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# STUDIES UPON PENETRATION OF PARALOID B72 INTO POPLAR WOOD BY COLD IMMERSION TREATMENTS

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***Abstract:*** *This work is an experimental study on the penetration of the synthetic resin Paraloid B72 into poplar wood. Paraloid B72 was prepared as diluted consolidation solutions (5% and 10%) in two solvents: a mixture of ethanol and acetone (1/1, v/v) and toluene. Sound poplar wooden samples (10x10x15) mm were employed to have a homogenous material. These samples were treated by long time immersion at room temperature. The consolidant retention in wood led to a weight percent gain WPG % up to 5% from the initial weight. The presence of consolidants into wood was also demonstrated by SEM microscopy. The maximum amount of consolidant was retained in wood when using Paraloid B72 10% in ethanol/acetone.*

***Key words:*** *Paraloid B72, wood conservation, weight percent gain, immersion treatment.*

## 1. Introduction

Most often old wooden objects present evidence of biological or chemical degradation which seriously affect their structural integrity and consequently reduce the mechanical resistance of the wooden material. The aim of a consolidation treatment of wooden objects is to render their mechanical strength and cohesion back without depleting their authenticity [10-12], [7], [5]. A consolidation treatment is therefore essential for the conservation of old historical wooden artefacts. As a principle, a solid polymer with adequate physical, mechanical and durability properties should be introduced into the degraded wood structure so as to fill, at least partially, the "extra-porosity" resulted from degradation. Diluted solutions of polymers, such as Paraloid B72, are most often employed, the process of consolidation having two phases: an "impregnation" phase when the solution of polymer is introduced into the wooden element by different methods (injection, immersion, vacuum treatment) followed by a conditioning phase when the solid polymer is fixed within the wooden structure. More solid consolidant is retained into the wood, the mechanical strength and the inner cohesion of the treated element are expected to be higher, which means an efficient consolidation.

Paraloid B72 is a well-known and studied consolidation material and has been extensively used in the consolidation of degraded wooden artefacts [2-4], [6],

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[13]. This synthetic polymer is composed of two monomers, methyl acrylate and ethyl metacrylate and has a low molecular weight. Based upon relative stability and reversibility (important criteria in the conservation science), this product found a particularly wide range of uses, as a coating, as an adhesive and mainly as a consolidant of porous materials, including wood.

The amount of solid consolidant retained into the wooden structure depends on several factors such as: the treating procedure and treating parameters, the composition and concentration of the consolidant solutions, the wooden species and the conservation state of the wooden support. An optimisation of a practical treating procedure in terms of solution uptake and solid consolidant retention is therefore essential for an efficient consolidation treatment. Moreover, a deep and uniform distribution of the solid consolidant, which depends essentially on the pathways of consolidant penetration are important.

The present study focuses upon the penetration and retention of Paraloid B72 into poplar wood, a widely used species in the history of art. The aim of this research was to optimise the process of consolidation by total immersion in terms of solid consolidant retention as a function of solutions concentration, type of solvent, duration of treatment. Small test samples of sound poplar wood (*Populus tremula*) were employed in order to have a homogeneous material, but higher values of consolidant retention are expected for old, damaged poplar wood.

## 2. Material and Methods

### 2.1. Synthetic Resin Consolidants

In this research, Paraloid B72 (from CTS Romania) was employed. Four different consolidant solutions ($C_1$, $C_2$, $C_3$, $C_4$) were prepared at two concentrations and in two different solvents accordingly: solution of Paraloid B72 5% in toluene, coded $C_1$; solution of Paraloid B72 10% toluene, coded $C_2$; solution of Paraloid B72 5% in ethanol and acetone 1/1 mixture, coded $C_3$ and finally a solution of Paraloid B72 10% in ethanol and acetone 1/1 mixture coded $C_4$.

The two solvents were chosen to test their influence upon the efficiency of the consolidation treatment. The mixture ethanol/acetone is a polar solvent and thus is compatible to wooden supports, which would mean higher solution uptake and consolidant retention, while toluene, an aromatic non-polar solvent (frequently used in conservation practice), would theoretically lead to a better penetration of the consolidant solution [6], [8]. A high retention and a deep penetration and uniform distribution of the consolidant into the wooden support are actual goals of consolidation treatments.

### 2.2. Wooden Samples

Specimens of sound poplar (*Populus tremula*) were used in this study due to its great importance in the history of art, poplar wooden panels being used in the past as supports for paintings and oil gilding [9]. On the other hand, the structural properties of poplar, like its quite homogenous structure (diffuse porous) and relatively good permeability, made it an ideal material in this research whose main objective was to achieve maximum consolidant retention in the wooden structure. Last but not least, the susceptibility of poplar wood to microbiological attack, often posing serious problems of loss of structural integrity, cohesion and mechanical resistance, were several other aspects considered in the current research, its natural low durability having a direct influence upon the faith of artefacts.

