

Study on the Use of Polymers in the Treatment and Conservation of Historical Wood

Medhat, Abdel Rahman¹; Zidan, Yassien; El Hadidi, Nesrin M. N.

Conservation Department, Faculty of Archaeology, Cairo University

Abstract

Repair and rehabilitation of buildings has increased in Egypt during the past decades, therefore it has become essential to develop techniques for restoration of archaeological and historical timber. Current restoration techniques vary, but sometimes they involve total replacement of damaged timber by similar elements of new wood.

From three chosen polymers available in Egyptian markets two polymers were chosen for evaluation and were tested on old deteriorated wood taken from an old navigation lock gate. Samples were studied before and after heat and UV- ageing using FTIR spectroscopy and compression was measured.

Introduction

The use of timber in Egyptian buildings dates back to the Ancient Egyptian Dynasties, but it may not have been widely used due to the lack of timber in Egypt. During Pharaonic times (since ca. 3500 B.C) imported wood was used for royal furniture, coffins, boats etc... Local wood was used for all other purposes. [1]

When wood trade and import became prosperous in Egypt, timber became part of architectural buildings, especially in ceilings, stairs, domes, built-in cupboards etc... In past few decades the interest to restore and preserve these buildings has grown extensively, but many problems and mistakes are gradually showing up. This work arises from the urgent need to study and develop new techniques that could be used in the treatment and conservation of historical wood in Egypt.

The current restoration technique in many cases of historical structures involves total replacement of damaged timber by similar elements of new wood instead of treatment or conservation of deteriorated wood. Usually it is presumed that new "pitch pine" is the solution for replacing old deteriorated wood members. Often new wood is not identified anatomically, and the compatibility of its mechanical properties is rarely put into consideration. Materials and techniques applied in such cases are not published, and conservation reports may not be easily obtained.

This research aims to study the use of epoxies in the restoration of deteriorated wooden members; a method which we hope may be applicable on Egyptian sites. The use of epoxies on wood is restricted at the time being, even though in some cases it was used. [2] by putting guidelines for choosing and applying polymers on historical wood it could be a solution to many problems. It is a fact that the use of polymers for treatment and conservation of historical wood *in situ* may be difficult due to working conditions, especially during the summer months, but solutions could be found.

There are many uses of epoxy and polyurethane in archaeological wood that have been studied [3-7]; but the main focus in our study is their use as adhesives or gap fillers in Egypt, putting into consideration the least loss of historical material during present and future repairs.

Materials and Methods:

A- Materials

Old wood samples were chosen for treatment with polymers available in the Egyptian market. The mechanical and chemical properties of all polymer samples were compared for their evaluation after application.

¹ Corresponding author: email:abdelrahaman1010@yahoo.com

1- A decayed hardwood beam was taken from old navigation lock gates in the Nile delta for treatment evaluation. The wood had been in use on a daily bases for several decades and had been exposed to mechanical, physical and chemical deterioration factors. Under the light microscope we can clearly note the partial deterioration of vessel walls in both T.S. and L.S. sections. Features of the wood (fig. 1) indicate that the wood is probably teak wood *Tectona grandis*.

