# Consolidation of Degraded Lime Wooden Support from Heritage Objects Using Two Types of Consolidant

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The novelty of the research consists in the fact that the decayed wood was taken from an old icon, on which several consolidation treatments were applied, and the improvement indices of the decayed wood (by Anobiidae insects) were also determined. This research investigated two types of the most used consolidant (Paraloid B72 10% and Regalrez 1126 25%) solubilized in three types of solvents, to improve the properties of lime wood samples coming from cultural heritage objects that presented different degrees of degradation. Testing methodology for dimensional changes and wood swelling due to solvents impregnation, retention of consolidant in the degraded Tilia cordata wood, and the effectiveness of the consolidation treatment by the Mark hardness method was extensively presented. The highest amount of consolidant was observed when using Regalrez solution, and the lowest amount of consolidant was determined for Paraloid B72 solubilized in acetone. As a general conclusion, the use of Paraloid B72 or Regalrez 1126 for the consolidation of old and degraded lime wood, regardless of the type of solubilizer, will lead to stabilization of the wood degraded properties and the life span of the heritage object.

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#### INTRODUCTION

The biodegradation of cultural heritage objects is a consequence of their improper storage, transportation and use, non-compliance with environmental conditions in exhibition spaces, and because the wood as a lignocellulosic material is vulnerable to biodeterioration (Singh *et al.* 2022; Teaca *et al.* 2019). Biodegradation is the breakdown of organic matter by microorganisms, such as bacteria and fungi. The choice of treatments and the level of intervention must be taken into account (along with the state of the heritage object, the level of degradation, age, species of wood, finish, *etc.*), including the materials used for restoration, which could cause damage of the support and the paint layer. Lahtela and Kärki (2017) and Kain *et al.* (2020) showed that an improvement of wood hydrophobicity should be made without additional bad effects (reducing the impact resistance), and one adequate method is impregnation (along with other methods such as vacuum-pressure application of consolidant, high temperature treatment of wood, chemical surface treatments, *etc.*), where the expected improvements are achieved by filling wood gaps (cell lumens and insect holes) with an inert material.

Treatments that strengthen the wood support can be at surface level or deep in the wood (Schniewind and Eastman 1994; Piena 2001; Tjeerdsma and Militz 2005; Mankovski at al. 2015; Zhang et al. 2019; Fierascu et al. 2020; Harandi et al. 2020). Surface treatments are the simplest and have a dual role of strengthening and water-proofing, but the application of most consolidants could also be done in the depth of the wood. These types of treatments depend on the consolidant viscosity, because at very low viscosity, it might become difficult to distinguish between surface treatment and in deep treatments or impregnation. There is also the use of natural resins, wax, wax and resin mixture, castor oil, or synthetic resins such as Paraloid B72, Regalrez 1126, etc. (Wang and Schniewind 1985; Charola et al. 1986; Smith et al. 2008; Crisci et al. 2010; Mankovski et al. 2015; Salem et al. 2017; Reinprecht et al. 2019; Harandi et al. 2020; Wang et al. 2020). Compared to deep impregnation, impregnation treatment at the surface level is a simpler one, and it is facilitated by enhanced porosity caused by holes and galleries of insects, degradation by xylophagous fungi and bacteria (Mankovski et al. 2015; Deng et al. 2016; Fierascu et al. 2020; Singh et al. 2022). The more degraded and porous the wood is, the easier its impregnation becomes.

Materials and substances that solubilize synthetic resin must meet several important criteria: increased penetrability (*e.g.*, polar solvents), low toxicity, and not producing great dimensional instability to the wood (Hamed *et al.* 2013). It is known that wood is more permeable to non-polar solvents than polar ones. Low polarity solutions allow deep penetration of solvent, and high polarity solutions produce high swelling of the wood. Synthetic resins allow good solubilizing in different solvents (toluene, acetone, ethanol, ethyl acetate, butyl acetate, *etc.*) (Lionetto *et al.* 2012). As mentioned above, a good saturation was obtained by dissolving the resin in acetone, but acetone lead to dimensional instability of the wood.

