



EVALUATION OF KEMAPOXY150 3D IN RESTORATION OF ARCHAEOLOGICAL GLASS

Mohammad HEFNI¹, Nagwa S. Abd AL-RAHIM², Hamdy Abd AL-MONEAM¹, Rasha T. HAMAD^{2,*}

¹ Conservation Laboratory, Museum of Islamic Art, Ministry of Tourism and Antiquities, Cairo, Egypt. ² Conservation Department, Faculty of Archaeology, Fayoum University, POBox 63514, Fayoum, Egypt.

Abstract

This research discusses an important topic related to the restoration of archaeological glass in museums and excavation sites, as well as in antiquities stores. Egypt abounds in many glass monuments dating back to different eras, starting from the ancient Egyptian era and the beginnings of the modern era (the era of Muhammad Ali's family). Due to the nature of the glass, that is easy to break, we often find the glass traces extracted from the excavations being in a state of fragmentation, in addition to the possibility of being exposed to breakage inside museums and archaeological stores, whether this resulted from the wrong handling of them, a mistake during the restoration, or a false display style of the glass antiques in the museum. We find that the basic restoration process in this case is represented in the assembly and completion phases, and here the primary material used in the restoration process is the adhesive material. The epoxy is the most used material in the collection and completion of the glass antiques. This research aims to identify the efficiency of the Kemapoxy150 3D epoxy material for use in restoring glass antiques, which is easily available for use in museums and archaeological sites in Egypt as it is made in Egypt. The research deals with the study of Kemapoxy150 3D in terms of identifying its physical and mechanical properties, and in terms of the result of different aging (thermal, UV, moisture). Results are evaluated by identifying the extent of color change of samples after aging by a colorimeter, as well as identifying changes in functional groups of samples after aging by analyzing the ATR infrared spectroscopy.

Keywords: Archaeological glass; Shattered parts; Assembly; Completion; Kemapoxy150 3D; aging.

Introduction

The process for assembly broken glass parts can be called the building process of the piece [1]. Initially, natural materials were used to glue the broken parts to glass traces such as animal glue, Arabic gum, natural waxes (beeswax) and others, but they were excluded with the passage of Time due to its exposure to many problems "brittleness, color change, drought, shrinkage, soil attraction [2, 3], chemical inertance, resistance to fungal infection and air pollution so as not to attract dust and plankton, resistance against moisture damage, high resistance to stress, tears and tensile strength, not to cause stresses during or after the hardening process that adversely affects the glass to become transparent and colorless" [4]. It also causes some color change [5]. After discovering the materials made from resins and adhesives, some were given good results in the field of collecting antiquities [6].

^{*} Corresponding author: rta00@fayoum.edu.eg

The refractive index of the glass can be defined as the ratio of the speed of light in a vacuum to the speed of light in the glass [7], given that the refractive indexes of alkali silica glass increase with an increase in the concentration of alkali oxide. The density of the glass also plays a significant role in affecting the refractive index [8, 9].

Materials and methods

Kemapoxy150 3D

It is an epoxy resin manufactured by an Egyptian company called "Chemicals Modern Building Company" (CMB Company, 2019) [10]. The epoxy material is characterized by its easy penetration and the high transparency and durability. The state of the gel generation until the hardening is complete [11]. The epoxy resin belongs to the group of thermosetting resins, as these resins are not able to be remodelled with heat after converting into a solid (non-regenerative) as a result of the formation of long polymeric chains intertwined with each other, which is called crosslinking. The epoxy resin contains two or more groups of epoxide groups, which consists of an oxygen atom bound to two carbons. The epoxy group chemically bonds with other molecules to form a three-dimensional network that is interlinked with the treatment process. The epoxy resin is characterized by relatively high hardness and chemical resistance, in addition to that, it has a high specific adhesion due to the chemical composition of this resin, which is represented by the group of ethers, hydroxyl, and polar groups that give high durability and adhesion, and the material gains hardness and strength [12].

Experimental study

This study is built on studying the properties of the material and the extent of color change, as well as the variation in the functional groups of the material after being subjected to various industrial obsolescence processes, where the material samples were exposed to different stages of aging, and the results were identified before and after the aging.

This study passed through the following steps study of the physical and mechanical properties of Kemapoxy150 3D followed by the study the effect of thermal, UV and moisture aging on the Kemapoxy150 3D.

Operating properties of Kemapoxy150 3D

In table 1 the operating properties of Kemapoxy150 3D are presented.

