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# Evaluation of green solvents on removal of aged Epoxy from ancient glazed surface.

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## ABSTRACT

This research elucidates the removal of aged epoxy layers from historic glazed surfaces, a significant challenge encountered by conservators. The deterioration results from mechanical methods that create surface scratches, or chemical approaches that employ hazardous chemicals detrimental to both artifacts and conservators. Epoxy was extensively utilized for assembly, consolidation, and protection. Their removal is typically necessary, since coatings can exacerbate the fragility of the glazed surface, leading to the accumulation of undesirable substances. While it has become prevalent in conservation, it is a sensitive technique that must be executed appropriately. The objective of this study was to assess the efficacy of green solvents in the removal of aged epoxy polymers, such as Araldite 2020, from historical glazed surfaces. This study examined two eco-friendly solvents: dimethyl carbonate (DMC) and ethyl lactate (EL). Microscopic and analytical techniques were developed for the epoxy-coated glazed surfaces and cleaned samples to assess the efficacy of the cleaning tests. Various microscopy techniques were employed, first with a digital microscope (DM) and subsequently utilizing a scanning electron microscope (SEM). Attenuated Total Reflection-Fourier Transform Infrared Spectroscopy (FTIR-ATR) identified the presence of araldite coating the surface in the study conducted at Zaafran Palace Museum, Egypt. Dimethyl carbonate demonstrated superior efficacy relative to ethyl lactate in the removal of both thick and thin aged epoxy, while minimizing the risks associated with chemical cleaning and mechanical procedures. Robust adhesion to the surface of the glazed artifacts.

## 1. Introduction

Epoxy resin can be used on a wide range of materials as stone, wood, metal, ceramics, and glass, making it highly versatile for various conservation projects. It has excellent chemical resistance, which helps in protecting artworks from environmental factors such as pollutants and moisture. Epoxy resins are one of the most used resins in glass and ceramic conservation and the Orasol dyes are formulated to color them and are used for color matching. It is used in the conservation and restoration of archaeological glass, variously for repair of fractures and edge-bonding, fixing loose paint layers, or as a protective coating for vulnerable areas. The epoxy, generically referred to as bonding agents, are applied as a solution, emulsion or glaze, and dry out to form a hard and adhesive film [1]. Epoxy resins are the commonest artificial resins used in stained glass restoration. These are resins with multiple additives based on an epoxy and an amine. By varying the composition of the components, it has been possible to create a wide range of products, which have been used as glues, liners or laminates for industrial and technical purposes. Epoxies have also been widely used in conservation on account of their superior adhesive qualities and strength, for example as metal-glue, or for the conservation of archaeological objects, ceramics and stone [2]. Epoxy resins have also been used since the beginning of the 1960s in the conservation of stained glass, mostly for break-fixing, but also for the targeted fixing of paint outlines, application over large areas, and plating [3]. The big advantage of epoxy resins compared with the thermoplastic polymerization resins (polyvinyl acrylates and acrylic resins) is their greater adhesive strength and their ability to adhere well to the base layer. It has already been mentioned that such adhesive strength can be problematic, in cases where the strength of the external film exceeds that of the glass matrix as conserved. This can lead to secondary breaks adjacent to the glued area, or the flaking or peeling away of the gel glass layers with the layer of bonding agent on account of its tensile strength, all typical symptoms of the use of epoxy resins. However, the biggest disadvantage of epoxy resins is their relatively poor durability. The reason for this is their polarized molecular structures: these can constitute a starting point for oxidation reactions, which are mostly photochemical in origin. This leads to yellowing or browning of the glass, often accompanied by changes in the physical characteristics of the film, which becomes

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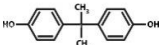
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considerably more fragile. The term yellowing is frequently used to describe this phenomenon. However, some polymers yield brownish and sometimes even different colors; so, the term discoloration is more preferred than yellowing. This discoloration is frequent with epoxy adhesives currently used for glass and non-porous ceramics restoration. In the case of glass, this phenomenon is particularly unsightly and understanding its origins could contribute to prevent steps to avoid its occurrence [4]. Epoxy materials are used to conserve glazed artifacts. Its Molecular Formula is  $C_{21}H_{25}ClO_5$ , It consists of two components, a resin and a hardener. At the beginning of the interaction between the two components, it is in the liquid state and then becomes in the gel state until the solidification process is complete [5].