Many authors and studies (Paris et al. 2015; Gravidel et al. 2020; Kain et al. 2020; Vitali et al. 2021; Santini et al. 2022) have made in-depth case studies on the restoration and consolidation of heritage objects based on wood or with wooden support, highlighting the materials used, the work procedures, and evaluation of the strengthening of the wooden support. The purpose of these studies was to strengthen the support to extend the life of the heritage object and increase the hydrophobicity (Macchioni et al. 2011) so that the heritage object will not degrade in the future. The diagnosis of some vernacular sculptures was made (Vitali et al. 2021), the evaluation of wood from old buildings (Teaca et al. 2019; Fierascu et al. 2020), as well as some methods of cleaning old wood (Hamed et al. 2013) were also analyzed. Modern methods of analysis, such as infrared spectrometry, Fourier transform infrared (FTIR) spectroscopy, and 3D digitization or tomography were used (Pavlidis et al. 2007; Liu et al. 2008; Popescu et al. 2010; Deng et al. 2016; Özgenç et al 2017; Zhao et al 2018; Neamtu et al. 2021). Factors considered have included the quality of adhesives used in the restoration (Walsh-Korbs and Avérous 2019), the effect of water absorption on the mechanical properties (Sakuno and Schniewind 1990), and the reversibility of some typically used resins in consolidation (Charola et al. 1986). Other books or review works (Crisci et al. 2010; Madhoushi 2016; Fierascu et al. 2020; Singh et al. 2022) highlighted the use of Paraloid B72 and Regalrez 1126 for the consolidation of heritage objects.

A critical analysis of the previous studies highlighted that most of them have focused on the analysis and consolidation of specific heritage objects, then generalizing these procedures and materials for the field of restoration/consolidation of wood-based objects (Madhoushi 2016; Ghavidel *et al.* 2020). Modern methods have investigated the

gaps inside of artifacts material (Popescu *et al.* 2010) or the new materials used to strengthen the wooden support (Zhou *et al.* 2020). Deficiencies or weak presentations are found in the evaluation of the effectiveness of the consolidates applied to the degraded linden wood, and especially on the influence of resin-solubilizing substances on the properties of the treated wood. Therefore, this study aims to show the effectiveness of the lime reinforcement treatments using two types of common consolidant, namely Regalrez 1126 in 25% concentration and Paraloid B72 concentration 10%, solubilized in two types of solvents and the influence of solvents on the swelling of degraded wood. The choice of the two consolidant was made due to their high effectiveness, in order to find the best consolidation treatment for degraded lime wood. Additionally, another objective was to obtain the highest possible effectiveness (by using an innovative method of evaluation by pricking) after the consolidation treatments, to increase the lost resistances/density/ swelling of the lime from the damaged cultural heritage objects (with different degrees of fragility/degradation).

### EXPERIMENTAL

#### Materials

The damaged linden wood was obtained from the "Assumption of Virgin Mary" icon, year 1830, taken out of use because of numerous degradations (broken areas, many gaps, and massive insect attack) and the impossibility of restoring it. Parts of this old icon, with different degrees of degradation, were used in experiments. The damaged lime wood was also studied, when consolidated by the action of two types of high effectiveness and used consolidant (Paraloid B72 10% and Regalrez 1126 25%), solubilized in three types of solvents with great capacity of solubilizing (acetone, mixture of ethyl acetate with toluene, and white spirit D40), by determining the mass and dimensional changes, the consolidant retention, the absorption spectroscopy, the effectiveness of consolidation treatments, and wood swelling due to solvent absorption.

Paraloid B72 is an acrylic resin, widely used as an adhesive and consolidant in the field of restoration/consolidation of wood-based heritage objects. This is a polymer composed of two monomers, namely methyl acrylate and ethyl methacrylate, and has a high molecular weight. Regalrez 1126 is an aliphatic resin with a low molecular weight and is the result of polymerization after the addition of monomers based on hydrogenated styrene. This is a cyclic and saturated hydrocarbon, very similar to paraffin wax. The main aim was to penetrate Paraloid B72 into the mass of degraded wood and obtain the effectiveness of the consolidation treatment, by determining the water absorption and Mark hardness of the degraded and consolidated lime wood.