Time of final drying	Operating time	Maxing ratio of the two compounds A& B by weight	Material
24 hours	45 minutes	Resin 3 hardener 1	Kemapoxy150 3D

Table 1. Kemapoxy150 3D operating characteristics

Samples

Samples of kemapoxy150 3D adhesives selected for experimental study with the abovementioned ratios of resin and hardener were prepared in a restoration laboratory at the Islamic Art Museum in Cairo, and the samples were prepared in two images, the first image in a form of templates and the second in a form of chips "a thin layer of epoxy".

The goal of preparing samples in the form of molds and chips is to see the effect of aging process on the material in its form as a supplement (image of molds), and in its image as a collection material (chips), knowing that measuring the degree of color change in different aging process was done on the epoxy material in the form of the sheets (The thin layers of epoxy material) only, with the intention to identify the total amount of the color change of the epoxy material after it has been subjected to aging.

As for the preparation of samples in the form of epoxy molds, plastic cylinders were used and covered in a tight and transparent form. The epoxy material was poured, and after drying, the plastic cylinders and the transparent tube were removed. As for preparing samples in a form of chips, two layers of polyethylene were used to make out each sheet so that they are Isolated by using cotton dampened with neutral soap to be dried. The epoxy material is placed between the two layers to put polyethylene on an equal surface and at the end an equal weight is placed on the polyethylene so that the epoxy sheet is equal. After having it dried, the polyethylene layer is removed while the epoxy sheet remains.

Kemapoxy150 3D Physical and mechanical properties

The compressive resistance and the volumetric weight of the samples were measured at the Housing and Building National Research Center (HBRC), while the hardness was measured for selected samples at the national research center (NRC).

Ageing tests

Aging processes were performed to determine the extent of the aging effect.

Study the effect of the (thermal - ultraviolet radiation (UV) – moisture). All ageing tests were done in the Restoration Laboratory of Islamic Art Museum in Cairo.

Thermal aging was done to Kemapoxy150 3D for consecutive sessions at 80°C temperature for 200 hours [12], UV and moisture aging used for 200 hours.

Colorimetry

Precise Color Reader - Model: WR-10QC was used to measure the color change (ΔE^*) in the samples. The total color difference ΔE^* was also calculated from the following formula: $\Delta E^* = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{\frac{1}{2}}$ [13-15].

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

FTIR- ATR analysis was used to identify the effective groups change of the samples after the aging processes. It should be clarified that the colorimeter is not used with transparent materials to know the degree of color of Kemapoxy150 3D using the device, but to identify the amount of color change. Therefore, one background was set for the samples (white background) and the measurements were taken using a measuring device Colorimeter in its original form and after different aging processes. Analyses were done in the laboratory of restoration of the Islamic Art Museum.

The device used is a ATR - Platinum 64 Scan - serial number 12382310 by Bruker.

Results and discussion

Physical properties

The color and degree of transparency of the samples was recognized by visual examination. The volumetric weight (density) of the sample of epoxy was measured, which is with a density of $1.08g/cm^2$ in Table 2 and that density is proportional to the glass [16].

Mechanical properties

Hardness

The hardness degree of Kemapoxy150 3D is 4 (Table 2), and that is very suitable to use as adhesive material in restoring glass antiques. Hardness is the body's resistance to scratching by another body, and the hardness of any material is determined according to the Moh's Scale for hardness, which contains ten minerals that start with the lowest hardness metals, Talc, and ends with the most hardneed metals, which is Diamond [17].

Pressure resistance

It gave a result of 297.03kg/cm² in Table 2, and this result did not contradict with its use as an adhesive material in restoring glass antiques [18].

Table2. Physical and Mechanical properties for Kemapoxy150 3D					
Pressure resistance	Hardness	Volumetric weigh g/cm ²	Color & transparency		
297.03	4	1.08	High transparency		

Colorimetry

In the thermal aging, it appears from the measurements using the Colorimeter that the amount of the total color change ΔE^* is 10.31 (Table 3). This means that there has already been a yellowing color change (Fig. 1), but it is not appreciated by the significant color change compared to the temperature and aging time represented by 200 hours. This is a better result than commonly used supplement materials such as Araldite [19].



Fig. 1. Sample of Kemapoxy150 3D: A. before; B. after thermal aging

UV aging showed that the total change of the color ΔE^* is 7.28 (Table 3). This is a small change (Fig. 2) compared to the aging time of 200 hours, at a time when UV aging has been proven to cause higher color changes to most epoxy materials used in glass restoration [11].

The aging by moisture did not cause a significant color change (Fig. 3) as the amount of the total change of color ΔE^* is 2.86 (Table 3), which is less than 5 and therefore are noticeable changes as it is less than the number 5 no It is a noticeable color change [20].