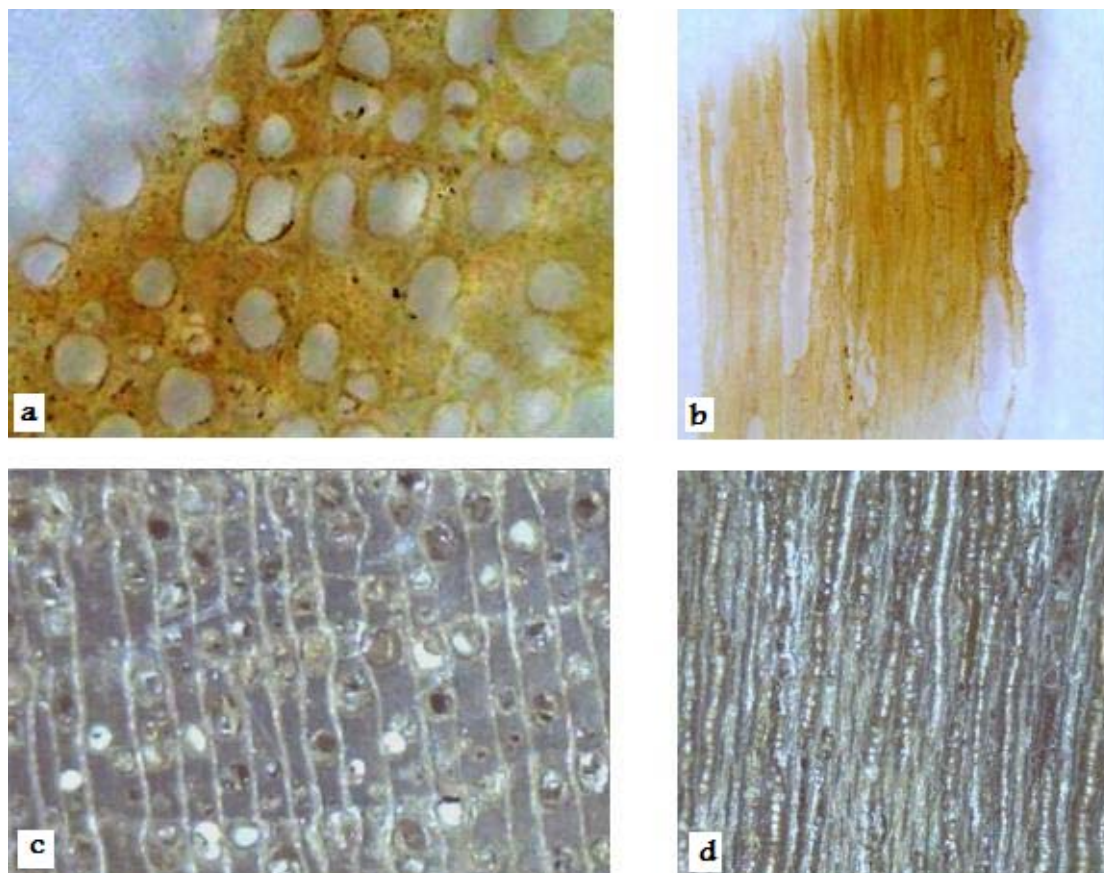


Fig. 1 Transverse (a & c) and longitudinal (b & d) sections of wood sample showing the decay of wood vessels in the wood taken from the naval lock gates. The features of the wood sample indicate that the wood is probably teak wood. (USB microscope, magnification 10x)

2- Polymers

Three polymers available in Egypt were chosen for evaluating their use for conservation purposes.

- Polymer (A) formulated from epoxy polyurethane which is free of solvents and has a high elasticity (KEMA POXY 175 manufactured by CMB Egypt)
- Polymer (B) consisting of diglycidyl ether bisphenol A [DGEBA] and polyamines (SIKA POXY CB 150 manufactured by Sika (non commercial))
- Polymer (C) consisting of diglycidyl ether bisphenol A [DGEBA] and polyamines + selected high strength fillers.(SIKA DUR PF manufactured by Sika)

The functional groups in the 3 chosen polymers were studied using FTIR spectroscopy.