**Table1.** Mechanical and chemical properties of epoxy resin [6].

PROPERTY	EPOXY RESIN
DENSITY(G/CM <sup>3</sup> )	1.15-1.18
SPECIFIC GRAVITY	1.14±
COLOR	colorless
VISCOSITY AT 25° C (MPA)	550±50
POT LIFE (MIN) AT 23° C	30±10
MIXING PROPORTION	10
GEL TIME (H) AT 23° C	24-36
MOLECULAR FORMULA	

Epoxy resin belongs to the group of thermosetting resins, as these resins cannot be reshaped by heat after turning into a solid material (non-Retrieval) as a result of the formation of long polymer chains intertwined with each other, which is called cross-linking. Epoxy resin contains two or more epoxide groups consisting of an oxygen atom bonded to two carbon atoms. The epoxy group is chemically bonded with other molecules to form a three-dimensional cross-linked network during the curing process. Epoxy resin is characterized by its relatively high hardness and chemical resistance. In addition, this resin has high specific adhesion ability due to the chemical composition of this resin, which is represented by ethers, hydroxyl groups, and polar groups, which give high durability and adhesion and give the material hardness and strength [7, 8]. Selecting a cleaning method is critical because it is irreversible and may cause permanent damage. Mechanical cleaning is considered one of the most appropriate for cleaning glazed and glass surfaces. However, it is insufficient when heavy hard layers of coating materials which used in conservation or crusts on glass surfaces. Furthermore, chemical cleaning based on the use of chemicals, such as solvents, acids, and various alkalis, is not desirable in the case of corroded glass artifacts, as they are fragile and full of pores, which penetrate those pores and make changes on the properties, weaken, and increase damage to the material [9]. Selecting a cleaning method is critical because it is irreversible and may cause permanent damage. Mechanical cleaning is considered one of the most appropriate for cleaning glazed and glass surfaces. However, it is insufficient when heavy hard layers of coating materials which used in conservation or crusts on glass surfaces. Furthermore, chemical cleaning based on the use of chemicals, such as solvents, acids, and various alkalis, is not desirable in the case of corroded glass artifacts, as they are fragile and full of pores, which penetrate those pores and make changes on the properties, weaken, and increase damage to the material [10]. Removing epoxy is considered one of the major challenges facing conservators and specialists in this field. There are many methods for removing epoxy from glazed surfaces. Heat treatment is used for the removal process, by heating it to reach the point of plasticity. It is one of the methods that are not recommended due to its danger and damaging effect on the artifacts [11]. Chemical methods are used in removing epoxy, and one of the materials used is dimethyl formaldehyde, which is considered a carcinogenic chemical that is harmful to conservators [12]. For all these reasons, the goal of the study was to use green, environmentally friendly materials and evaluate their efficiency in removing epoxy from the glazed surfaces.

Green solvents have been implemented and promoted in conservation as an alternative cleaning approach. Different organizations have made available guides classifying solvents according to their impact on the environment, health and safety (EHS), their life cycle assessment (LCA), and inherent energy costs of manufacture, application, and reuse or disposal. However, these various guides differ in compounds' classification due to the influence of the companies' individual preferences, differences in the numerical calculation of the gradings, and the diversity of type and number of parameters considered. The collaborative project CHEM21, developed jointly by entities that have written some of the previously mentioned selection guides, highlights this lack of consensus. The main drawbacks of green solvents are the possible impregnation and retention in the substrate and it's not a problem on the glazed surface due to its non-porosity [13]. Conservatives have recently been searching for greener alternatives to reduce environmental, healthiness and artifact impacts. In the past few years, green chemistry has gained ground foothold in the field of cultural heritage cleaning using micro-emulsions, gels and ionic liquids. [14-17] that have served in toxicity minimization with continuous development to achieve more desirable results for the safety of the artifact itself [13]. In this study, Dimethyl carbonate (DMC) and Ethyl lactate (EL) were evaluated as green solvents for the cleaning of the aged epoxy layer from the two glazed surface samples [18,19].

## 2. Materials and methods

### 2.1 Samples

The glazed samples were prepared and age as a 19th glazed vase dedicated to the Elzafran museum, Ain-Shams University, Cairo, Egypt. Each sample is 10 ×10 cm followed by aged epoxy (araldite 2020). Two Sample were prepared: first part includes glazed surface followed with araldite in thin layer (1mm) in the same concentration of coating process (3 resin: 1 hardener) Dissolved in 3% acetone [20]. The second part includes glazed surface followed with araldite in thick layer (3 mm) in the same concentration of assembling and completion processes (3 resin: 1 hardener) [21]. The prepared glazed samples were left after araldite application in laboratory conditions at a room temperature of 24° C and relative to completely harden for 14 hours.

## 2.2 Materials

### 2.2.1 Araldite®

Transparent epoxy material that is characterised by a high luminosity and high viscosity. It adheres to surfaces of complementing materials and fiberglass, and benefits from a high chemical resistance. Araldite as most epoxy resins, is based on reacting epichlorohydrin (ECH) with bisphenol A, which results in a chemical substance known as bisphenol A diglycidyl (C<sub>15</sub>H<sub>16</sub>O<sub>2</sub>) [22, 23].

