric Fiber Composite" PP 1:10

Mohamed, " Conservation and protection of the Damaged Archaeological Glass by the Explosion" pp 101:105

⁴⁰ Hamad "Restoration and conservation of a model of archaeological glass objects used for medical purposes. museum of Islamic art in Cairo (Applied Study)" pp51:58

Hamad et al.,"Comparative study of traditional adhesives used in restoration of archaeological glass"

⁴² Coutinho, "Studies of the degradation of epoxy resins used for the conservation of glass" pp127 :133

Cracks on the surface of Araldite 2020 appeared after thermal ageing because of internal stresses by the tensile strength and pressure (Fig. 6), while there were none in the Araldite 2020 + CMB Fiberglass samples. This confirms the added support by fiberglass to the completing material, which is characterized by a strong support and adhesion to the resinous materials.⁴³ Both samples were highly transparent, and the addition of fiberglass did not affect the degree of transparency. It also did not affect the degree of colour in the coloured samples (Fig. 8); this was tested due to the common use of coloured antique glass.^{44 & 45}

ATR-FTIR show in Araldite 2020 with fiberglass relative match in the extension of the functional groups after moisture ageing on the contrary of Araldite 2020, where an increase is seen due to moisture. The extension of the functional groups increased after the thermal ageing in Araldite because of the oxidation processes (Figs. 8, 15).⁴⁶

5. CONCLUSIONS

Archaeological glass is difficult to consolidate and restore due to the fragile nature of the material. So far epoxy resins have been used, but it need further strengthening. As a result, to offer a solution to this issue as well as to provide structural support to pieces where large parts are missing, a material composed of Araldite 2020 and CMB Fiberglass was tested.

Fiberglass used in supporting epoxy completion materials for restoration of archaeological glass brings additional resistance to thermal, moisture and UV ageing. Indeed, through analyses it has been demonstrated that fiberglass improves the efficiency of the physical and mechanical properties of epoxy resins. In combination, they can be used with transparent or coloured samples. Fiberglass helps and provides positive results when completing large missing portions. In conclusion, the present study recommends conducting future applied research in the use of fiberglass to support completing materials, which are used for archaeological glass windows.

⁴³ Satheeshkumar et al., "Glass fiber-reinforced polymer composites" pp1258 - 1275

⁴⁴ Shelby, "Introduction of glass science and technology" pp209

⁴⁵ Pollard, et al., "Archaeological chemistry" pp156-158

⁴⁶ Mohamed, "Conservation and protection of the Damaged Archaeological Glass by the Explosion"pp106:123

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