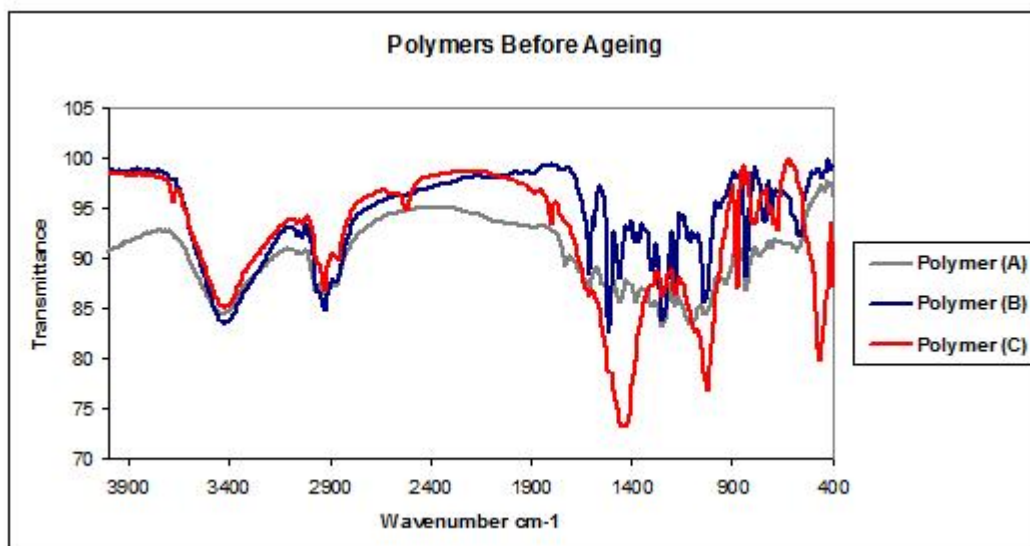


Fig. 2 FTIR spectra of the three polymers before ageing, showing the functional groups, which are present in each polymer.

From the FTIR spectra (fig. 2) it was possible to identify the main bands in the polymers.

Polymer (A) contained:

- OH stretching at 3422 cm^{-1}
- Aromatic CH stretching at 3034 cm^{-1}
- methyl ($-\text{CH}_3$) symmetrical at 2870 cm^{-1}
- Cyanate group at 2068 cm^{-1}
- C-O groups at 1726 cm^{-1}
- CH_2 bending at 1457 cm^{-1}
- CH_3 Umbrella vibration at 1376 cm^{-1}
- Epoxy and oxirane rings at 869 cm^{-1}
- The band at 828 cm^{-1} might be a Peroxide C-O-O stretching vibration, but is too weak to assign for certain
- The long chain band (4 or more aliphatic C atoms in a row C-C-C-C) could be seen at 720 cm^{-1} .

Polymer (B) contained:

- NH stretching vibration at 3795 cm^{-1}
- either C-H stretching vibration from aromatic H or simply combination or overtones at 3422 cm^{-1} & 3058 cm^{-1}
- aliphatic CH (more than one type) in the polymer at 3034 cm^{-1} and 2966 cm^{-1} and some kind of CH bending at 1475 & 1411 cm^{-1}
- aromatic vibration at 1833 cm^{-1} & 1726 cm^{-1}
- An aromatic ring vibration at 1511 cm^{-1} .
- Aromatic ring stretch C=C-C at 1609 cm^{-1}
- C-O group at 1324 cm^{-1} & 1269 cm^{-1}
- Silicate ion at 1038 cm^{-1} & 927 cm^{-1}
- Epoxy and oxirane group at 869 cm^{-1}
- The band at 828 cm^{-1} might be a Peroxide C-O-O stretching vibration, but is too weak to assign for certain

Polymer (C) contained:

- NH stretching at 3676 cm^{-1}
- OH stretching at 3421 cm^{-1}
- aromatic C-H stretching at 3061 cm^{-1} & 3033 cm^{-1}
- aliphatic CH stretching at 2923 cm^{-1} & 2854 cm^{-1}
- Aromatic ring vibration at 1609 cm^{-1}
- Aliphatic CH vibration at 1428 cm^{-1}

- Aliphatic CH vibrations (bending or combination vibrations) at 1247 cm^{-1} & 1181 cm^{-1} .
- C-O (ether) bands at 10801 & 1020 cm^{-1}
- C-H bending from the ring at 875 cm^{-1}
- The band at 828 cm^{-1} might be a Peroxide C-O-O stretching vibration, but is too weak to assign for certain

