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# PRELIMINARY STUDY OF A POLYCYANOACRYLATE ADHESIVE FOR GLASS: CHANGES UNDER AGEING CONDITIONS

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## ABSTRACT

Cyanoacrylate adhesives, although not widely used in substrates' maintenance compared to other polymeric materials, do find application for direct, instant welding. Their use is recommended against epoxies and acrylics in cases of easier application and faster polymerization. The objective of this study is the investigation of the commercial polycyanoacrylate adhesive of medium viscosity Loctite® Super Attack Glass (Henkel Germany) regarding its suitability for glass consolidation initially for a test glass plate. More specifically, the choice of the adhesive (glue) was based on the advantages it claims to offer, such as reversible results and the clarity that is maintained over time at the area of use. The aim of the research is to evaluate a) its functional groups, using the FTIR technique and b) the change of the colour or thermal behaviour of the specimen after its submission to conditions of accelerated ageing with UV irradiation, heat or pyrolysis. It was found that under usual ambient conditions the glue does not change its physical characteristics (elasticity, colour, transparency) while a certain deterioration is noticed when the glue meets abusive conditions. The UV irradiation provokes yellowing in short time, the thermal ageing is harmful at 75 °C after some days, while pyrolysis highlights some volatile by-products emerged by the adhesive when treated at high temperatures. The potential of using polycyanoacrylate adhesive as a restorative material for cultural heritage glass artifacts, direct welding under mild, controlled conditions is proposed in controlled environments.

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**KEYWORDS:** Loctite® Super Attak, glass restoration/conservation, UV-ageing, polymerization, adhesive, pyrolysis, FT-IR, welding

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## 1. INTRODUCTION

The special feature of polycyanoacrylate adhesives is their high activity coexisting with strong binding forces, especially in polar substrates. Due to their high relative molecular mass, polarization and good wetting properties, cyanoacrylate adhesives exhibit good penetrating properties (Fink, 2013). Their case is different from the rest, due to the presence of monofunctional monomers (vinyl bond) without special impurities in their mass. From the structural formula of the monomer, 2-cyanoacrylate esters belong to a wider group of vinyl monomers (Korde *et al.*, 2018). The most important feature of monomer chemistry is the electron density deficit in the double bond between  $\alpha$ - and  $\beta$ -carbon due to the influence of the strongly electronegative groups  $-\text{CN}$  and  $-\text{COOR}$ . This means an excess of positive charge  $+\delta$  locally on the  $\beta$ -C which becomes vulnerable to nucleophiles. The absence of substituents at  $\beta$ -C also enhances susceptibility to nucleophilic attack. In addition, electronegative groups of the molecule highly stabilize the resulting negative charge on the carbon after the nucleophilic attack

(through coordination structures). The combination of stereochemically unobstructed and electrophilic  $\beta$ -carbon on the one hand, and very stable carbonanion due to resonance on the other hand, makes the 2-cyanoacrylate molecule extremely active (Millet, 1986; Burns, 2018) at room temperature.

2-Cyanoacrylates can be polymerized by three mechanisms, anionic, zwitterionic and free-radicals polymerization, forming macromolecular chains through covalent bonds. These polymerization mechanisms lead to chain growth and are carried out quickly at room temperature. Anionic polymerization is the major type of polymerization for cyanoacrylate polymers. The initiators are nucleophiles, such as anions, weak bases (tertiary amines, tertiary phosphines), Lewis bases ( $\text{OH}^-$ ),  $\text{H}_2\text{O}$  and  $\text{ROH}$ . The nature of the polymerization depends on the substrate where the polymerization will be initiated. In general, the polymerization of a cyanoacrylate adhesive begins fairly quickly (fig. 1) due to the moisture of the substrate onto which it is applied. The polymerization starts at the adhesive-substrate interface and proceeds to the inside of the adhesive layer.