#### **Dimensional Changes Due to Solvents**

Three groups of different preservation state of linden heartwood (*Tilia cordata* Mill.) obtained from the restoration laboratory (Ionescu Constantin Restoration, Sibiu, Romania), respectively, were very degraded (by Anobiidae insects) old wood -8 samples, medium degraded old wood -8 samples, and new (healthy, undamaged) wood -8 samples. The porosity was microscopically determined, as the average number of insect holes, which were identified on the surface of a square with a side of 1 dm<sup>2</sup>. The degradability assessment of the wood material was made based on the number of holes and insect galleries (a scale of three levels), respectively 150 to 180 holes/dm<sup>2</sup> for highly degraded

lime, 80 to 140 holes/dm<sup>2</sup> for lime with medium degradation, and under 80 holes/dm<sup>2</sup> (Singh *et al.* 2022). The medium diameter of insect holes, measured on the wood surface, was 1.4 mm. The samples were cut into test pieces with a square section of 20 x 20 mm<sup>2</sup> and a length of 30 mm and were immersed in two types of solvents (pure and mixed), namely acetone and a mixture of ethyl acetate and toluene 1:1. The specimens were marked, and their dimensions were determined in the radial (R) and tangential (T) sections. Mass (M) for the three immersion stages, of 1 h, 2 h, and 24 h was also determined. Prior to testing, the wood was conditioned for 60 days in a controlled environment with a humidity of 55 ± 5% and a temperature of 20 ± 2 °C. The mixture of solvents (ethyl acetate and toluene) changed the color of the wood (yellowish-brown), because of mixed solvent oxidation (Fig. 1).



**Fig. 1.** Image of high degraded lime wood samples  $(20 \times 20 \times 30 \text{ mm}^3)$  after immersion in acetone (a), and mixed solvent (b)

The two dimensions of samples were measured on perpendicular direction (2 measurements for each sample) with an electronic caliper of 150 mm with precision of 0.01 mm. The mass was determined with a precision balance EWJ 600-2M Kern (Merck KGaA, Darmstadt, Germany), with an accuracy of 0.01 g, and the moisture content with humid-meter Gann HT65 with hammer M20 (GANN Mess, Gerlingen, Germany) in 5 points. Because of the low precision of the electrical humidity meters ( $\pm$  2%) for validating the moisture content of the samples, checks and corrections of the moisture content were made using the gravimetric method (weighing - drying at 105 °C – weighing) (EN 13183-1 2002). Thus, the values obtained by drying the samples in the oven were used. After 1 h, 2 h, and 24 h, the specimens were extracted from the immersion solution, the excess liquid was removed on an absorbent paper, and then the dimensions and mass were measured. The calculation equation used to find the swelling coefficient ( $\beta$ ) according to standard (ISO 13061-15:2017) were the following,

$$\beta_{\rm R} = \frac{LR_{\rm max} - LR_{\rm min}}{LR_{\rm min}} \cdot 100 \quad \beta_{\rm T} = \frac{LT_{\rm max} - LT_{\rm min}}{LT_{\rm min}} \cdot 100 \tag{1}$$

where  $LR_{min}$  is the size before immersion of dried (anhydrous state) samples, in the radial direction (mm),  $LR_{max}$  is the size of wet samples when they are extracted from immersion solvents, in the radial direction (mm),  $LT_{min}$  is dimension before immersion of dried samples (anhydrous state), in the tangential direction (mm), and  $LT_{max}$  is the dimension of wet samples when they are extracted from the immersion solvents, in the tangential direction (mm).

#### **Determination of Consolidant Retention**

A comparison of the masses of solid substance Paraloid B72 and Regalrez 1126 absorbed and left in the wood was desired, depending on the solvent that solubilizes the consolidant and becomes its "transport vehicle" in the depth of the wood. The test specimens with a square section  $(20 \times 20 \text{ mm}^2)$  and a length of 30 mm, in a number of 24 pieces, made of linden wood, and degraded by xylophagous attack of Anobiidae, were cut. The specimens had a moisture content of 10%. The same 24 specimens used in the previous section were divided into three groups, as follows: in the T1 treatment the specimens were immersed in Paraloid B72 10% (Dow Chemical Company, Hayward, CA, USA) solubilized in 1:1 mixture ethyl acetate and toluene, in T2 treatment the specimens were immersed in consolidant Paraloid B72 10% solubilized in acetone, and in T3 treatment the specimens were immersed in consolidant Regalrez 1126 (Eastman Chemical Company, Kingsport, TN, USA) 25% solubilized in white spirit D40, and the trade name as an authorized restoration product was Rexil. These products (Paraloid B72 and Regalrez 1126) are considered non-hazardous under the OSHA Hazard Communication Standard (29CFR1910.1200). The immersion time was 2 h (120 min), and the measurements (mass and dimensions) were made immediately after extraction, and at 2 days after drying at a temperature of 30 to 40 °C and relative humidity of  $45 \pm 5\%$ . The relationship for determining the amount of consolidant was based on the ratio between the mass of the consolidant (obtained by weighing) and the initial mass of the sample.