B - Methods

- 1- Sample cubes ($4 \times 4 \times 4\text{ cm}$) of all three polymers were prepared for ageing and testing according to ASTM C 365-94.[8]
- 2- A group of samples was exposed to UV Fadeometer, Philips, UK, 1980 according to ASTM G 23-89; and ASTM D-529 for 50 Hours without water spray to evaluate photo degradation. [9-10]
- 3- A second group of samples was exposed to complete thermal cycle at 23 and 78°C (Vestor, Ltd, UK, 2005).
- 4- Compression of the old wood was measured at the National Research Centre in Cairo. The dimensions of the wood samples were cubes ($5 \times 5 \times 5\text{ cm}$) according to ASTM D 143-94.[11]
- 5- Compression tests of 9 samples of all three polymers were measured, after 4 weeks curing, at 25°C , at the National Research Centre in Cairo (3 without any treatment, 3 after UV ageing and 3 after thermal cycle for each adhesive) using Instron 5500, UK, upgrading 2005, according to ASTM C365-94 [8], BC 6319-1986. [12]
- 6 – FTIR (qualitative) was used for monitoring the changes caused by the two ageing techniques. (FTIR spectrometer 6100 Jasco – Japan, using KBr Disc method). [13-15]
- 7- T_g of both polymer (B) and polymer (C) was measured using DSC-60 (Differential Scanning Calorimetry), Tasy, Japan at the Micro analytical Centre at Cairo University.

Results and Discussion:

After exposing the three polymers to either UV or heat, samples were taken for FTIR spectroscopy and results were compared to monitor changes (figures 3-7). The average of compression of the three polymers and old wood was measured for further evaluation of the polymers (fig.8). That was followed by measuring the T_g of polymers (B) and (C) after eliminating polymer (A) (Fig.9).

Polymer (A) before and after ageing:

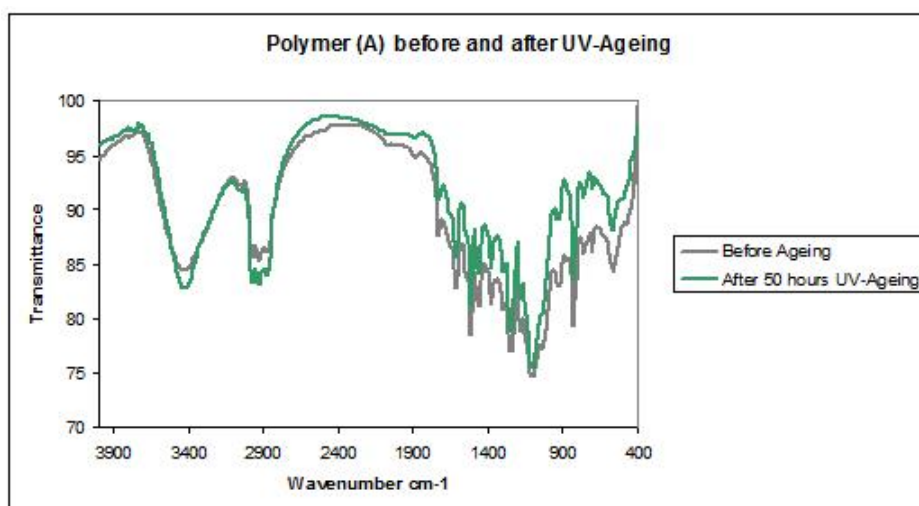


Fig. 3 FTIR spectra of polymer (A) before and after UV-ageing.

The OH-stretching region 3422 cm^{-1} in polymer (A) seems slightly affected by UV ageing, which may partially be due to excess water evaporating from the polymer. The aromatic peaks don't seem very affected by heat ageing or UV treatment and the interval area (1726 - 1182 cm^{-1}) changed - especially with UV treatment - but it is also reduced in relative intensity