The specimens, with the dimensions 10x10x15 mm (longitudinal x tangential x radial) were sanded on all surfaces with H 240 grit size sandpaper and conditioned at 20 °C and RH = 55% until constant weight, these being the ambient conditions wooden objects are usually kept in.

### 2.3. Consolidation Treatment

Consolidation treatments with thermoplastic resin by total immersion at room temperature, also known as cold immersion treatments, are the most used treatments in conservation practice due to their ease of use, simple and rapid technique, and final potential properties they render to the object: partial reversibility, an important property in conservation practice [12], improved mechanical strength and physical properties.

The conditioned samples were weighed to register their initial mass ($m_i$), treated by total immersion for different periods of time (1 h, 2 h, 24 h) in the prepared consolidant solutions ($C_1$, $C_2$, $C_3$, $C_4$). After the immersion treatment, the samples were re-weighed ($m_u$). For each treatment a batch of 5 wood test pieces was used. During the immersion treatments the samples were fixed in a specially manufactured device of stainless steel to prevent them from floating and to ensure constant treating conditions, permanent liquid contact on all the surfaces (Figure 1). The samples were kept during the entire treatments in airtight boxes to refrain solvent evaporation.

After treatment, the samples were kept in the laboratory for 48 h, hanged on a special support to avoid consolidant loss and allow solvent evaporation. After this period, the samples were reconditioned at the same parameters ($T$ = 20 °C and $RH$ = 55%) until constant weight, the final weight after treatment being recorded ($m_f$).

Based on this data, the solution uptake $Csp$ [Kg/m$^3$] and the weight percent gain

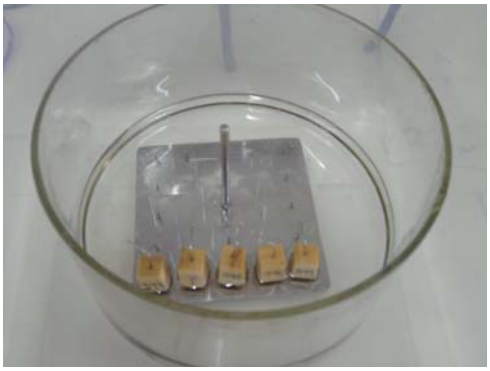


Fig. 1. *Immersion treatments: the wooden samples fixed into the device*

WPG [%] of the test samples, as a result of consolidant retention were computed as follows:

$$Csp = 10^{-3}\,\frac{(m_u - m_i)}{Volume_{sample}}\ [\mathrm{Kg/m^3}], \qquad (1)$$

where: $Csp$ - consolidant solution uptake, in [Kg/m$^3$]; $m_u$ - mass of the treated sample immediately after immersion, in [g]; $m_i$ - initial mass of the sample, in [g];

$$WPG = 100\,\frac{(m_f - m_i)}{m_i}\ [\%], \qquad (2)$$

where: $WPG$ - weight percent gain, in [%]; $m_f$ - final mass of the treated and conditioned sample, in [g]; $m_i$ - initial mass of the sample, in [g].

### 2.4. Microscopic Investigation

The presence and the distribution of the consolidant into the wooden support were investigated by Scanning Electron Microscopy (SEM). For the SEM observations, small examination samples were prepared as follows. The treated samples were first totally immersed into distilled water for two days, than the transversal face was straightened by a

microtome and small pieces of approximately (3x3x3) mm on the three fundamental directions (longitudinal x radial x tangential) were cut. These small samples were dehydrated by gradual drying in an air circulating laboratory oven and mounted on metal bases. Finally, the mounted samples were gold coated under vacuum and kept in proper storage conditions for further SEM examination. The microscopic investigation was performed on a SEM microscope model VEGA II LSH.

## 3. Results and Discussions

### 3.1. Sample Experimental Testing

The treatment of poplar specimens by total immersion in the Paraloid B72 solutions $C_1$, $C_2$, $C_3$, and $C_4$ resulted in solution absorption (uptake) expressed as *Csp* [Kg/m$^3$] which increased as a function of the treatment duration (Figure 2).

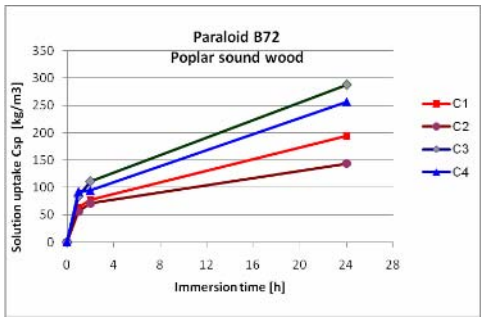



Fig. 2. *Comparative solution uptake of poplar samples treated by total immersion in the four consolidants $C_1$, $C_2$, $C_3$ and $C_4$ of Paraloid B72*

It is obvious from the shape of these curves, that, as expected, the absorption was higher at the beginning of the treatment (around one third from the total amount of solution absorbed in 24 h, being actually absorbed in 2 h) and then proceeded slower. The curves in Figure 2 also demonstrate that both the type of solvent and the concentration of solution will influence the solution uptake.