Two or three applications of consolidant Paraloid B72 and Regalrez in solution (for each type T1, T2, and T3) were also repeated, with the intermediary drying of 24 days, the percentage of consolidant remaining in the wood being determined for a single application. These repeated treatments aimed to increase the dried consolidant mass and depth of thought in the wood samples.

# Absorption Spectroscopy in Infrared with Attenuated Total Reflection (FTIR-ATR)

To identify the penetration levels with the consolidant Paraloid B72, FTIR-ATR analysis was utilized at the Physical-Chemical and Biological Investigation Laboratory of the National Museum of History (Bucharest, Romania). The FTIR-ATR measurements were performed with an Alpha Bruker spectrometer Optics (Ettlingen, Germany), equipped with the Platinium ATR accessory, in the spectral range 4000 to 400 cm<sup>-1</sup>, with a resolution of 4 cm<sup>-1</sup> and 32 scans. At least three spectra for microsections were collected for each wood sample. OPUS 7.0 software (Bruker Optics, Ettlingen, Germany) was used for spectrum processing and evaluation. The FTIR-ATR spectrum of wood is composed of a multitude of absorption bands corresponding to the infrared vibrations of the functional groups of cellulose, hemicellulose, and lignin. According to literature data (Tjeerdsma and Militz 2005; Liu *et al.* 2008; Popescu *et al.* 2010; Özgenç *et al.* 2017) in the region 3900 to 2700 cm<sup>-1</sup> are the vibrations of the functional groups OH and CH, and the region 1900 to 800 cm<sup>-1</sup> represents the fingerprint of the wooden structure.

Samples of sound (new wood, clean and without defects)/undamaged lime and degraded lime wood, untreated and treated by immersion in Paraloid B72 solution in ethyl acetate and toluene (1:1), of 10, 15, and 30% concentrations, were analyzed after 24 days. After drying, the moisture content of samples was approximately 10%. To be able to evaluate the degree of penetration of Paraloid B72 in the wooden samples subjected to the study, at least three micro-sections taken from the whole thickness were analyzed for each sample. On the thickness of the sample, the first microsection was taken 2 mm from the

surface, the second from the middle of the sample, and the last one 2 mm from the other side of the sample.

#### Determination of Water Absorption after Each Treatment with Consolidant

The application of consolidant, in addition to the strengthen role, also had the role of reducing water absorption (waterproofing), thus increasing dimensional stability. To provide evidence of this phenomenon, the specimens previously treated in consolidating solutions (24 pieces) were immersed in distilled water at 20 °C for 2 h, to observe the water absorption, compared to similar specimens, without treatment (EN- 317 1993; ASTM D7433-19 2019). The same 24 linden specimens in different stages of degradation caused by xylophagous attack, with a square section with a side of  $20 \pm 0.2$  mm and a length of  $30 \pm 0.2$  mm, were conditioned and weighed on precision balance. The specimens had an initial moisture content of 10% (conditioned to 20 °C temperature and 55% air humidity), both were cut from healthy/sound/undamaged wood and those with consolidant. The specimens were extracted from the water after immersion, stored on absorbent paper for 2 to 3 s to remove excess water adhering to the wood surface, after which they were again weighed on an electronic precision balance.

Water absorption was determined for 8 replicates of each treatment with the following equation Eq. 2,

$$WA = \frac{M_{\rm f} - M_{\rm i}}{M_{\rm i}} \cdot 100 \tag{2}$$

where  $M_f$  is final mass, after immersion in water, of wet samples (g), and  $M_i$  is initial mass of the test piece with consolidant, of dry sample (g).

#### Effectiveness of Consolidation Treatment by the Wood Puncturing

The method of determining the hardness of wood by puncturing with a Mark dynamometer, also called Mark hardness ( $H_M$ ), is a simple and versatile way of assessing the surface and the interior of the wood, both for the sound/undamaged and damaged ones. The level of degradation was determined also as a measure of the effectiveness of consolidation to which the degraded wood has been subjected.

Measurements were made in the tangential direction. This direction is recognized as having a higher hardness (Crisci *et al.* 2010). The Mark hardness method used a hardened steel tip, with a diameter of 1.34 mm, on a depth of 6 mm (the taper being only 2 mm). In addition to the fact that a hardness is indicated, the compressive strength of the wood and its level of degradation can be ascertained.