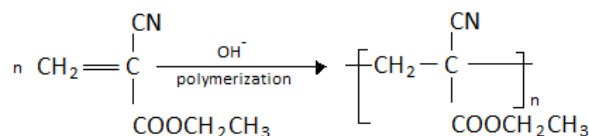


Figure 1. schematic reaction of poly(ethyl 2-cyanoacrylate) (PECA) production

The deterioration of the quality of the cultural glass, which is caused by various factors and results in the loss of its aesthetics, shape, structure or functionality, is faced through the restoration process. Plus, the conservators take in consideration rescue measures, the examination of the material and the types of corrosion, the cleaning and removal of the deposits, the fixing of the loose surface if any, the bonding of the various fragments that have been found, the filling of the missing areas, the aesthetic restoration (or exhibition). The conservation tactic of a glass object and the choice of suitable adhesives depend on the size of the fragments, their width, type and condition, etc. It is essential for the welding method, finally, followed to allow it to get disassembled if necessary.

The advantages of the produced adhesives are summarized to the following: application on most substrates, extremely fast curing at low pressures/temperatures, satisfactory strengths in tensile and shear forces, less risk of fire and low toxicity due to lack of solvents, easy application (glue of one component) (Cooke *et al.*, 1993), strong fresh welding (Korde *et al.*, 2018), colourless and transparent welding layer - clarity (Duffy *et al.*, 2018), no measurement or mixing is required during the application (Petrie,

2014). As it is assumed, this category of polymers adhesives should not be omitted from neither the scientific research and upgrade nor from the technical application.

On the opposite, as for the inconvenient of polycyanoacrylates, they may be the insufficient performance in exfoliation, impact and load deposition operations, the limited ability to fill relatively large gaps, the sensitivity to acidic or dry surfaces, the short service life (Cooke *et al.*, 1993), the brittleness (especially in thermal exposure) with low elasticity, the penetrating odour for some handlers (Korde *et al.*, 2018), the poor quality resistance to moisture and water, the low heat resistance (Duffy *et al.*, 2018), the solubility in polar solvents, especially in acetone (Petrie, 2014), the low viscosity that makes it difficult to apply accurately. The major reason behind the disadvantages is the lack of polymeric network in the structure of the adhesive, *ie.* the absence of crosslinks among the macrochains which would create a stiffer form.

The polymeric materials used in the context of the preservation of artworks must present structural stability since any alteration causes damage to the object. Polymers are applied with the expectation that they will retain their properties in long terms, but in prac-

tice the period of 20 years has been set as the minimum period of need for repeated maintenance (Alley, 1992). The functional life of a polymer used for the maintenance of artworks should be evaluated and determined prior to application of the maintenance material. Feller developed a method for defining criteria for categorizing maintenance materials according to their functionality over time, from Class A1 of very durable materials that retain their properties for more than 500 years to Class T for temporary application materials with durability less than 6 months (Feller, 1978; Feller, 1994).

The term corrosion defines any structural deterioration in the polymer. Some of the alterations that develop as a result of degradation of polymers are chromatic alteration, fragility, insolubility, shrinkage, material flow, bonding and retention of dust and dirt on the surface. These effects may be caused by various corrosive environments such as heat, photolysis and oxidation, air pollutants, hydrolysis or due to the dyes and the substrates with which it comes in direct contact. The research for new scientific methods and materials for effective and sustainable interventive and preventive conservation strategies is the main goal of conservators (Liritzis et al 2020).

Thermal degradation occurs at higher maintenance temperatures or due to an increase in surface temperature by direct exposure to the sun, resulting in the gradual decline of polymers. Therefore, the examination of polymers after exposure to high temperatures provides information based on the modification of the structure of the materials as a result of their heat absorption and subsequent corrosion.

Sunlight includes a wide range of wavelengths of electromagnetic radiation while artificial room lighting involves certain wavelengths. Photochemical alteration is the result of the absorption of a photon by a compound resulting in the breaking of a bond. Every chemical reaction that takes place in the polymer molecule requires a small amount of energy to break down the first chemical bond of the compound. UV-irradiation is heavily destructive for ester and cyanogroups causing radical reactions and oxidations (Pintus *et al*, 2011). Artificially, this phenomenon can be simulated in a short period of time in laboratory through the accelerated testing by using concentrated natural sunlight at high flux solar furnace and/or exposing the glass to the effect of both  $\beta$ - and  $\gamma$ -irradiation (Abd-Allah, 2009).