**Table2.** Operating properties for Araldite 2020.

SAMPLE	OPERATING TIME	TIME OF FINAL HARDENING	PARTS BY WEIGHT
ARALDITE 2020	25:40 minutes	14 hours	3 resin: 1 hardener

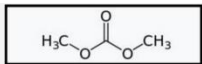
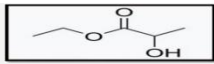
### 2.2.2 Dimethyl Carbonate (DMC)

Dimethyl Carbonate (DMC) is a carbonate ester with the formula of (C<sub>3</sub>H<sub>6</sub>O<sub>3</sub>). It possesses the properties of nontoxicity and biodegradability [14, 24]. It is an aprotic solvent classified in the greenest bracket according to solvent selection guide. DMC was purchased from Loba Chemie PVT.LTD with 99% purity.

### 2.2.3 Ethyl lactate (EL)

Ethyl Lactate (EL) is monobasic ester of ethyl -2 - hydroxypropanoate. It has the (C<sub>5</sub>H<sub>10</sub>O<sub>3</sub>). It is found in some variety of food naturally and can be produced from carbohydrates feedstock since it is produced from ethanol and lactic acid by fermentation of the biomass [25, 26]. It has been purchased from alpha chemicals with 99%.

**Table 3.** physiochemical properties of (DMC) Dimethyl carbonate and (EL) ethyl lactate [13, 19, 20, 27-31]

Property	Dimethyl carbonate	Ethyl lactate
Boiling point (°C)	90.3	151
Viscosity	0.589	0.0047
Density (g/cm <sup>3</sup> )	1.069	1.0328
Solubility of water	Partially soluble	Very soluble
Flash point(°C)	16	46
Vapor pressure (kPa)	0.002-0.01	0.0047
Hydrogen bonding parameter	1.9	6.1
Polar Hansen parameter	4.7	3.7
Hildebrand parameter	20.3	21.3
Dispersion Hansen parameter	8.5	7.8
Chemical formula		

## 2.3 Methods

### 2.3.1. Ageing

Samples were aged according to standard ISO for epoxy resin 7142-2023 with accelerated thermal aging (dry- heat) carried out at 105° C. Ageing times were 0, 0.6, 1.2, 3, 6, and 12 days, which corresponds approximately to 0, 5, 10, 25, 50, and 100 years of natural aging [14,32]. The samples were aged by thermally accelerated aging at 105° C for (360 hours) 15 days which is assessed to correspond to 125 years.

### 2.3.2. Cleaning test methodology

All coated aged glazed samples with araldite were cleaned with the chosen green solvents. Compresses and mechanical wiping were the selected cleaning methods. All samples are divided into four quarters. The number of compresses, wiping and cleaning time were unified for each quarter at a room temperature of 24° C. 10 ml of each solvent were used to clean the thin layer while 30 ml were used for the thick layer of aged araldite. To obtain correct results, a protocol must be followed based on unifying the principles [33], which was followed in this study by standardizing the time, thickness of the layer to be cleaned, and the amount of solvent, and the only variable was the green solvent.

### 2.3.3. Microscopy

They were used to examine the samples surface before and after cleaning and evaluate the effectiveness of the selected green solvents using the follows: Leuchtturm 20:500 x provided with 8 led lights (China) USB digital microscope, and Jeol (Tokyo, Japan) JSM 5600 LV equipped Scanning electron microscope (SEM), The images were taken under low vacuum conditions where samples did not show any charging effects.

### 2.3.4. Colorimetric measurements

Colorimetric measurements were performed in accordance with the (CIE) Commission International de l'Eclairage, lab color system "1976" using Spectror densitometer " Exact X-Rite, Switzerland".

### 2.3.5 Attenuated Total Reflection- Fourier Transform Infrared Spectroscopy (ATR- FTIR)

Used for analysis and identification of the coated layer over the glazed vase (case study) in compare of resin standard samples. FTIR spectra of samples were collected from Thermo Scientific- Nicolet Summit Lite FTIR spectrophotometer. Non-destructive analyses were conducted with Everest ATR –Factory Preset- ZnSe with a resolution of 4 cm<sup>-1</sup> in the 4000-400 cm<sup>-1</sup> range, 16 scans.