The damaged wooden specimens were the same 24 pieces used for the consolidation treatment (minimal 3 tests on each sample face), with dimensions of  $20 \times 20 \times 30 \text{ mm}^3$ , and a moisture content of 8 to 10% (so called consolidated wood specimens). Similarly, sound wooden specimens are made, with the same dimensions, as benchmarks. Measurements of  $H_M$  hardness were made on treated samples 21 days after the first treatment and 60 days after the last  $3^{rd}$  treatment, on the dried and conditioned samples with 10% moisture content.

To determine the hardness of the wood for all three types of treatment (T1, T2, and T3), the calculation equation was used,

$$H_M = \frac{F}{A_l} \tag{3}$$

where  $H_M$  is Mark hardness of dry samples (N/mm<sup>2</sup>), *F* is maximum force, read on the Mark 10 dynamometer (N), and  $A_1$  is the total lateral surface of the penetrating tip, determined as a sum of the cylindrical and conical part (21.28 mm<sup>2</sup>).

The differentiated efficacy between consolidation treatments based on Mark hardness was obtained using Eq. 4,

$$EH_M = \frac{H_{Mf} - H_{Mi}}{H_{Mi}} \cdot 100 \tag{4}$$

where  $EH_M$  is effectiveness of consolidation treatment by measuring Mark hardness (%),  $H_{Mf}$  is Mark hardness obtained after first consolidation treatment, second consolidation treatment, or final hardness after the third treatment (N/mm<sup>2</sup>), and  $H_{Mi}$  is hardness in the previous stages (before the first treatment, the second, and the third treatment), or the initial hardness (N/mm<sup>2</sup>).

A relationship similar to Eq. 4 was used to determine the increase/decrease of some parameters (absorption of solvent, amount of consolidant, *etc.*) from one treatment stage to another or between two different treatments.

#### **Statistical Analysis**

As the main statistical parameters of the trend and spread survey, the arithmetic mean and the standard deviation of the data were determined. The aim of this statistical analysis was to obtain an overview of some parameters, beyond the stringing of their values, to be able to make comparisons and other comparative analyses. Through using the capabilities offered by MS Excel (Microsoft Corp., v.2019, Redmond, WA, USA), specific graphs were created, on which the standard deviations and the average values were arranged, and by using the statistical program Minitab 18 (Penn State University, State College, PA, USA), two types of comparison graphs were created, an Empirical Cumulative Distribution Function (eCDF) and Probability Plot, with different statistical parameters.

# **RESULTS AND DISCUSSION**

#### Mass and Dimensional Changes in Solvents

The solvents used for the consolidates implicitly led to the swelling of the wood, regardless of its degradation. In addition, the combination of water from the wood with the highly volatile solvent of the consolidation resins caused more swelling (Fig. 2).

Based on Eq. 2, different absorptions of solvents were obtained depending on the degradation of lime wood (highly degraded, medium degraded, and sound/undegraded wood), immersion time (1 h, 2 h, and 24 h), and type of solvent as mix (either ethyl acetate + toluene or acetone), as can be seen in Fig. 2.

Figure 2 shows that acetone had a higher absorption than the mixture of ethyl acetate with toluene, 1.11 times higher for highly degraded lime and 1 h of immersion. There was a noticeable increase from 1 h immersion to 2 h of immersion in acetone, 18.6% for lime with medium degradation and 55.1% from 2 h to 24 h immersion in the same solvent. The explanation of this phenomenon is given by the fact of wood gaps, the degraded wood having many insect holes beside the cell gaps (lumen and intercellularly holes). The largest differences in absorbed solvent were observed between highly degraded and sound lime wood, with values of 6.47 times in the case of acetone and 1 h of

immersion, 5.23 times in 2 h of immersion, and only 2.44 times for 24 h of immersion. It was observed that acetone had a better absorbance than the mixed solution of toluene with ethyl acetate, because of different polarity.



Very degraded Medium degraded Sound

**Fig. 2.** Absorption of solvents in very degraded, medium degraded, and sound/undamaged lime wood samples, with mix of ethyl acetate + toluene and acetone (Mix) for different immersion times

In terms of solvent absorption, acetone resulted in larger swelling and mass increases than the mixture of ethyl acetate and toluene because of polarity. These laboratory tests are in line with the results obtained in current practice, when panels of considerable size are treated with acetone and change their size, especially on the tangential section. For example, a value of tangential swelling at 24 h immersion for highly degraded lime of 7.68% for an icon with dimensions of 600 to 800 mm, will cause large dimensional increases of 4.6 to 6.1 mm. This dimensional increase was given by large cell wall swelling. Dimensional increases due to swelling were differentiated by sections of wood, solvents, and wood degradations. Thus, in the tangential direction the increase was higher than in the radial one (because of different anatomical structure) by 4.9% for medium degraded lime immersed 1 h in acetone. It was higher for immersion in acetone compared to the mixture between ethyl acetate and toluene 1:1 by 135%, and it was higher for the average degraded wood than for the sound/undamaged lime wood by 28.1% (Fig. 3), because acetone is a polar solvent.