The main climate factors affecting polymers are solar radiation, temperature, humidity, oxygen and air pollutants. Ageing experiments are performed either in the environment, and are called natural ageing experiments, or in the laboratory in devices that simulate environmental conditions and are called artificial

ageing experiments (Abd-Allah, 2009). These experiments are always accelerating. Accelerated artificial ageing experiments have been performed for at least the last 80 years in order to detect, in a short period of time, the changes caused by materials due to natural ageing. The ability of these experiments to be performed in controlled and reproducible conditions makes them very important. Nevertheless, their use to predict the life of materials is still under study and development. The natural environment is quite complex, so the efforts for these experiments should at least simulate solar radiation, humidity and temperature.

Corrosion of a glass object can be defined as the degradation or deterioration caused by external (e.g. environmental conditions) and/or internal factors (e.g. glass chemical composition) leading to a partial or complete loss of its functionality, (micro-)structure and/or shape. The chemical processes associated with this deterioration are the result of the internal composition of the glass being attacked on a molecular level by external forces, principally involving water. The two primary mechanisms involved, namely de-alkalization, also referred to as leaching, and network dissolution (Zacharias et al. 2020, Palomar et al. 2012).

The purpose of this work was the study of the polymers' behaviour over ageing, by studying the preparation of polymer films of a commercial cyanoacrylate adhesive addressed for glass restoration, and in a later stage to treat them under deteriorative conditions to examine the endurance of the product. The two steps of ageing include heat and UV-rays, while the characterization techniques were FT-IR for the detection of functional groups' alteration and Py-GC/MS for the material fragments' identification. To our knowledge there is little available relevant work, thus it concerns the basic characterization. Since the adhesive is specialized on glass applications and concerns the rapid curing, it finds usefulness in restorations of glass artifacts of cultural heritage, too. Yet, no effort has been made on that category of glass in present study.

## 2. MATERIALS AND METHODS

### 2.1. Commercial adhesive studied

Loctite® Super Attak (3g, Henkel, Henkel Hellas SA, Athens) especially for glass, is a cyanoacrylate adhesive with ethyl cyanoacrylate (CAS# 7085-85-0) as the main monomer. It contains triethyl *o*-acetylacrylate ( $C_{14}H_{22}O_8$ ) as an inhibitor to suppress polymerization, indicated on the package and hydroquinone ( $C_6H_6O_2$ ) as an inhibitor to suppress free radical polymerization, listed on the escorting sheet. It is a colourless liquid with low viscosity and a slight odour in pure

form. It has a density  $d = 1.06 \text{ g/mL}$ , melting point at  $-22 \text{ }^\circ\text{C}$  and boiling point at  $54\text{--}56 \text{ }^\circ\text{C}$ . Cures quickly in the presence of moisture whereas it is soluble in acetone and other common solvents (TDS of Loctite® Super Attak, Henkel, *retrieved online*).

A glue quantity was transferred in a square polyester mold of  $2 \times 2 \times 0.7 \text{ cm}$  size placed between two glass plates (3mm thick) covered with PP sheets and gathered tightly with clamps, placed in horizontal position in cool and shady place for 4 days to polymerize thoroughly. After the careful detachment a colourless, transparent adhesive film was produced, the procedure was repeated as many times as needed to produce the films for the ageing ovens.

## 2.2 Ageing environments

Thermal ageing was performed at  $25 \pm 0.1$ ,  $50 \pm 0.1$  and  $75 \pm 1 \text{ }^\circ\text{C}$  ovens (Mettler, Thermoset, Gallenkamp ovens correspondingly) for 12, 7 and 5 weeks correspondingly. Two pieces of cured adhesive were placed in horizontal position in the oven and their FT-IR spectra were monitored at certain time intervals.

UV irradiation affected the glue samples when they were placed in the Gallenkamp chamber, on microscope type glass plates, under 6 lamps ( $UV_c = 254 \text{ nm}$  Osram lamps, 8W each) at 10 cm distance, for total time of 48 h. The observation of the sample under a chamber equipped with D65 lamp, at preset time intervals (for 48 h in fig. 2).