As concentration is concerned, a higher absorption can be achieved with more diluted solutions, the curves for $C_1$ and $C_3$, representing solutions of 5% concentration, being on top of the corresponding curves $C_2$ and $C_4$ representing solutions of 10% concentration.

When comparing the absorbtion curves of Paraloid B72 solutions of similar concentration but with different solvents ($C_1$ to $C_3$ and $C_2$ to $C_4$) it can be seen that the solutions in ethanol/acetone mixture are better absorbed than the corresponding solutions in toluene. This confirms previous research results [8].

In a consolidation process the impregnation phase is followed by a conditioning phase, during which the solvent will evaporate and the solid consolidant will be fixed into the wood structure rendering it a weight increase accompanied by strength and cohesion improvement. The actual values that reflect the retention of the solid consolidant into the wooden structure and could be, therefore, correlated to the actual expected consolidation effects are the weight percent gain WPG [%] values (Figure 3).

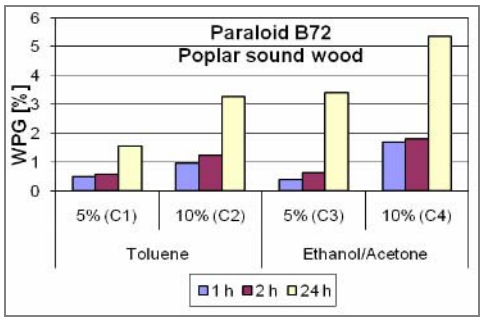



Fig. 3. *Comparative data of the weight percent gain of poplar samples treated by total immersion in the four consolidants $C_1$, $C_2$, $C_3$ and $C_4$ of Paraloid B72*

It is perfectly expectable that these values will depend not only on the solution uptake but also on the concentration of the treating solutions. With respect to this, concentration acts in two ways: a lower concentration is in favour of higher solution absorption but at the same time a higher concentration of the treating solution will ensure a higher amount of solid consolidant.

The column graph in Figure 3 outlines the cumulative influence of the treating time, concentration of the solution and solvent type on the final WPG values. The lowest WPG values were noticed in the case of Paraloid B72 5% in toluene ($C_1$), values between 0.5-1.56%, whilst the highest retention was obtained when treating with Paraloid B72 10% in a mixture of ethanol and acetone 1/1 ($C_4$), where the values ranged between 1.7% and 5.34%.

An interesting aspect could be noticed when comparing the WPG values resulting from treatments with the solutions $C_2$ and $C_3$, for which very similar values were obtained: 3.26% versus 3.39% (Figure 4). More explicitly, at the same treating time, 24 hours, the same retention of consolidant Paraloid B72 could be achieved either using a solution of 10% in toluene or a solution of 5% in ethanol/acetone, a solvent which is better absorbed into the wood structure. This is again a cumulative effect of solvent type and concentration of solution, which also results from the curves in Figure 4 referring to the in-time evolution of the consolidant retention (as WPG) as a function of the solvent type and concentration of the treating solutions.

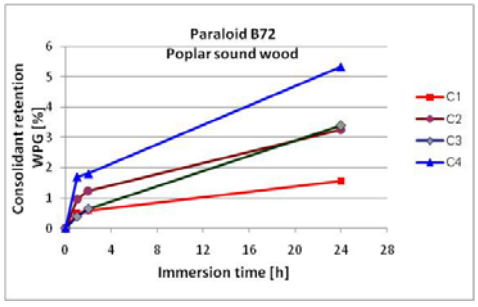



Fig. 4. *Efficiency of the immersion treatment in terms of maximum solution retention with respect to treating time, solution concentration and solvent type*

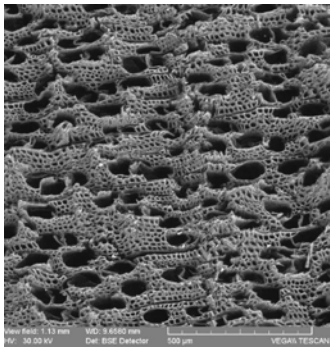
It is interesting to compare these curves with the similar ones from Figure 2 in order to see how they were reversed due to the influence of concentration of the solution on the actual retention.