The swelling of the wood under the action of solvents was different, depending on the degradation of the lime wood, the type of solvent used, and the direction of measuring the dimensional increases. For example, for very degraded wood immersed in acetone, the initial size of 19.98 mm, and the final size after immersion of 20.17 mm was determined. Through applying the Eq. 1, a swelling of 0.95% was obtained (Fig. 3). Regarding the influence of wood degradation on swelling, it was found that highly degraded wood had the largest swellings (Fig. 3), and undegraded wood had the smallest swellings in both solvents, the differences being between 2.88 to 4.32% for a 24-h immersion period in acetone in a tangential direction.

Regarding the action of the solvent type, acetone produced the largest swellings of the wood compared to the mixture of ethyl acetate and toluene 1: 1, with differences of 0.77%, 2.99%, and 2.2% for 1 h, 2 h, and 24 h immersion durations, respectively, in the tangential direction of new/undamaged wood and 4.23%, 5.01% and 4.29% for highly degraded wood. It is observed that the degraded wood has much larger swellings than new/undamaged wood, the swelling increasing from 1 h to 2 h, but decreased slightly after 24 h immersion. The explanation for the large differences in swelling between sound/undamaged and degraded wood is that the degraded wood has more empty spaces where the immersion solvent will enter. All empty spaces in degraded lime wood were the sum of the insect holes plus the inherent porosity of the wood. The obvious conclusion is that the time immersion is not necessary for 24 h, a period of only 2 h being sufficient to obtain the expected effects of swelling.



Fig. 3. Swelling of degraded/undegraded lime wood samples in solvents

# **Results on Consolidation Retention**

After immersion in the consolidant solution, different amounts of dry mass of consolidant remained in the wood, depending on the type of treatment (Table 1).

Table 1 shows different values of consolidant retention for the T1 treatment specimens (B72 dissolved in solvent mixture), from 9.87% to 16.98%, resulting in an average value of 13.39%. For the T2 treatment, the specimens immersed in Paraloid B72 dissolved in acetone with a concentration of 10%, the average consolidant retention was 11.62%, and the treatment T3, the specimens immersed in Rexil (Regalrez 1126 dissolved in white spirit), had an average consolidant retention of 36.21%. The greater the mass of consolidant left in the wood, the better the properties of the consolidated wood, *i.e.*, greater hardness and hydrophobicity.

Treatment Type		Sample Number								
		1	2	3	4	5	6	7	8	Mean
T1	Initial (g)	5.10	3.14	4.75	3.53	4.65	3.77	5.61	4.82	4.42
	Final (g)	5.61	3.65	5.22	4.16	5.23	4.41	6.22	5.43	4.99
	Amount (g)	0.51	0.51	0.47	0.63	0.58	0.64	0.62	0.61	0.57
	Amount	10.01	16.24	9.89	17.85	12.47	16.98	11.07	12.65	13.39
	(%)									
T2	Initial (g)	5.41	4.99	5.09	2.68	5.23	5.45	4.87	5.02	4.84
	Final (g)	5.97	5.47	5.66	3.14	5.78	5.98	5.49	5.61	5.38
	Amount (g)	0.56	0.48	0.57	0.46	0.55	0.53	0.62	0.59	0.545
	Amount	10.35	9.62	11.21	17.16	10.52	9.72	12.73	11.7	11.62
	(%)									
T3	Initial (g)	5.55	5.05	5.26	4.82	4.46	5.40	5.86	4.75	5.14
	Final (g)	7.28	6.91	6.75	6.74	6.57	7.31	7.77	6.58	6.98
	Amount (g)	1.73	1.85	1.49	1.92	2.11	1.91	1.91	1.83	1.84
	Amount	31.17	36.63	28.33	39.83	47.31	35.37	32.57	38.53	36.21
	(%)									

Table 1. Consolidant Retention	Values for T1, T2,	and T3 Treatments
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For the situation in which the consolidant is applied in 2 to 3 stages (Table 2), it was observed that the retention of the consolidant in the second stage was higher for the specimens that were immersed in the consolidant solubilized in acetone, compared to the specimens that had the ethyl acetate and toluene mixture. An explanation of this difference is given by the polarity of acetone. Additionally, in the third stage this retention decreases noticeably, because the wood gaps were already full of consolidant. As with the first consolidation, the T3 treatment is the best related to T1 and T2, the retention reached maximum values of 51.75%, after the 3<sup>rd</sup> stage of consolidation treatment. The classification of the 3 types of treatment from the point of view of retention after 3 stages of application of the consolidant is kept as a single stage of application.