Figure 2. Neat (on the left) and UV-treated (on the right) films of Loctite® Super Attak

## 2.3 Characterization techniques

FT-IR recordings were obtained in a Spectrum One (Perkin Elmer, USA) spectrometer, equipped with v.5.3.1 software through ATR probe for total reflectance (ZnSe plate,  $45^\circ$ ). The film was placed on the plate, scanned 32 times at  $4000\text{--}700 \text{ cm}^{-1}$  range (resolution at  $4 \text{ cm}^{-1}$ ) and removed carefully to be placed back to ageing chamber.

Pyrolysis experiments were carried out at a QP2010 Ultra GC/MS (Shimadzu, Japan) equipped with a Multi-Shot Pyrolyzer EGA/PY-3030D (Frontier, Japan). Pyrolysis was executed at  $355 \text{ }^\circ\text{C}$  for 0.5 min, while injection temperature at  $200 \text{ }^\circ\text{C}$ . The GC column oven remained at  $50 \text{ }^\circ\text{C}$  for 1 min, heated until  $300 \text{ }^\circ\text{C}$  at  $20 \text{ }^\circ\text{C}/\text{min}$  and then held for 4 min. The column flow at  $0.7 \text{ mL}/\text{min}$ , the purge flow (He) at  $3 \text{ mL}/\text{min}$ , the split at 200. The MS parameters involve the ion source at  $200 \text{ }^\circ\text{C}$ , the detector voltage at 1.2 kV, the  $m/z$  range at 10-500 and the scan speed at 10,000. Tiny quantity of each specimen is required, transferred in cups (stainless steel, coated with molten  $\text{SiO}_2$ ). Mega 5HT capillary column was equipped (Italy) of  $30 \text{ m} \times 0.25 \text{ mm} \times 0.25 \mu\text{m}$  dimensions, non-polar covered with 95% dimethylsiloxane. The evaluation of the results is carried out through GC/MS Lab Solutions, v.2.71 (Shimadzu, Japan 2011) and "Search analysis program & Data" (NIST111.00, Shimadzu, 2011, Japan).

## 3. RESULTS AND DISCUSSION

Beginning with IR characterization, the most popular technique for conservation materials (Liritzis *et al.* 2020), the polymerized adhesive has shown the anticipated changes in initial spectrum absorptions compared to the liquid glue, as seen in Fig. 3, since the functional groups of the adhesive have undergone no great changes. The principal alteration regards the transformation of  $\text{C}=\text{C}$  to  $\text{C}-\text{C}$  while polymerizing, illustrated in the reduction of the peak at  $1617 \text{ cm}^{-1}$  ( $\text{C}=\text{C}$ ) corresponding to the conversion of monomers to polymer. In details, the evaluation of the IR spectrum for Loctite® is given in Table 1 (Yordanov *et al.*, 2011). The strong absorbance of  $1749 \text{ cm}^{-1}$  is attributed to ester  $\text{C}=\text{O}$ , sharp and strong peak, unchanged throughout polymerization, while the diminution of the peaks at  $2237 \text{ cm}^{-1}$  and  $3462 \text{ cm}^{-1}$  stands as outcome of the structural changes in the macrochains which become bigger and stiffer. The Tomlinson *et al.* claim that there are three areas with particular interest in the spectra of the ethyl cyanoacrylate:  $3500\text{--}2700$ ,  $2300\text{--}2200$  and  $1900\text{--}1500 \text{ cm}^{-1}$  (Tomlinson *et al.*, 2006). At  $3462 \text{ cm}^{-1}$  there is a peak of the stress vibration of the vinyl structures ( $=\text{CH}-$ ,  $=\text{CH}_2$ ) and between  $3000$  and  $2800 \text{ cm}^{-1}$  the stress vibrations (symmetrical and asymmetrical) of  $-\text{C}-\text{H}$  of methyl and methylene groups.