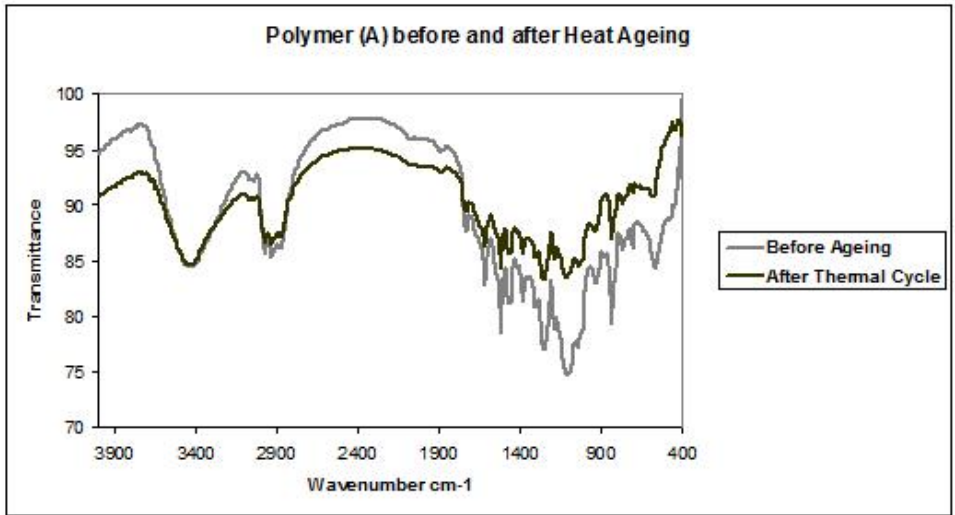


Fig. 4 FTIR spectra of polymer (A) before and after heat ageing, showing the slight changes in the sample after exposure to heat after complete thermal cycle

Polymer (B) before and after ageing:

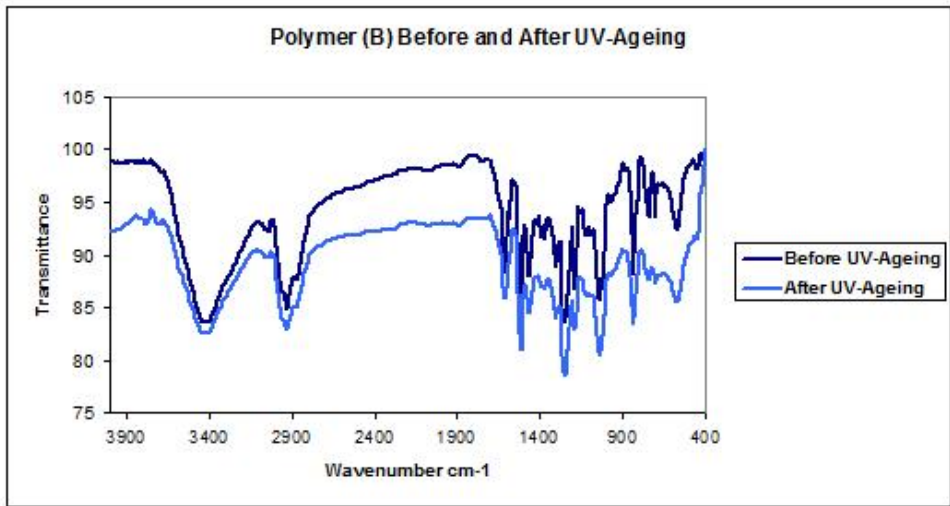


Fig. 5 FTIR spectra of polymer (B) before and after UV-ageing.

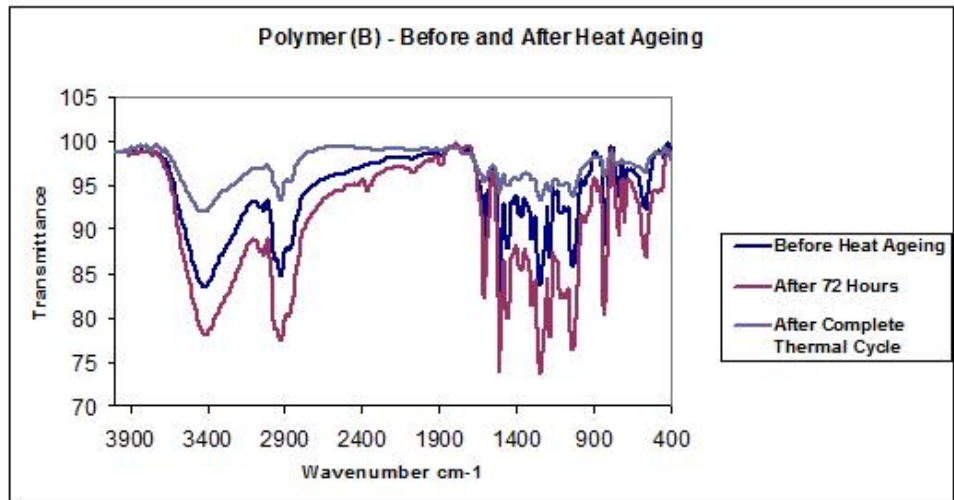


Fig. 6 FTIR spectra of polymer (B) before, during and after heat ageing, showing changes in the sample after exposure to heat during complete thermal cycle.