Т	reatment Type	Initial Mass	Final Mass (g)	Retention (g)	Retention (%)
		(g)			
T1	First time	4.77	5.41	0.64	13.41
	2nd time	5.41	5.99	0.42	7.76
	3rd time	5.99	6.19	0.20	3.3
	Total	4.77	6.0	1.23	25.7
T2	First time	4.3	4.8	0.5	11.6
	2nd time	4.8	5.0	0.2	4.1
	3rd time	5.0	5.1	0.1	2.0
	Total	4.3	5.1	0.8	18.6
T3	First time	5.14	6.98	1.84	36.21
	2nd time	6.98	7.59	0.61	8.73
	3rd time	7.59	7.80	0.21	2.76
	Total	5.14	7.80	2.0	51.75

**Table 2.** Average Consolidant Retention on the 3 Stages and Types of

 Treatment for Medium Degraded Wood Samples

For several consolidation stages (Table 2), it can be seen that the retention of the consolidant increases from one stage to another. Practical implications of these results are found in the fact that in restoration processes only one application can be used when it ensures the amount of consolidant and the necessary hardness. For the T1 treatment type,

the retention of consolidant after the 3 stages increased 91.6% compared to the first stage, for the T2 treatment type the increase was 60.3%, and for the T3 treatment type the increase was only 42.9%.

Regarding the retention of consolidant, comparing the T1 and T2 treatments, there was an improvement from 11.62% to 13.39% (an increase of 15.23%) in retention of consolidating Paraloid B72 dissolved in the mixture of ethyl acetate and toluene 1:1, compared to acetone. The highest retention of consolidant had the specimens from the T3 treatment, the Regalrez 1126 consolidant dissolved in white-spirit D40, with the best capacity to penetrate the wood structure. Retention of T3 treatment increased 3.11 times compared to T2 and 2.7 times relative to T1. The increased efficiency of T3 treatment is arguably in line with the research (Mankovski et al. 2015), which presented the following: Regalrez 1126 has a low molecular weight and tends to penetrate deeper into the parenchymal cell area and leave the general porosity of the wood unaltered; in contrast, Paraloid B72, with higher molecular weight, tends to concentrate in the cell lumen and thus decreases the overall porosity. Drying of the treated specimens should be done slowly for 72 to 96 h, after extraction from the consolidant solution. From here it can show that to perform an effective consolidation treatment with Rexil, it is recommended that it must be done more than 72 h after the first treatment and, in accordance with Crisci et al. (2010) double and triple treatment produces increased benefits for the consolidation of de-graded wood. The conclusion is that the use in the restoration of low molecular weight resins (Regalrez 1126) and low polarity solvents (acetone) has among its advantages an increased resistance to yellowing and the possibility of the resins to solubilize in the same solvents, non-polar, even after aging (Crisci et al. 2010). Additionally, the acetone solvent produces dimensional changes or deformations from the flatness. From this point of view, the solvent mixture of ethyl acetate and toluene is preferable to acetone.

For the multi-stage treatments, it was found that the specimens had a different absorption and residual mass of the consolidant, depending on the solvent used. Because some improvement was observed, reflected in the remaining mass and high hardness, from one treatment to another, it resulted that it is important to perform multiple treatments, in 2, 3, or even more stages. Further, it was also found that the solvents acetone and mixture of ethyl acetate with toluene for Paraloid B72 led to a retention of consolidant after the 3 treatments that was substantially equal, only that the mixture of toluene with ethyl acetate changed dimensionally and deformed the wood, less than acetone. Noticeably higher mass values of T3 treatment with Rexil were observed, which had a retention of 178.2% higher than T2 (B72 in the mixture solvents) and 101.3% compared to T1 (Paraloid B2 in acetone), but it had a slow evaporation. This increase is because the Rexil resin has a low molecular weight.