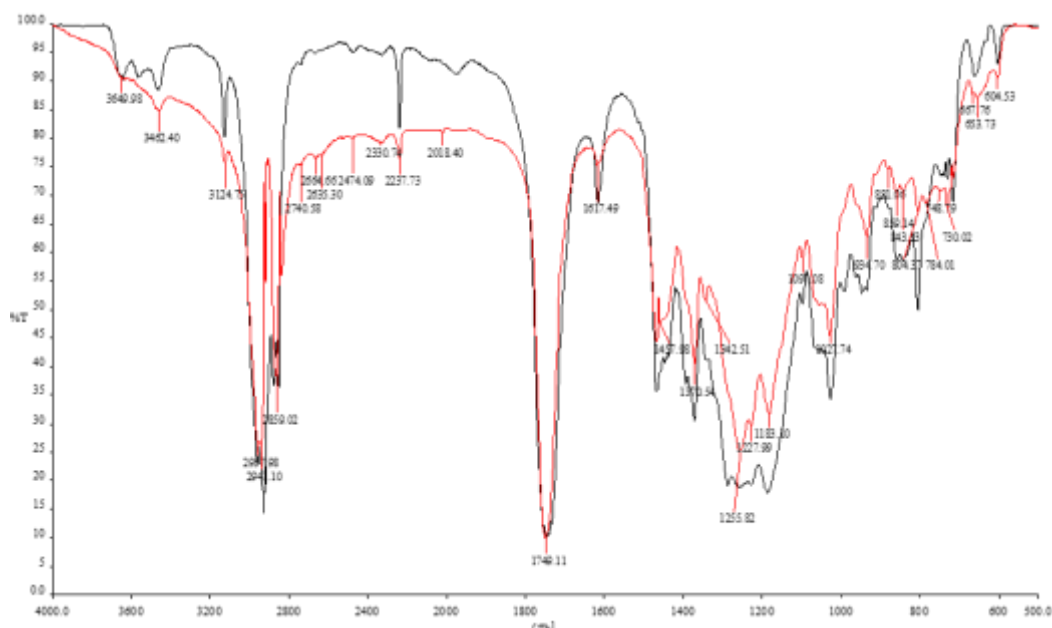


Figure 3. The FT-IR spectra of Loctite® adhesive in liquid form (black line) and film right after polymerization (red line)

Table I. IR interpretation of major spectra peaks

$\lambda$ (cm <sup>-1</sup> )	Peak shape	bond
3462	weak	=C-H (stretch)
2957-2859	medium, sharp	C-H (sp <sup>3</sup> , sym and asym stretch)
2237	weak	-C≡N (stretch)
1749	sharp, strong	>C=O (ester, stretch)
1617	sharp, strong	C=C (stretch)
1457 -1342	medium, sharp	-CH <sub>2</sub> - (scissoring) -CH <sub>3</sub> (asym bending) -CH <sub>3</sub> (sym bending)
1255, 1183	strong, sharp	C=O (ester, bending)
934	medium, wide	O-H
881	medium	C=C
859	medium	C-H
843	medium	C=C
804-667	weak	C-H

Next, polymerized adhesive films have been treated thermally. The effect of thermal ageing on adhesive films is noticed slightly in FT-IR spectra since a few changes are recorded in Fig.4 for the examination time of 5 weeks, as an example here. Notably, at 25 °C maintenance no great change is observed in FT-IR during 12 weeks of thermal aging. This is very important in the case where a glass work of art, restored and exhibited in the museum, where the prevailing temperature is on average 23 °C. At the 50 °C ageing, it is observed that the peaks in the area of the fingerprint after 24 days of thermal aging begin to get

wider. This is due either to the production of a larger quantity of these characteristic groups or to its overlap with new peaks, which of course is minimal. Also, a gradual shift of the -C≡N peak from 2237 cm<sup>-1</sup> to 2255 cm<sup>-1</sup> is noted, most likely from the hydrolysis reaction of the cyanogroups in an alkaline environment. As for intensive ageing at 75 °C, at 3400 cm<sup>-1</sup> region the baseline begins to curve from 30<sup>th</sup> day onwards. This is due to the formation of -OH from the hydrolysis of the ester bonds in an alkaline environment (Boytatzis *et al*, 2007).



