

# External Evaluation of Treated Historical Wooden Samples

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## Abstract

This study aims to identify suitable adhesives for the conservation and restoration of historical *Zelkova Carpinifolia* wood located in wet conditions. The Superficial properties and hardness of 14 compounds treated with several consolidants were compared. The consolidants have been applied alone, with synthetic resin or with protein glues and natural resins by the brushing method. Colorimetric measurements, observation methods and hardness tests were conducted before and after aging to verify the possible changes of the treated wood and the consolidating resistance. The compound 1:2 of Butvar B98 and sandarac in 5% ethanol was found to be more effective, providing a suitable compound compared to the other consolidants tested.

Keywords: *Zelkova Carpinifolia*, consolidation, synthetic resin, penetration depth, Hardness test

## Introduction

Traditional wooden architecture and native materials are becoming less favorable for modern architecture, especially in humid climates. As moisture is the main factor affecting the rate of wood deterioration, the consolidation of wooden fabrics in areas exposed to high levels of humidity has been a major concern for restorers. Although, there are many reports of significant methods for treating waterlogged wood [1, 2], any conservation literature virtually does not deal with the cases that are located in wet conditions.

During the mid-1980s, scientific testing of thermoplastic resins was conducted. In the following year, Barclay (1981) and Payton (1984) used polyvinyl butyral as a potential consolidant for ancient wood [3, 4]. Schniewind and Kronkright (1984) determined the effectiveness of polyvinyl butyral, and polyvinyl acetate types in strengthening deteriorated wood. Systematic tests were made on materials from

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Douglas-fir foundation piles that were buried for about 70 years. They found that acrylic (Paraloid B72) and polyvinyl butyral (Butvar B98) gave the greatest improvements in bending strength [5]. Also, a previous study by Wang and Schniewind (1985), yielded evidence of solvent plasticization using Butvar B98 and Paraloid B72 in the consolidation of Douglas fir [6]. In 1988, Grattan investigated the consolidation of deteriorated wood using a number of commercial thermoplastic resins and concluded that acrylics and polyvinyl butyrals were the most effective [7]. Tomoyasu and Schniewind (1990) used Butvar B98, Acryloid B72, and AYAT with both polar and nonpolar solvents for the consolidation of wood. They indicated Butvar B98 and Acryloid B72 are the most effective for strengthening resins in deteriorated wood [8]. Moreover, Crisci et al. investigated the properties of two polymeric products, Regalrez 1126 and Paraloid B72, and their potential synergic action on Norway spruce and white poplar wood species. They found that the treatment with Regalrez + Paraloid had a significant advantage in terms of mechanical resistance and pore size distribution [9]. Kučerová and Drncová (2009) evaluated the influence of three factors on the penetration of wood with Paraloid B-72. For the consolidation of wood, the optimal impregnation concentration of Paraloid B-72 solution in toluene was 10-20 wt. % (Weight percent), which represents the best balance between the solution concentration and the solution viscosity [10].

Some consolidants before synthetic resins have also been used in melted or in hot solvents; for instance, Sandarac [11, 12, 13], animal glue [14, 15], and drying oil and wax [16, 17, 18]. However, because of the humid climate of the project site, each of these methods has its own draw backs. Thus we conducted a comparative study to achieve desirable results.

When it is necessary to apply a consolidation technique to wood, the properties of both the consolidant and solvent are important. In addition, one of the most significant factors for effective strengthening is a good adhesion between the wood and the consolidant because some of the adhesives may not be suitable for the wooden objects that are located in humid situations. In this study, the influence of 14 traditional consolidants and their compounds with synthetic resins on the appearance properties and hardness of historical samples in wet conditions were investigated. Furthermore, experiments were conducted on consolidants commonly applied to dry or waterlogged archaeological woods to examine their efficacy in wet conditions.

## 2. Material

### 2.1. Wood Samples

The wooden samples used for the experiments were taken from wooden timber structures of *Zelkova Carpinifolia* which belong to the traditional houses exposed to humid weather for at least 100 years. These houses are located in the Saravan Eco museum in Gilan province, Iran, where the climate is very damp with over 85% relative humidity [19]. *Zelkova Carpinifolia* has a religious aspect and is cultivated basically near the tombs in the north of Iran, thus most of the decorative wooden objects that belong to the mausoleums of Gilan were made from *Zelkova Carpinifolia* wood. This valuable timber is prized for its characteristics and longevity, and



therefore the distribution of *Zelkova Carpinifolia* population has been considerably dimensioned activity in recent centuries [20].