## 3. Results and discussion

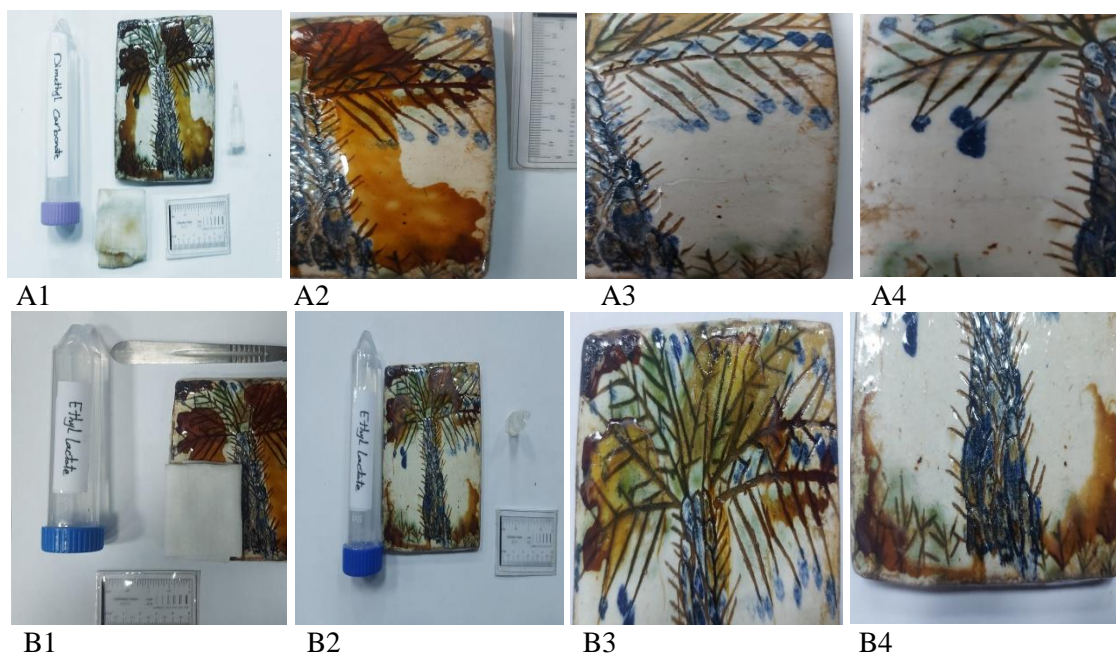
### 3.1 Case study documentation

Ceramic vase made in the De Sevres style, French. 19th c. AD/ 13th c. AH. It was customary for Muhammad Aly's Dynasty to decorate palaces with objects from Europe and other countries. This vase reflects the characteristics of the modern French school. It suffers from yellowish coated layers on its glazed surface This vase had previously been restored and some of its parts had been assembled and completed, but the restoration was unprofessional, as there was a thin yellowish layer covering the assembled and completed part and some parts of the surface on both sides of the vase. The analysis showed that this layer is made of araldite, which was used in the process of assembling, strengthening and consolidating it. The presence of this layer resulted in the obliteration of the decorations and colors of the vase in these parts.

### 3.2 Visual evaluation of cleaning test

Visual investigation of the cleaned samples with the selected solvents (dimethyl carbonate and ethyl lactate) showed good results for Araldite removal. This was clearly observed due to the color change of the compresses to yellow from the first compress. Dimethyl carbonate showed very effective results in removing both thin and thick layers of araldite. There was no difference except in the time required for the cleaning process. It also showed promising results in cleaning glazed surfaces that contain recessed and prominent decorations. Ethyl lactate showed slower results in araldite removal; it looked effective in the thin layer but in the thick it tacked more time. Control of cleaning test according to (Fig.1) was achieved unifying by the (solvent amount which used for cleaning each of the thickness of aged epoxy layers, the time exposure to the Solvent and compresses number) This could refer to the longer time needed for completely remove the all-epoxy layer.

Dimethyl carbonate gave promising results when removing a 1 mm thick epoxy layer in 6 minutes with an amount of solvent of 10 ml with an area of 6.25 cm. The same effective results were achieved with the same area, but with a thickness of 3 mm of epoxy in a period of about 18 minutes and 30 ml. The results were that ethyl lactate was less effective at the same previous rates, and the effectiveness was higher with doubling the time and amount of solvent used for cleaning with the same area to be cleaned and thickness.