After exposing polymer (B) to partial thermal cycle and UV a reduction in the relative intensity is evident. (fig.5) After exposure to the complete thermal cycle weak bands, which are difficult to assign, are observed in the area between 3970 cm^{-1} to 3960 cm^{-1} . (fig.6)

Polymer (C) before and after ageing:

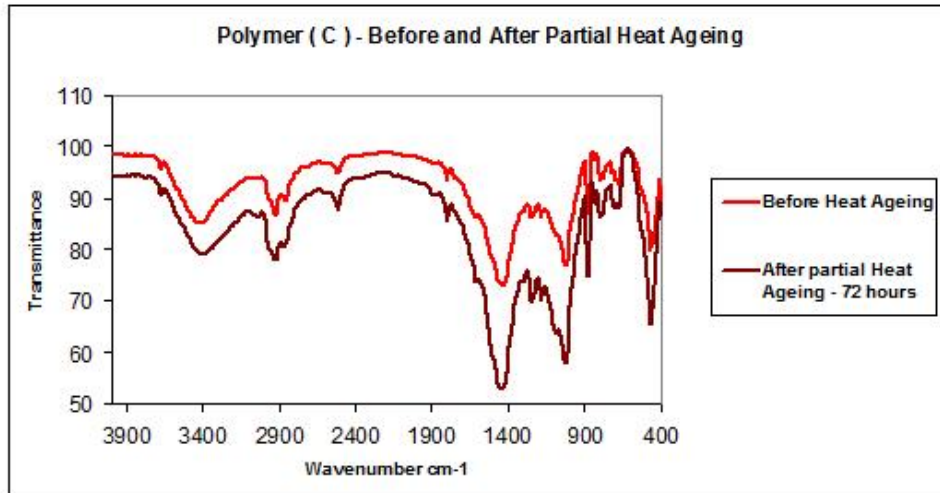


Fig. 7 FTIR spectra of polymer (C) before and after partial heat ageing, showing slight changes in the sample after exposure to heat.

Although the region from $800\text{--}1500\text{ cm}^{-1}$ in the spectrum of polymer C (see fig.7) seems to change during heating this effect could be primarily due to a different overall intensity of the two spectra. The band around 1200 cm^{-1} seems to get more intense (relatively) after ageing, which is usually the opposite of what we would expect.

Measurements of the average of compression of all polymer samples and old wood showed the following results:

- Polymer (A) and (B) had a high resistance towards the compression force, and polymer (C) had the least resistance, which was still higher than the compression strength of the old wood.
- The decrease of compression strength of the three polymers decreased to approximately 90% after exposure to a complete thermal cycle.
- The decrease of compression strength of the three polymers decreased to approximately 21% after exposure to UV. (fig. 8)