### *Products used for wood treatment*

The treatment of wood samples was carried out using wax, animal glue, Sandarac and three types of synthetic resins generally considered suitable for degraded wood in wet conditions. These synthetic resins were acrylic (Paraloid B72, Paraloid B48 and Primal Ac33), a polyvinyl butyral (Butvar B98), and polyvinyl acetate (Mowilith 35/73 and Mowilith 20). Based on the combination of consolidants, many solvents were tried to find the best solvents for dissolving both natural and synthetic resin at room temperature. The solvents were of the polar type, such as thinner and ethanol, respectively used for Acrylics and polyvinyl acetates and polyvinyl butyral. Because Gilan is a humid territory where wood is not dry, the utilization of thinner for the consolidation of wet wood is not a risk. The nonpolar solvent (e.g. trichloroethylene) was used for microcrystal wax. This solvent was appropriate for the study, because the wax was dissolved in the other named solvents at room temperature. Thus the consolidant could not penetrate deeply [21]. In all cases, the concentration of the solution was kept constant at 5% and 10% on a weight basis [6].

## Methods

### *Wood characterization*

The wooden samples were cut into  $2.5 \times 2.5 \times 0.9$  cm dimensions to form test specimens according to ASTM specification D2017-81(1994) [22]. The specimens were conditioned to a relative humidity of 18% in a controlled temperature and humidity room. Based on the pH measurement carried out before the conservation treatment, the mean pH content of the samples was 5.5.

The wood specimens were marked based on the consolidants (Table 1). The samples treated with Microcrystal wax in 5% and 10% Trichloroethylene [23, 24] were marked with A1 and A2, respectively. Primal Ac33 in 5% water [23, 24] was marked with B. Mowilith 35/73, in 5% ethanol [7] was marked with C1 and the same

Table.1: Utilized samples, Solvents and impregnation solutions

No.	Resins	Solvent	Impregnation solution
A1	Microcrystal Wax	Trichloroethylene	5%
A2	Microcrystal Wax	Trichloroethylene	10%
B	Primal Ac33	Water	5%
C1	Mowilith 35/73	Ethanol	5%
C2	Mowilith 35/73	Ethanol	10%
C3	Mowilith 20	Ethanol	5%
C4	Mowilith 20	Ethanol	10%
C5	Animal glue+Mowilith 20	Ethanol	5%

D1	Butvar B98	Ethanol	5%
D2	Butvar B98	Ethanol	10%
D3	Animal glue+Butavr B98	Ethanol	5%
D4	Sandaracs+Butvar B98	Ethanol	5%
E	Paraloid B48	Thinner	5%
F	Paraloid B72	Thinner	5%

compound in 10% ethanol was marked with C2. Mowilith 20 in 5% ethanol was marked with C3 and the same compound in 10% ethanol was marked with C4. The constituent 2:1 of animal glue and Mowilith 20 in 5% ethanol was marked with C5. Butvar B98 in 5% and 10% ethanol [25] were marked with D1 and D2, respectively. The constituent 2:1 animal glue and Butvar B98 in 5% ethanol was marked with D3 and the compound 2:1 of Sandarac and Butvar B98 in 5% ethanol was marked with D4. Paraloid B48 in 5% thinner [2, 26] was marked with E and Paraloid B72 in 5% Thinner [2, 26] was marked with F.

### Technique of consolidation

Stabilization was carried out using the brushing method, as it has less harmful effects on the traditional wooden fabrics compared to, for example, consolidation by vacuum impregnation which may not be fully reversible. Moreover, this method is low cost and the application is easier when the conservation is applied in situ. Experiments were made on sets of specimens, using each of the named consolidants with the related solvents indicated in Table 1. Afterwards, the consolidants were applied to the surface of the samples with a soft brush; it is essential to stop the process before chalking (a white coat within resin layer) occurs to keep changes in the surface appearance at a minimum [3].

After conditioning, for accelerated aging, the specimens were put for a period of three weeks at  $26 \pm 1$  and  $70 \pm 4$  relative humidity based on ASTM specification D2017-81(1994) [22].

### Evaluation of the treatment

During the conservation treatment, the hardness of the surface of the samples was assessed. Moreover, macroscopic and microscopic evaluation on the shape and appearance of samples were added.

### Physical characterization

Hardness is the resistance of a material to permanent indentation. There are several different hardness tests that each may determine a different hardness value for the same piece of material. The hardness test uses a specifically shaped indenter, such as carbide ball, pressed into the material with a known force using a defined test procedure. The hardness values are determined by measuring either the depth of indenter penetration or the size of the resulting indent.