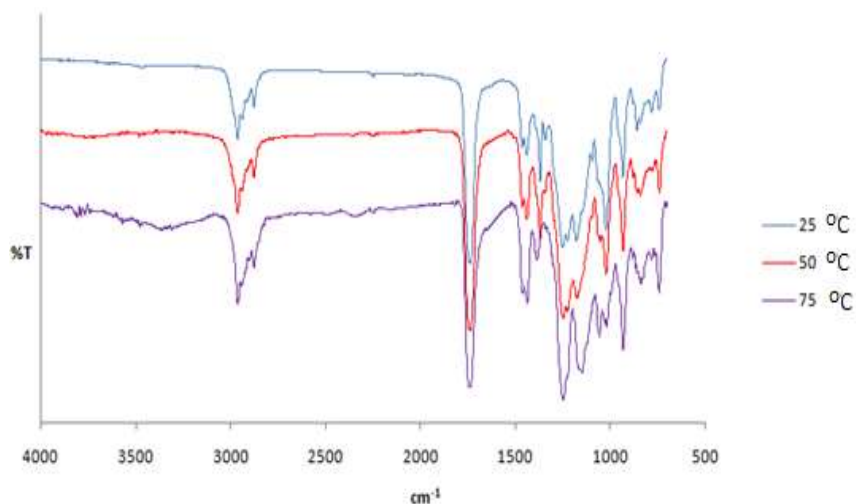


Figure 4. The FT-IR spectra of Loctite® adhesive after thermal ageing for 5 weeks period time.

Thermal aging at 25 °C was applied to study the thermal behaviour of the adhesive over time at room temperature, while at 50 °C and 75 °C to determine the thermal curing (post-curing and degradation occurring simultaneously) of the adhesive (Estan-Cerezo, 2019). Noting some visual observations, at 75 °C a faster evolution of ageing and yellowing took place. The samples at 25 °C after 11 weeks began to show elasticity while the samples kept at 50 °C after the first month of their thermal ageing began to soften once pressure was applied. During the 7<sup>th</sup> week of ageing the specimens began to acquire a yellowish hue and harden. Finally, the specimens of 75 °C after 10 days began to turn yellow and then showed brittleness, cracking, and gradually began to harden. We consider the visual observations and the handling differences noticed by the experimenters are of great importance, since the field technicians will cope with them during application.

Moving to the second treatment, it is generally known that the UV irradiation strongly affects the polymers which bear heteroatoms or unsaturated bonds in their molecule. The conclusion that emerges for the texture of the samples is that up to the period of 48 h under 254 nm the samples remain in good condition, without losing their elasticity and remain easy to handle. However, we observe that by increasing

the photooxidation time above 3 h the samples undergo some softening. Certainly, the main effect is the changing of the cyanoacrylate film from colourless to yellow (fig. 2). It is worth noting that even with the visually apparent oxidation changes of the polymer, the films remain transparent throughout the experimental process.

Fig.5 demonstrates the effect of UV irradiation on Loctite® polymer. The molecular changes observed after photooxidation of the polymer are not great, yet they include the partial conversion of carbonyl groups to carboxyl (oxidation of the ester) as the literature indicates, since 3459 cm<sup>-1</sup> peak is reinforced (-OH) (Suetaka, 1983; Tomlinson *et al.*, 2006; Faggi *et al.*, 2019; Quilez-Molina *et al.*, 2020). We are sure that further exhibition to UV rays would degrade more the polymeric structure, yet it was difficult to operate further due to the softening of the film and surface inhomogeneities occurring by time passing. Note that no absorption peak is moved through in wavelengths or disappears in 2 days-time UV exposure. While most publications examine the exposure to fluorescence and Xenon lamps of polymeric materials found in restoration of cultural heritage, there is interest in UV ageing too regarding on the degradation results it provokes (Domenech-Carbo *et al.*, 2008; Arbizzani *et al.*, 2004).