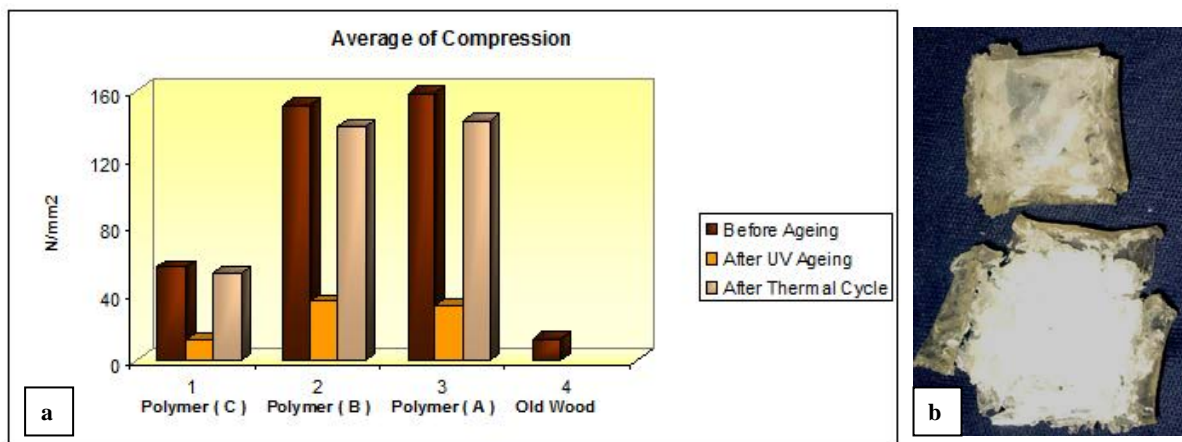


Fig. 8 a) Average of Compression of the old wood and three polymers before and after ageing
 b) Polymer (B) after thermal cycle and compression

Compression measurements were followed by measuring the T_g of both polymers (B) and (C) for further evaluation, and polymer (B) showed a higher T_g , which makes it more suitable for Egyptian hot weather (fig. 9)

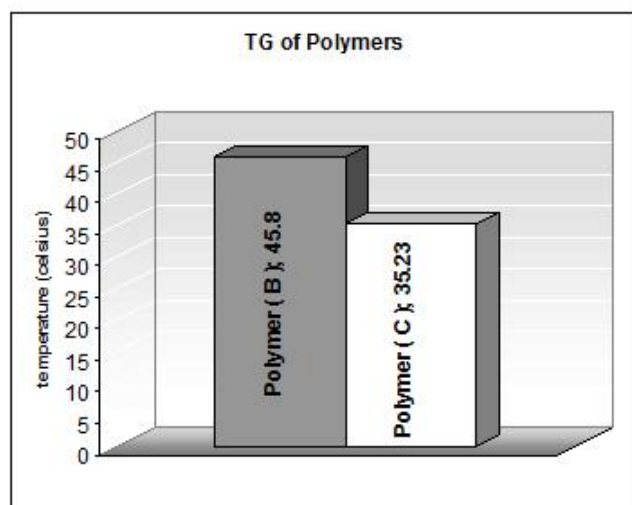


Fig. 9
 T_g of polymer (B) at 45.8 °C and polymer (C) at 35.23 °C.

Conclusion:

At this stage of research it may be difficult to decide the repair systems that will be applied in different cases. Further research should help estimate methods of ageing, testing, evaluation and treatment [16]. Handling properties and application of any polymer are yet another issue [17]. In some cases it may happen that the polymer may give good ageing – resistance results, and be compatible with old wood, but its application *in situ* may be difficult due to other properties. When choosing the three polymers for this research, different handling properties and curing time were considered, as a first stage. The chemical composition of the three polymers and their T_g were another point to consider, and can be explained as follows:

- Polymer (B) contains less oxygen than Polymer (C), thus it should be better able to withstand deterioration, and that was evident in the samples that were aged using UV. The presence of ethers as in all epoxies means that there is oxygen in the chain, which could be a reason for degradation. However, the large size of the molecule makes it unlikely that it will degas by itself.

- Both polymer B and polymer C contains ethers. Due to the higher T_g for polymer B, which is 45.8 degrees C, and the lower T_g of polymer C, which is 35.25 degrees C, we might guess that polymer B contains more aromatic groups than polymer C. The higher T_g makes polymer B more suitable for the relatively hot Egyptian climate, as it means that this polymer is unlikely to soften in the museum environment.

Compression strength of both decayed wood and modern polymers, were the guidelines of available mechanical properties. The compatibility of both wood and polymer are essential criteria for choosing restoration materials and methods. From our research we may presume that:

- 1- Polymer (A) is not suitable for use in conservation projects due to difficulty in handling and inappropriate physical properties for restoration and conservation purposes. Moreover, it is not a thixotropic material and has an unsuitable viscosity. After UV ageing it yellowed giving an unsuitable colour. It was therefore eliminated at an early stage of research.
- 2- Polymer (B) showed good results as a coating material and consolidant for aged wood samples, and that is due to its suitable viscosity and thermal behaviour.
- 3- Polymer (C) proved to be suitable for indoor use in Egyptian Museums due to its thermal behaviour.
- 4- Polymer (C) and polymer (B) need further research to evaluate their use as gap fillers.