Hardness measurements were carried out on treated and untreated samples to evaluate the effect of stabilization on the hardness of historical wooden objects. Samples were put in a table model Instron testing machine, over a span of 11.5 mm/n and with a loading speed of 5mm per minute. The size of the indent was determined optically by measuring two diagonals of the round indent using either a portable microscope or one that is integrated with the load application device. The indentation was considered to be spherical with a radius equal to half the diameter of the ball. The average of the two diagonals was used to calculate the hardness of the historical samples.

### Macroscopic evaluation

All stabilized samples were photographed by Loup after the end of the whole consolidation and accelerated aging process in order to evaluate the changes in their appearance and shape (i.e. color, gloss and the growing of mold and fungi).

### Microscopic observation

The Cross-sectional surfaces of the treated samples were examined to assess the deposition of the consolidating substance into the wood cells. To get a better insight to the consolidants penetration, small fragments sectioned along the diagnostic direction of the wood were cut approximately below 1 mm from the transversal surface of the treated samples [27].

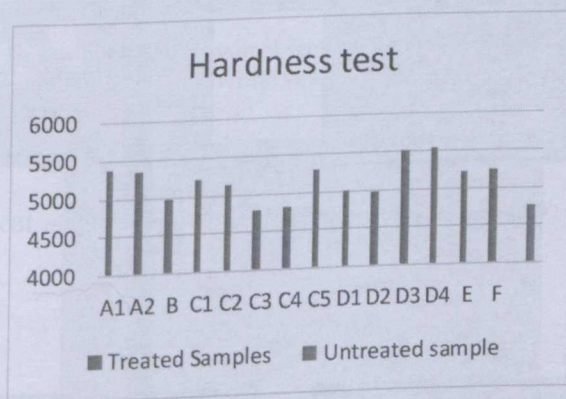


Figure 1: Hardness of the treated and untreated samples.

## Results and discussion

### Physical characterization

The results of the hardness test on the treated and untreated samples are shown in Fig. 1. The samples of C3 and C4 did not substantially differ from the untreated specimens. Similarly, D1, B, and D2 produced minor effects on the hardness of the samples. Consolidating with D3 and D4 showed substantial results, as the hardness of the wood increased considerably at a 5% concentration compared to those of the

untreated samples. However, all the consolidants had a considerable effect on the resistances of wooden cases.

### Macroscopic observation

The effect of each treatment on samples after consolidation and accelerated aging was evaluated through macroscopic observation. There were some changes in color for the several pieces of wood that were treated with Mowilith 35/73 and Mowilith 20 in ethanol (Fig. 2). Furthermore, gloss has been observed in the samples treated with Paraloid and Mowilith 35/73 (see Fig. 3). Treatment with polyvinyl acetate 5%, and polyvinyl butyral 5% and 10% in ethanol caused homogeneous penetration within the wood. Moreover, there was no change in color and no gloss in samples treated with Butvar B98 and sandarac in ethanol, and Mowilith 20 and animal glue in ethanol.

In the case of Wax, Paraloid B72 and B48, the surface was very hard. Also, the specimens that were treated with Butvar B98, Sandarac and Mowilith20, animal glue have shown sufficient hard textures; however, polyvinyl acetate5%, and polyvinyl butyral 5% and 10% in ethanol did not produce a very hard surface.

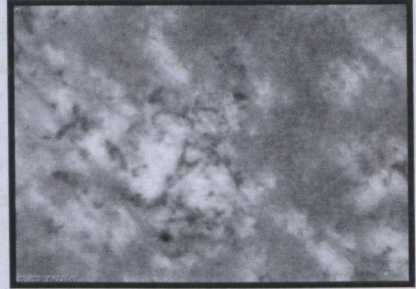
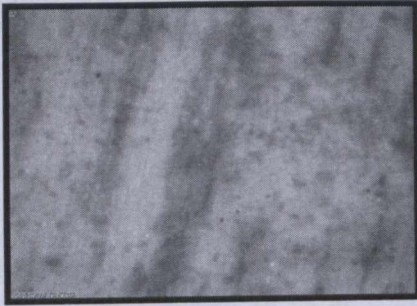


Figure 2: Changes in color of the samples treated with Mowilith 20 in 10% ethanol and Mowilith 35/73 in 5% Ethanol