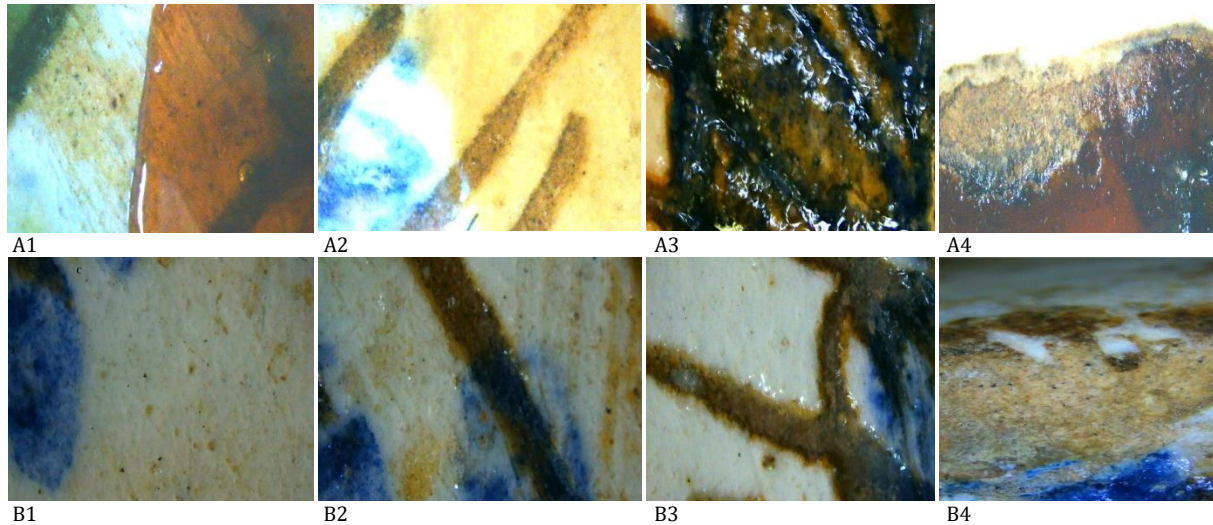
**Fig. 1.** Visual cleaning test for the epoxy layer with dimethyl carbonate and ethyl lactate. A1. Cleaning test with DMC, A2 thick layer of aged epoxy "araldite2020" A3. After remove thick layer of aged epoxy by DMC in 18 minutes, A4. After 6 minutes removing thin layer of aged epoxy by DMC. B1. cleaning test by ethyl lactate, B2. During removal thick layer of aged epoxy "araldite2020" B3. After 18 minutes of removing thick layer of aged epoxy by EL, B4. After 6 minutes of removing thin layer of aged epoxy by EL.



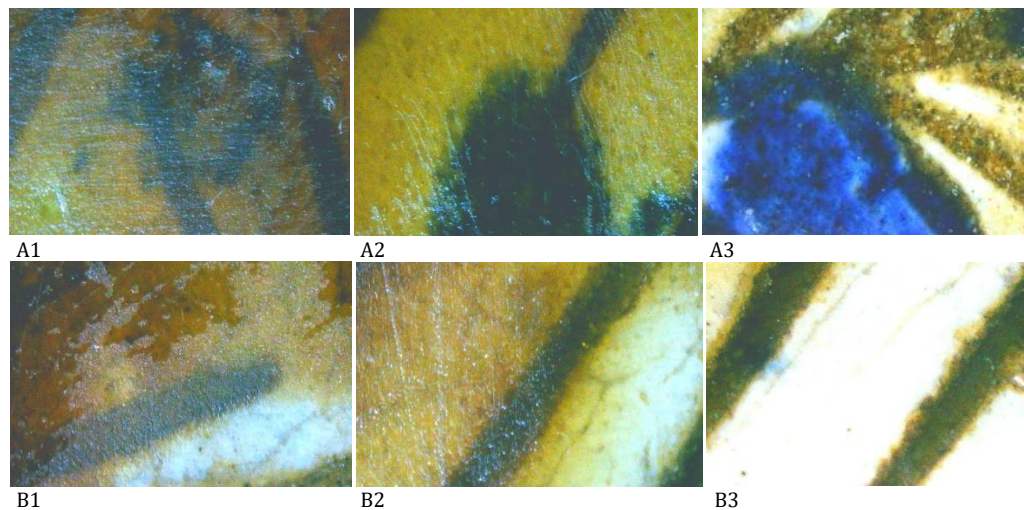
### 3.3. Microscopy

#### 3.3.1. USB Digital Microscope (DM)

USB digital microscope images showed the yellow color of the thin layer of araldite, which is used in the case of using araldite as a consolidation and coating material, and the very dark yellow color of the thick layer, which is applied in cases of assembly. Epoxy is used by weight (100 parts of part A to 30 parts of part B [22, 34]. Change in aged layer of surface color and yellowing of decorations, which blurs the decorations and distorts the artifacts surface, the color change of epoxy is one of the most common defects and its clearly appeared by ageing. [21,35] By comparing of EL and DMC; Ethyl lactate showed slower results in araldite Removal, it looked effective (Fig. 2) in the thin layer but in the thick it tacked more time This could refer to the longer time needed for using compresses and wiping to completely remove the all aged epoxy layer.