Fig. 2. Sample of Kemapoxy150 3D: A. before: B. after UV aging

Fable 3. Results	of color	change of Ke	emapoxy150 3D
------------------	----------	--------------	---------------

ΔE*	ΔE*	ΔE*	Material
Thermal	(UV)	Moisture	
10.31	7.28	2.86	Kemapoxy150 3D



Fig. 3. Sample of Kemapoxy150 3D: A. before; B. after moisture aging

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

After the thermal ageing step, there was an apparent decrease in the extension of OH 3300 - 3400cm⁻¹, and an apparent decrease in the extension of C = O 1650 - 1750cm⁻¹, as well as a decrease in the extension of CH 2800 - 3000cm⁻¹ (Fig. 5). UV ageing showed that there was a significant increase in the extension of OH 3300- 3400cm⁻¹, and there was a slight increase in the tide of C = O 1650 - 1750cm⁻¹, and an increase in the extension of CH 2800 - 3000cm⁻¹ (Fig. 6). While in moisture aging step, there was a decrease in the extension of OH 3300- 3400cm⁻¹, and a match occurred in the extension of C = O 1650 - 1750cm⁻¹, and a decrease in the extension of CH 2800 - 3000cm⁻¹ occurred (Fig. 7).

The extension changes between increase and decrease because of the oxidation during ageing processes [21-23].



Fig, 4. ATR-FTIR spectroscopy for Kemapoxy150 3D, standard sample before aging

The experimental study showed a high efficiency of Kemapoxy150 3D for use in restoring the glass antiques in Egypt. Therefore, the objective from the applied study is to introduce Kemapoxy150 3D in the framework of practical application in the restoration of the archaeological glass effects only and that, due to its satisfactory results on the experimental study.

It was applied to two antique pieces that are parts of colored glass bracelets at the Islamic Art Museum in Cairo, where the first piece consists of three fractures and the second piece consists of two fractures, both of which require the assembly process in which the Kemapoxy150 3D material was used: antiques No. 1: 19781/5 (Fig. 8A) and antiques No. 2: 19781/88 (Fig. 8B).



Fig. 5. ATR-FTIR spectroscopy for Kemapoxy150 3D, before and after thermal ageing



Fig. 6. (ATR-FTIR) spectroscopy for Kemapoxy150 3D before and after moisture ageing



Fig. 7. (ATR-FTIR) spectroscopy for Kemapoxy150 3D before and after UV ageing



Fig. 8. Glass antiques No 1 and 2 before restoration

In the first antique, initial assembly was carried out by fixing the two pieces in their correct place on a slice of wax [24, 25]. The final assembly was carried out by applying the epoxy (Kemapoxy150 3D) using a scalpel (Fig. 9).



Fig. 9. Glass antique No 1 during adhesive process by Kemapoxy150 3D



Fig. 10. Glass antique No 2 during adhesive process by Kemapoxy150 3D

As for glass antique No. 2, the initial collection was carried out by fixing the two pieces in their correct position on a slice of wax. The final assembly was carried out by using Kemapoxy150 3D (Fig. 10). In figure 11 is presented the objects after restoration using kemapoxy150 3D material.



Fig. 11. Glass antiques after restoration

Conclusion

Kemapoxy150 3D gave very good results and demonstrated high efficiency for use in restoring the archaeological glass in assembling and completion. As for the color property, the good degree of transparency and the mechanical properties, they are suitable for use in restoring the glass antiques.

The study recommends using Kemapoxy150 3D. It gave better results than the common materials used for restoring archaeological glass, while it is a locally made material that are available and cost-effective, which helps to carry out the restoration process in a good and fast way within museums and archaeological sites while promoting national industry.

Experimental studies of local restoration materials are recommended to identify quality material that can be used in the restoration field.

References

- [1] M. Moustafa, *Comparison study of the kinds of pottery and ceramic in Egypt with restoration and conservation of selected pottery antiques*, Master Thesis, Conservation Department, Faculty of Archaeology, Cairo University, Egypt, 1991, p. 131.
- [2] S. Bradley, *Preventive Conservation Research and Practice at the British Museum*, Journal of the American Institute for Conservation, 44(3), 2005, pp. 159-173.
- [3] S. Davison, Conservation and Restoration of Glass, Oxford, Second edition, 2006, p. 180.
- [4] S. Koob, New techniques for the repair and restoration of ancient glass, Studies in Conservation, 45, 2000, pp. 92-95.
- [5] W. Hyun, C. Som, B. J. Soo, A Study on the Development and Physical Properties of Low Yellowing Epoxy for Ceramic Preservation, Journal of Conservation Science, 30(2), 2014, pp. 181-187.
- [6] S. Davison, A review of adhesives and consolidants used on glass antiquities, Studies in Conservation, 29, 1984 - Issue sup1, pp. 191-194.
- [7] J. Jerzy, K. Goldfinger, Unstable historic glass: Symptoms, causes, mechanisms and conservation, Studies in Conservation, 53, 2008 Issue sup2, pp. 47-60.
- [8] H. Abd Alhafez, Physiochemical deterioration phenomena in the archaeological glass and its relation to chemical composition" experimental and applied study, Master Thesis, Conservation Department, Faculty of Archaeology, Cairo University, Egypt, 2005, p. 35.
- [9] J. Shackelford, R. Dormeus, Ceramic and Glass Materials "Structure, Properties and Processing" Springer, USA, 2008, p. 182.