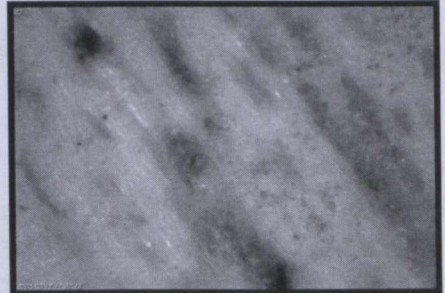
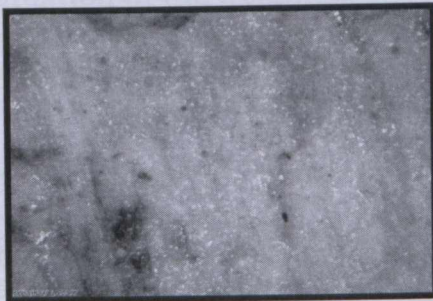


Figure 3: Gloss has been observed in the samples treated with Paraloid B72 and Mowilith 35/73 in 10% Ethanol



## Microscopic observation

For all the treated samples, the penetration of consolidant into the wood cells was evaluated. The results of the observation show that samples which were treated with animal glue and Mowilith 20, animal glue and Butvar B98, and Sandarac and Butvar B98 have higher diffusion; also cell wall and lumina appeared filled. The examination of the samples treated with Microcrystal wax highlighted that the penetration process of wood involved filling the inside of the fibers, while vessels lumina were still empty. However, the fact that some of the treated samples behave in a way very similar to untreated wood reflects the variation of environmental humidity (see Fig. 4-7).



Figure 4: Microscopic image 400X of transversal section of samples treated with Microcrystal wax. Fibers are filled by conservative products while vessels lumina are still empty

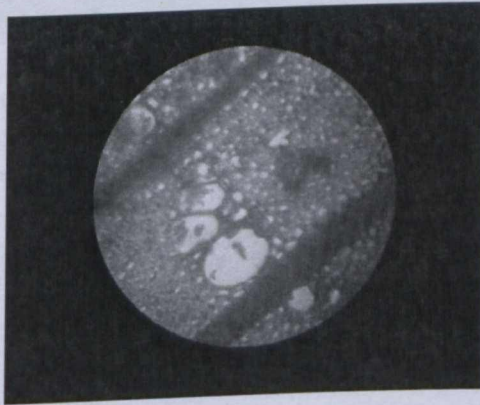


Figure 5: Microscopic image and 400X of transversal sections of samples treated with animal glue and Mowilith 20. Fibers and vessels lumina approximately appeared filled with consolidant.



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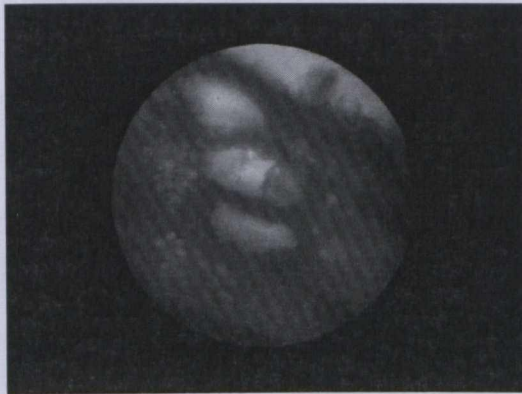


Figure 7: Microscopic image 400X of transversal sections of cases treated with Sandarac and Butvar B98. Fibers and vessels lumina appeared filled with consolidant.

## Conclusion

In this study, several consolidants have been tested, and some solvents selected to carry the consolidants into the wood to find the best materials to strengthen historical wooden objects that are located in wet conditions. Therefore, 14 groups of consolidants were investigated for their qualities and compared to each other. The compound 1:2 of Butvar B98 and sandarac in 5% ethanol, the constituent of Mowilith 20 and animal glue in 5% ethanol with the ratio of 1:2, and constituent 2:1 animal glue and Butvar B98 in 5% ethanol were found to be among the most suitable for the consolidation of wet wood. They produce sufficient consistency in the wood, while they can be removed with the same solvent. Furthermore, no marked change in color has been observed in the samples treated with these consolidants. However, consolidation with Microcrystal wax in solvent was effective as well, providing a very consistent consolidation and strengthened structure of the wood.



Based on the results of this study, the consolidation treatment of deteriorated wood with the compound of Butvar and Sandarac with the ratio of 1:2 in 5% ethanol is very effective. This is because while providing acceptable penetration no change in the appearance of the wood and no change in the color have been observed; also, the hardness of the samples that were treated with this compound is higher than in other samples.

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