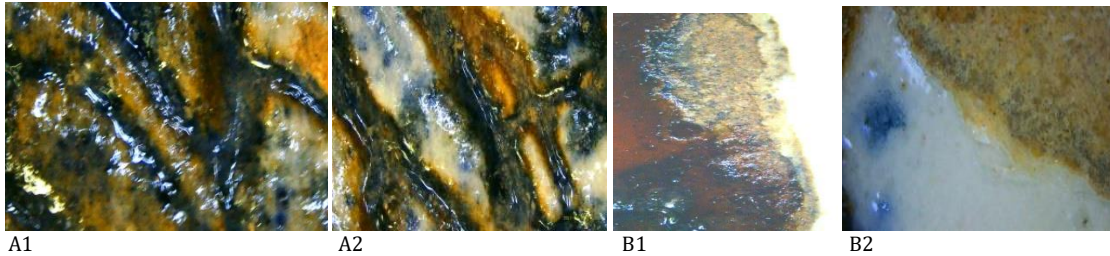
**Fig. 2** USB Digital images; A1. thick layer of aged epoxy on glazed surface. B1. After 18 minutes cleaning by DMC, A2. Thin epoxy layer, B2. After 6 minutes cleaning by DMC, A3. Aged epoxy on folds of the decorations on glazed surface, B3. After cleaning and removal by DMC, A4. Aged epoxy on the glazed and unglazed surface, B4. After cleaning both glazed and unglazed surfaces with DMC.



**Fig. 3** USB Digital images; A1. thick layer of aged epoxy on glazed surface. A2. After 18 minutes cleaning by EL, A3. After 30 minutes cleaning with EL, B1. Thin epoxy layer, B2. After 6 minutes cleaning with EL, B3. After 10 minutes cleaning with EL.

USB digital microscope images of the coated glaze surface with aged araldite cleaned with dimethyl carbonate showed the thin layer of araldite was removed after 6 minutes of using compresses with wiping mechanical removal. Microscopic images also showed the softening of the thick layer of araldite at the same time, and with continued application of compresses for a similar period, the removal process was complete. The results were effective in parts containing folds due to the presence of recessed and prominent decorations. One of the disadvantages of epoxy is the occurrence of color change on the surface, which works to hide the inscriptions and colored decorations that carry many cultural characteristics on the surfaces of glazed antiques [21]. Microscopic images of surfaces that were cleaned with ethyl lactate showed the removal of the yellow araldite layer, while the thick layers especially between the folds of the decorations, remained visible and did not completely remove, in the same time it took for dimethyl carbonate; it refers to the increasing in the effectiveness of DMC (Fig.3). While ethyl lactate and dimethyl carbonate were tested to swell the Araldite from glazed surface, the results showed that dimethyl carbonate is more effective (Fig. 2&3&4) than ethyl lactate as a good sweller to Araldite. While ethyl lactate and dimethyl carbonate are considered from the ester's family, the experimental study showed that ethyl lactate needs a longer time to

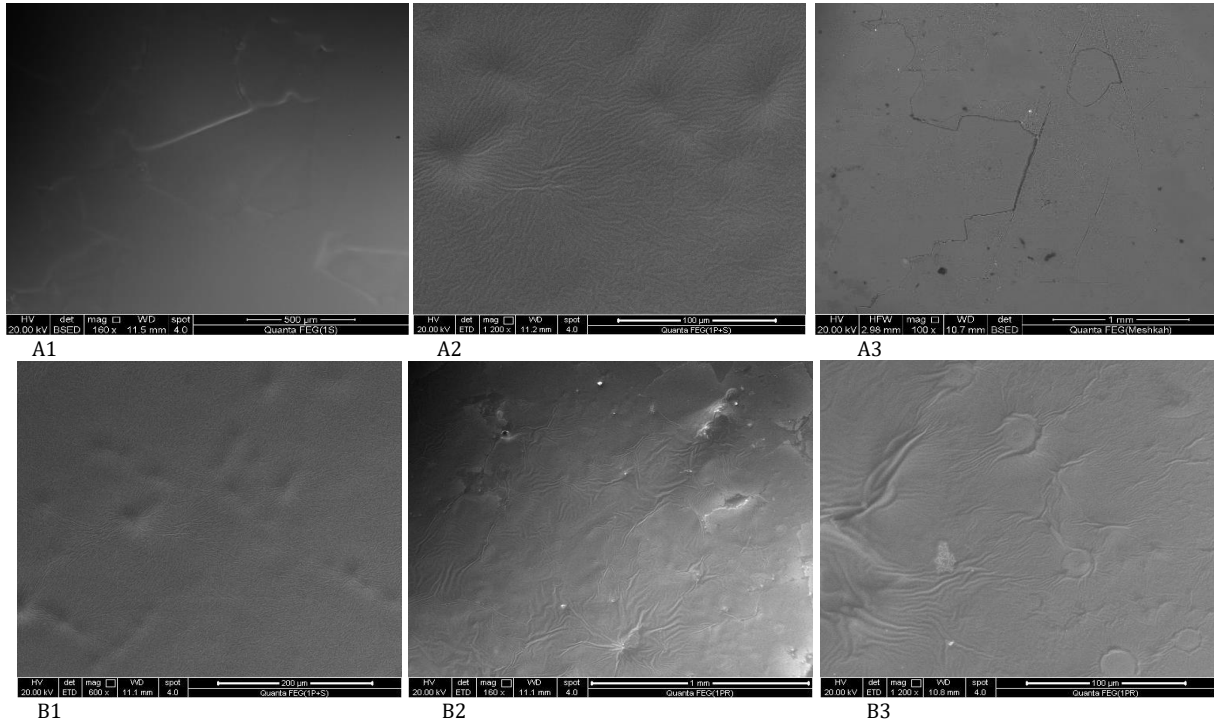
achieve swelling of Araldite. The longer time needed for ethyl lactate to swell the hydrophobic layers of Araldite could be interpreted from its amphiphilic nature due to the existence of O-H, C=O, and hydrocarbon functional groups [5]. The existence of O-H strong group could play a role in delaying the role of the C=O functional group in ethyl lactate to directly swell the coating. While dimethyl carbonate showed faster results due to the direct role of dipole-dipole bonding to the partial breakdown of the Epoxide groups in epoxy, which leads to the collapse of the three-dimensional cross-linking polymer network [36].