### 3.2. Microscopic Characterization

A microscopic analysis was performed in order to assess the efficiency of the consolidation treatments in terms of consolidant presence into the wooden structure as a direct and visible proof of the consolidant retention. The investigation was undertaken by comparison between a control group of untreated poplar samples examined in all three grain directions (Figure 5) and samples treated with Paraloid B72 solutions (a sample treated with solution $C_4$ is presented in Figure 6).

An expected effect of the consolidation treatment is the filling of voids so that this particular aspect was relevant in the microscopic characterisation of the efficiency of consolidation treatments. The investigated sections (Figure 6) outlined the path ways of the consolidant penetration through the wooden structure. The main anatomical elements involved in the penetration of consolidants are the vessels and the rays, as well as the interconnecting pits, observations confirmed by previous research regarding the distribution of consolidants [6], [8].

The presence of the consolidant could be evidentiated for all the treatments, irrespective of the treating time, concentration of the solution and solvent type, though the amount of consolidant detected obviously varied as a function of these influencing factors.



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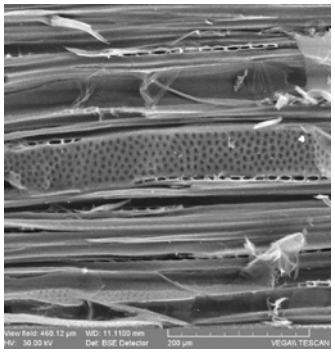

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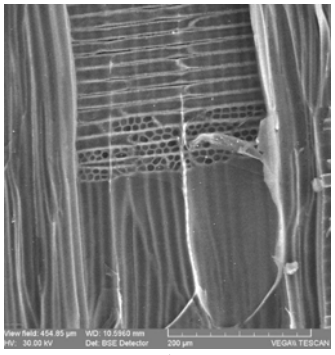
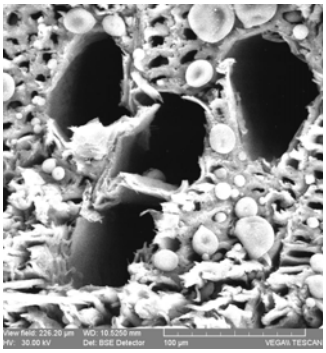


c)

Fig. 5. *SEM micrographs of control* (*untreated*) *poplar samples, magnification 500X: a*) *cross-section; b*) *tangential section; c*) *radial section*



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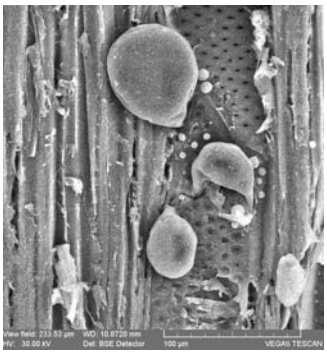

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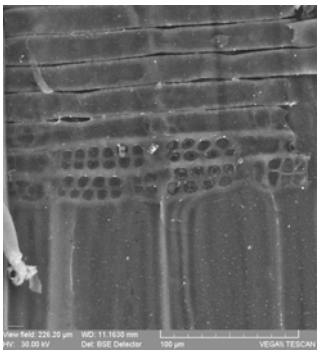

c)

Fig. 6. *SEM micrographs of consolidated samples by total immersion in Paraloid B72* (*$C_4$*) *for 1 h; magnification 1000X: a*) *cross-section; b*) *tangential section; c*) *radial section*

The deposited consolidant is clearly visible after each treatment either as reflective deposits in bubble shape that fill the lumen of fibres (Figure 6a) or spots (bubbles) lighter in colour than the wooden support (Figure 6b) or as deposits that completely fill the pits of the rays (Figure 6c). The SEM analysis of all the sections

suggested an uneven distribution of consolidants into the wooden structure, no obvious regular pattern being noticed, except the fact that a better penetration occurred on the longitudinal direction via fibres and vessels.

## 4. Conclusions

The solution absorption values confirmed the influence of the three treating parameters: concentration, time and solvent type upon the efficiency of the consolidation treatment.

The experiment outlined that the greater the treating time, the higher the solution absorption and the consolidant retention. In the case of concentration as a treating parameter, it was proved that the smaller the concentration, the higher the solution absorption, but a higher concentration is in the favour of the actual retention.

As far as the solvent type is concerned, the mixture of ethanol and acetone led to the highest solution uptake and highest consolidant retention WPG [%].

As an overall conclusion, the weight percentage gain in conjunction with the microscopic analysis may be considered performance indicators in consolidation treatments of old wooden objects with soluble resins and with other newly developed consolidation products.

According to the results of the current research, the optimum variant for wood consolidation by cold immersion treatments with Paraloid B72 is solution $C_4$ of 10% concentration in a solvent mixture of ethanol and acetone 1/1.

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