- [10] R. Hamad, Study of factors affecting deterioration of archaeological glass objects used for medical purposes and methods of treatment and conservation (Applied on some selected objects)", PhD Thesis, Conservation Department, Faculty of Archaeology, Cairo university, Egypt, 2014, p. 90.
- [11] * * *, CMB GROUP, <u>www.cmb.eg</u> [accessed on 12.03.2020]
- [12] H. Bristow, J.D. Cutajar, Archaeological Glass Conservation: Comparative Approaches and Practicalities of Using Acrylic Resin Films as Gap Fills, AIC American Institute for Conservation Objects Specialty Group Postprints, 24, 2017, pp. 188–206.
- [13] J. Schanda, Colorimetry. Understanding the CIE System, Wiley-Interscience Hoboken, New Jersey, United States of America, 2007.
- [14] A.M. Saviuc-Paval, I. Sandu, I.M. Popa, I.C.A. Sandu, V. Vasilache, I.G. Sandu, Obtaining and Characterization of Ceramic Pigments for Polychrome Artistic Elements II. Microscopic and colorimetric analysis, Revista de Chimie, 63(2), 2012, pp. 170-178.
- [15] A.M. Saviuc-Paval, A.V. Sandu, I.M. Popa, I.C.A. Sandu, A.P. Bertea, I. Sandu, *Colorimetric and microscopic study of the thermal behavior of new ceramic pigments,* Microscopy Research and Technique, 76(6), 2013, pp. 564-571. DOI: 10.1002/jemt.22201.
- [16] E.J. Lee, Y. Sung, Evaluation of Physical Properties according to Mixing Ratio and the Survey of the Current Situation for Epoxy Resin used in Conservation, Journal of Conservation Science, 32(2), 2016, pp. 223-234,
- [17] I. Coutinho, A. Ramos, M. Augusta, B. Francisco, Studies of the degradation of epoxy resins used for the conservation of glass, Ancient and Modern Approaches to Joining, Repair and Consolidation Conference Proceedings, London England, February 2008, pp. 127-133
- [18] R. Hamad, Restoration and conservation of a model of archaeological glass objects used for medical purposes. museum of Islamic art in Cairo (Applied Study), SHEDET (Annual Peer-Reviewed Journal Issued by The Faculty of Archaeology, Fayoum University), 1, 2014, pp. 51-58.
- [19] R. Abd-Allah, Study of the effective factor on deterioration of buried glass objects and its recent techniques of treatment and conservation, PhD Thesis, Conservation Department, Faculty of Archaeology, Cairo University, Egypt, 2002, p. 20.
- [20] S. Jeong, N. Chul, A study on aging characteristics of epoxy resins for conservation treatment of cultural heritage by adding UV stabilizer, Analytical Science and Technology, 24(5), 2011, pp. 336-344,
- [21] E. Lopez-Ballester, M.T. Domenech-Carbo, J.V. Gimeno-Adelantado, F. Bosch-Reig, Study by FT-IR spectroscopy of ageing of adhesives used in restoration of archaeological, Journal of Molecular Structure, 482(Special Issue: SI), 1999, pp. 525-531
- [22] S. Mascarós, Glass science in art and conservation, Journal of Cultural Heritage, 9(Supplement), 2008, pp. e1-e4
- [23] W. Xiaoyu, W. Mingliang, G. Feng, Y. Xuemei, *Effect of Epoxy Resin on the Actuating Performance of Piezoelectric Fiber Composite*, Sensor Journal, Basel Switzerland, 19, 2019, pp. 1-10.
- [24] M. Hefni. Conservation and protection of the Damaged Archaeological Glass by the Explosion (Applied on some selected objects from Islamic art museum in Cairo), Master Thesis, Conservation Department, Faculty of Archaeology, Fayoum University, Egypt, 2020, pp. 170-220.

[25] Y. Shuichiro, *Quantitative evaluation of an epoxy resin dispersion by infrared spectroscopy*, **Polymer Journal**, **46**(7), 2014, pp. 430-434.

Received: April 05, 2020 Accepted: February 23, 2021