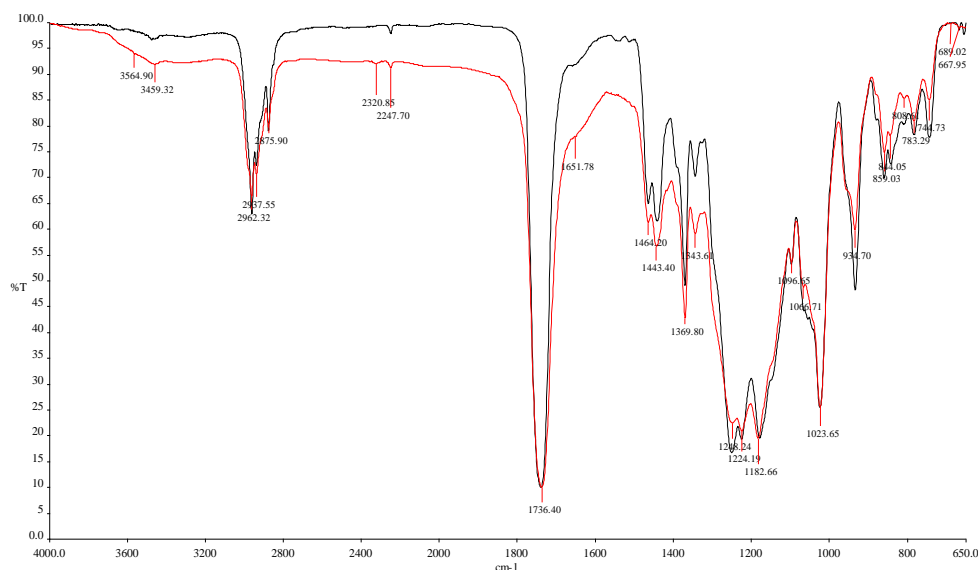


Figure 5. FT-IR spectra of adhesive untreated (black line) and photooxidized for 2 days exposed in UV (254 nm).

The third part of the study includes the pyrolysis of the polymer in some preselected temperatures where it was observed that the rate of the pyrolyzates' elution is greater. Py-GC/MS was applied for the case, using MS for mass spectra gathering (Liritzis et al. 2020). The temperatures of 190, 250, 300 and 420 °C were selected. From Fig.6a it can be concluded that the main fragments peaks appear at approximately the same elution time (corresponding to the same compounds, as seen when analyzed), with the volume of the fragments, especially in the 7-15 min elution area, increasing with temperature. Exception is the chromatogram at 420 °C where strong peaks are observed in the last minutes of the total time. Approximately the same compounds have been identified along the elution region and it is worth noting the detection of the MMA monomer in the first small peaks of the chromatogram at 420 °C. MMA is an ingredient claimed in literature to exist in polycyanoacrylic materials too, apart from ethyl cyanoacrylate monomer.

The diagrams record a parallel evolution in time in terms of crack intensity, which reveals some similarities in the behaviour of the two polymers (neat and

photooxidized) or in other words proves that the cyanoacrylate adhesive chemically resistant to the photooxidation executed. In general, the concentration of oxidized molecules, or those that have been fractured by ageing, is small compared to that of macromolecules in the mass of the material, unless it has been severely degraded. For this reason, the differences are small at the lowest pyrolysis temperatures especially at 190 °C. In contrast to chain macromolecules and the difficulty of observing their degradation, volatile small molecules are often as chain fission products and also molecules such as plasticizers, stabilizers, etc. migrate out of the polymer volume during ageing (Yu et al, 2013). This may explain the elution of more compounds from the raw material than perhaps that sample which was shot at 250 °C. The numerous smaller, identical peaks belong to compounds with C19 - C24. These compounds may have originated from polymer chain cleavage but may also be products of ageing.