**Fig.4** USB Digital images; A1. Aged poxy on folds of the decorations on glazed surface, A2. After 30 minutes cleaning and removal by EL. B1. Aged epoxy on the glazed and unglazed surface, B2. After 30 minutes cleaning for both glazed and unglazed surfaces with EL.

### 3.3.2 Scanning Electron Microscope SEM

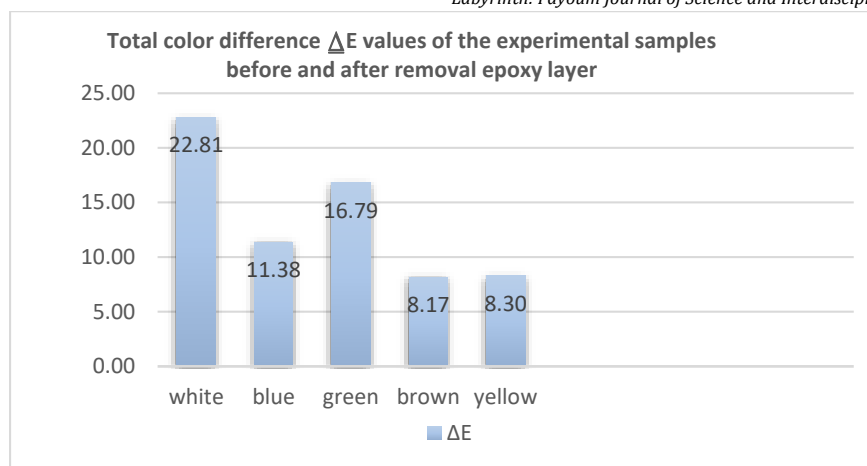
SEM images show the softening of the epoxy layer. The stages of removing the epoxy layers using dimethyl carbonate and ethyl lactate were clearly demonstrated through the SEM images, by standardizing the time period and the amount of solvent, the epoxy layer was removed completely and quickly when using dimethyl carbonate, while the remains of the epoxy layer appeared on the surface and some parts were covered of it. By comparing the average of the thin and thickness with the cleaned ones, they showed convergence in values referring to the cleaning effectiveness in DMC more than in EL. Mixing of aged epoxy, its covering of the surface, and gradual removal appeared at a faster rate with dimethyl carbonate as a result of its stronger effect in removing the aging epoxy surface (Fig. 5), which was one of the biggest challenges facing restorers in this specialty [37,38].



**Fig. 5.** SEM images A1. Glazed surface followed by 3mm araldite before cleaning, A2. After 6 minutes cleaning with DMC, A3. After 18 minutes cleaning with DMC, B1. Glazed surface followed by 3mm araldite before cleaning, B2. After 6 minutes cleaning with, A3. After 18 minutes cleaning with EL.