Acknowledgments:

We would like to thank Dr. Helena Cruz, Dr. rer. nat. Uwe Noldt and Prof. Ing. Luca Uzielli for their encouragement during this research and their assistance that made it possible to participate in COST IE0601.

We are also indebted to Mikkel Christensen from the Museum for Cultural History, University of Oslo for his help in the identification of FTIR analysis.

REFERENCES

1. Davies, W. V. (1995): Ancient Egyptian timber imports. An analysis of wooden coffins in the British Museum. In Davies, W. V. and Schofield L. (eds) *Egypt, the Aegean and the Levant*, London, 146-156.
2. The Supreme Council of Antiquities (2002): " Samples of fine restoration works in the project" in *Historic Cairo; The Supreme Council of Antiquities*; 427-456.
3. Mettem, C.J & Davis, G. (1996) : Resin bonded repair systems for structural timber [part1] in *Construction Repair*; March/April .1996; 23-28.
4. Mettem, C.J & Davis, G.(1996): Resin bonded repair systems for structural timber [part2] in *Construction Repair*; May/June .1996; 43-47.
5. Graham, T. (2003-2004): Resin in bonded timber repair and the preservation of historic timber surfaces, Master degree, Bath University, Department of Architecture and Civil engineering. 1-33.
6. Maurin, E& Surleau, J. (2005): "Local reinforcement of the Structural using "Resin-Based" Methods" in *Conservation of historical wooden structures. Volume 2.*; 323-327
7. Musilová, Z., Mikeš, K.(2007): Rehabilitation of Timber Structure with Reinforced Epoxide Resin; Department of Steel and Timber Structures, Faculty of Civil Engineering, Czech Technical University in Prague, Thákurova;2007 ; 1-2.
8. ASTM C 365-94 (1994): "Standard Test Method for Flat wise Compressive Properties of Sandwich Cores"
9. ASTM G 23-89 (1989): "Standard Practice for Operating Light-Exposure Apparatus (Carbon-Arc Type) with and without Water for Exposure of nonmetallic Materials"
10. ASTM D-529 (1989) Practice for accelerated weathering test of Bituminous Materials"
11. ASTM D 143-94 (1994): "Standard Methods of Testing Small Clear Specimens of Timber"
12. BS 6319-1986 (1986): "British Standard, Testing of resin compositions for use in construction" part 2, Method for measurement of compressive strength.
13. El Hadidi, N.M.N & Darwish, S. (in press): "The use of acids and bases in cleaning archaeological wood" in *Giza Through the Ages Conference-2008* , Faculty of Archaeology - Cairo University
14. Darwish, S. & El Hadidi, N.M.N. (in press): "The Effect of Solvents on the Chemical Composition of Archaeological Wood" in *Giza Through the Ages Conference - 2008*, Faculty of Archaeology - Cairo University
15. Custódio, J. , Rodrigues, D., Rosa, A., Ferreria, J., Cruz, H. & Broughton, J.(2008): "Thermal behavior of epoxy and polyurethane adhesives and bonded timber joints- preliminary results", 1-11.
16. Cruz, H. & Machado, J. S. (2002): "Epoxy resins used for the repair of timber structures. The problem of short and long-term performance evaluation", *COST E34 – Sopron*, 1-7
17. Cestari, B.C., Toulaitos, P., Miltiadau, N.A., Dwlinskiolas, N., Mennichelli, G., Jamariz, J., Tsakanika, E., Pignatelli, O., Biligane, R.G. (2007): The Timber Roof at Hagia Paraskevi Balasika in Chalkida-Greece: Multi-disciplinary Methodological approaches for The Understanding of Structural Behavior Analysis and Diagnosis from material to structures *ICOMOS IWC-XVI International Symposium-Florence Vicenza 11th -16th November; 2007*, 1-30.