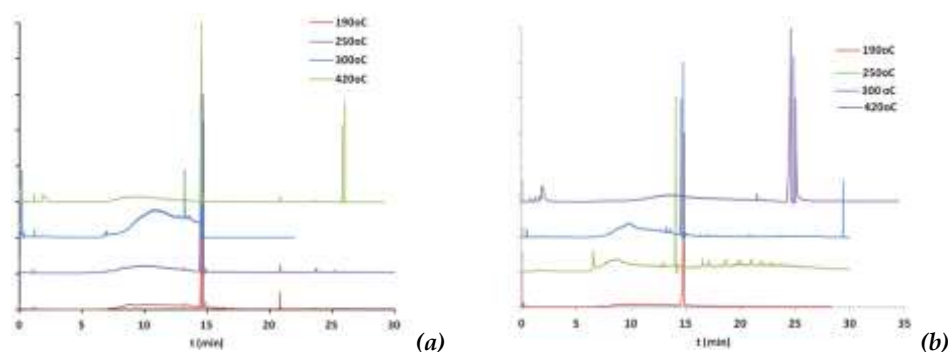


Figure 6. The chromatograms retrieved by pyrolyses of Loctite® untreated (a) and Loctite® UV-aged (b) at various pyrolysis temperatures.

As mentioned, in similar cases the products of photo-oxidation may be saturated alcohols, aldehydes, monocarboxylic acids and ketones (Cadd *et al.*, 2015). In the present study we have located as pyrolyzates alcohols, esters and aldehydes mainly, such as farnesol, 3-methyl-1-butanol, 1-butanol, 3-methyl-1-pentanol, 1-eicosanol, butyl formate, hexyl propanate, methyl methacrylate, ethyl cyanoacetate, 4-pentenal, ethanedial, 3-methyl-pentanal. Moreover, ingredients of the commercial adhesive have been noticed in chromatography, such as *o*-acetyl citric acid triethyl ester, diethyl adipate and triethyl citrate used as plasticizers, methyl methacrylate as thickener, phenol, 2-2'-methylenebis (6-(1,1-dimethylethyl))-4-methyl as stabilizer, dibenzo-18-crown-6-ether as accelerator or succinic anhydride as hardener.

When the degradation thermal profile is recorded (EGA), the UV-aged glue, yellow in colour, shows a deterioration of thermal resistance since its degradation starts 70 °C lower than the raw glue while the maximum cracking rate is illustrated around 20 °C earlier as well. As the cracking temperature increases Loctite® (with PECA as dominant ingredient) shows more decomposition products with greater diversity at 250 °C compared to the fragmentations at 300 and 420 °C. Not forget to note that the gaseous volatile products are CO, CO<sub>2</sub>, NH<sub>2</sub>NH<sub>2</sub> and slightly HCHO, common for fragments of the skeleton chain. The range of MB ranges from m/z 40-78 for the majority of fragments.

There are several glass types used in cultural artefacts, in a variety of chemical composition (Zacharias

*et al.* 2020). Yet, the glass plates used in this study was the microscope glass slides for laboratory use. This is a first step and eventually the present results shall be used for the study of the effect of aged polymer on glass composition. The characterization here concerned only the polymer's composition. The treatment periods were short anyway, assuming glass sherds do not present significant signs of corrosion (Zacharias *et al.* 2020) and no glass composition was made (covalent elements, oxides, silicates) (Hodgkinson *et al.* 2020, Hodgkinson *et al.* 2012, Palomar *et al.* 2012).

#### 4. CONCLUSION

The main conclusion is that Loctite® Super Attak adhesive, special for glass restoration/conservation, can be an auxiliary tool for the rapid bonding of glass surfaces in mild conditions where it retains its good behaviour (ambient conditions, internal room space). However, long-term exposure of the polymer to sunlight will damage the adhesive (yellowing, unwanted degradation) degrading the aesthetic and thermal (therefore mechanical) value of the joint, so it is not recommended to expose the preserved object to sunlight or great temperatures. Yet, for internal areas, room-conditioned exhibitions, its efficiency is well-secured. No relevant experiments have been conducted in this study regarding the safe period of time for Loctite® application, to be named, but the present preliminary results are useful for elaboration and application to material glass culture.

#### AUTHOR CONTRIBUTIONS

Conceptualization, D.A.; methodology, E.V.; software, P.M., E.C and G.T.; validation, P.M., E.C and G.T.; investigation, E.V., P.M., E.C and G.T.; data curation, E.V., P.M., E.C and G.T.; writing – original draft preparation, D.A. and E.V.; writing – review and editing, D.A. and E.V.; visualization, E.V.; supervision, D.A. All authors have read and agreed to the published version of the manuscript.

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