### 3.3.3. Colorimetric measurements

The total color difference  $\Delta E^*$  refers to the large color change before and after removal the aged epoxy layer from the glazed surface in experimental study. The color change after removal in all colors in glazed surface due to the effectiveness of DMC. The total color difference  $\Delta E^*$  below "3" refers to an insignificant color change to the naked eye [13-38]. In case, it records a value greater than (3), color change recorded (22.81 for white color which is the greatest value, 11.38 in blue color, 16.97 for green, 8.30 for yellow and finally the smallest value of color change was for brown (Fig. 6 - Table 4)



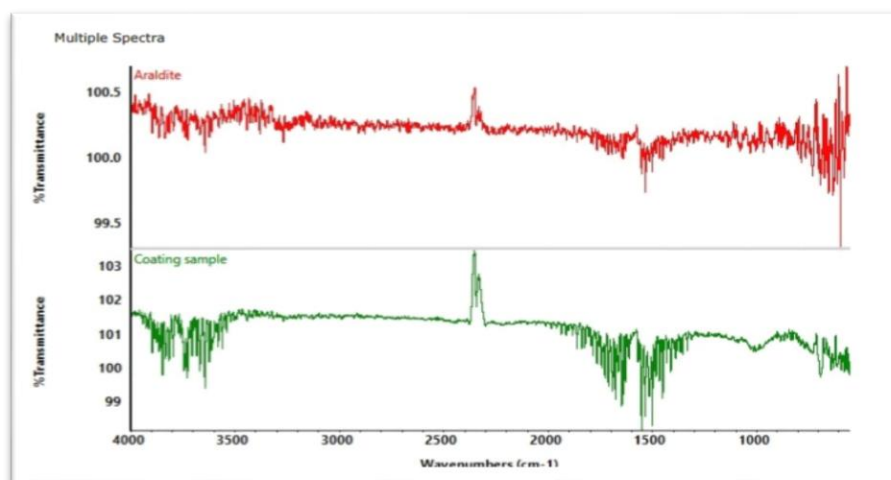
**Fig.6** Total color difference  $\Delta E$  values of the experimental samples before and after removal epoxy layer.

**Table. 4.**  $L^*$ ,  $a^*$ ,  $b^*$  and total color difference  $\Delta E$  values of the experimental samples before and after removing epoxy layer

SAMPLE NAME	$L^*$	$A^*$	$B^*$	$\Delta E$
WHITE	50.11	20.69	20.49	-----
	40.89	2.08	11.06	22.81
BLUE	38.44	3.74	11.51	-----
	48.08	-2.18	10.32	11.38
GREEN	62.11	12.06	15.54	-----
	65.83	-4.22	13.82	16.79
BROWN	27.76	11.38	21.34	-----
	25.74	14.32	28.69	8.17
YELLOW	55.97	9.67	35.24	-----
	63.37	11.37	31.88	8.30

### 3.3.4 Attenuated Total Reflection- Fourier Transform Infrared Spectroscopy (ATR- FTIR)

Attenuated Total Reflection- Fourier Transform Infrared Spectroscopy (ATR- FTIR) analysis was used to identify and analyze a sample of the material used to consolidate and support the surface of the applied study. A sample of aged epoxy "Araldite" was also analyzed, then functional groups of both samples were compared and the result indicated a near match between the two samples (the applied model - araldite), and there was only a slight decrease for the applied study sample in the OH 3300 band - 3400, C = O band 1650 - 1750, as well as a slight decrease in CH band 2800 - 3000 (Fig. 7). The band changes between increases and decreases due to oxidation during aging [39].



**Fig.7** FTIR spectra of coated layer from applied study and aged araldite 2020.

### 3.3.5. Case study cleaning

Dimethyl carbonate is the green solvent that was used to remove the epoxy layer "Araldite", which was identified through ATR-FTIR analysis. Removing of epoxy layer was done using dimethyl carbonate compresses, which softened the araldite, then using swaps to wipe and remove these layers. Cleaning process did not cause any damage to the surface of the glazing layer of applied study. This green, environmentally friendly solvent is



distinguished by its ease of application and its effective results in a short time, in addition to providing safety for the conservators and the artifact (Fig. 8).



**Fig. 8** A1. One side of the applied study with epoxy layer before cleaning, A2,3 and 4 during removal with DMC, A5 after cleaning. B1. The other side of vase before, B2&3 during removal epoxy layer by DMC, B4. After completely removing aged epoxy "araldite" by DMC.

#### 4. Conclusion

The research examines the efficacy of green solvents in removing aged epoxy from historical glazed surfaces. The extraction of weathered epoxy from the cemented glazed surface without compromising the integrity of the surface can be achieved utilizing the green solvents dimethyl carbonate and ethyl lactate. Dimethyl carbonate and ethyl lactate are non-toxic solvents that are environmentally benign and safe for conservators' health. Microscopic and analytical investigations have demonstrated that dimethyl carbonate is safe for cleaning ancient epoxy off glazed surfaces, regardless of thickness. The solvent's fast evaporation rate enabled it to effectively clean the glazed surface. Ethyl lactate was effective but inferior to DMC in the removal of old epoxy from glazed surfaces. Additional research and experimentation to enhance ethyl lactate with sustainable resources and methodologies.

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