#### **FTIR-ATR Results**

The FTIR-ATR results of degraded and sound/undegraded lime wood with Paraloid B72 consolidant are shown in Fig. 3. All FTIR-ATR spectra of the sound/undamaged or degraded wood samples showed the infrared absorption bands characteristic of the functional groups of cellulose, lignin, and hemicellulose. The wood-specific peaks (P) were visible at 1605, 1510, and 1269 cm<sup>-1</sup> for lignin and 1737, 1370, and 895 cm<sup>-1</sup> for carbohydrates (Liu *et al.* 2008). Paraloid B72 (the upper spectrum in Fig. 3) had other bands, namely 2950, 1700, 1410, 1200, 1050, 850, and 750 cm<sup>-1</sup>. Sound/undamaged lime consolidated with 10% B72 (the middle spectra from Fig. 3) has diminished or lost the bands peaks for Paraloid B72 of 2950, and 850 cm<sup>-1</sup>. For the degraded lime wood,

penetration of the Paraloid B72 consolidant was noticed (Fig. 3). It was rendered visible by the peak of 1737 cm<sup>-1</sup>, even if it was half attenuated. Additionally, the band peak of 895 cm<sup>-1</sup> specific to linden wood was visible. When the wood samples were immersed in 10%, 25%, and 30% concentration of solution, the main bands of linden wood and Paraloid B72 were observed. This penetration of consolidant can be explained in response to the measurement of the spectra, by observing the decreasing specific peaks of Paraloid B72 (from 30 to 10%) at 1410 cm<sup>-1</sup>. Increasing the percentage of Paraloid from 10 to 30% intensified the bands specific to it and slightly decreased the peak specific to linden wood (Fig. 3).



**Fig. 3.** Example of FTIR-ATR spectra for sound (new) lime wood treated with B72 10%; T-ref – new (sound) reference lime;  $T_10_1$  - new lime with Paraloid B72 10% concentration, section 1;  $T_10_2$  -  $T_10_1$  – new (sound) lime with B72 10%, section 2;  $T_10_3$  – new (sound) lime with B72 10% concentration, section 3); Td\_ref - degraded lime as reference, without consolidant

Correlating the methodical part with the one from the results (Fig. 3), it can be concluded that the immersion of wood in Paraloid B72 had visible influences on the composition of the degraded wood through its penetration into the wood. These influences were highlighted by the absorbance bands visible in Fig. 3, more intense on the surface of the wood (first lamella) and less intense towards the core of the wood (due to progressive penetration). In this way, plotting both graphs of the absorbance of the Paraloid and the reference linden sample with those of the immersed samples, the degree of absorption of the consolidant in wood was highlighted.

The data obtained from the FTIR analysis both for lime wood and Paraloid B72 were in agreement with the literature data (Liu *et al.* 2008). The infrared absorption bands obtained from the FTIR-ATR analysis of degraded lime were generally equivalent to sound wood, respectively to its functional groups of cellulose, lignin, and hemicelluloses, and their values do not differ depending on the wood species used (sound/undegraded lime

wood, high and medium degraded lime wood), and the consolidation treatment used (Paraloid B72 with different concentrations of 10%, 25%, and 30%). From Fig. 3 it is observed that the Paraloid B72 consolidant entered the wooden structure for all wood samples treated with solutions of different concentrations. It is also observed that the intensity of the infrared absorption bands of Paraloid B72 for micro-sections increased in the order of increasing the concentration of B72 solution, from 10% to 25% and to 30%. The explanation is because a higher concentration of B72 led to a more visibility of its specific bands.

The current analysis on a single diagram (Fig. 3) clearly highlights the followings: - the main spectral points of Paraloid B72;

- the main spectral points of clean linden wood;

- the spectral points of the lime wood treated with Paraloid B72, highlighting the spectral points of both linden wood and those of Paraloid B72;

- the existing spectral losses on the lamella extracted from the middle area of the specimens.

#### Water Absorption after Consolidation

The results of water absorption for the 3 types of treatments T1, T2, and T3 applied to degraded lime wood are presented in Fig. 4. For consolidated lime wood, the average values of water absorption decreased from 5.27% to 2.03% (a decrease of 61.4%) for T1 treatment, from 5.03% to 1.41% (a decrease of 71.9%) for T2 treatment, and from 5.42% to 3.67% (a decrease of 32.2%) for T3 treatment.



**Fig. 4.** Water absorption (before and after treatment of linden samples): T1-treatment type; Meanaverage value; StDev - standard deviation; N-number of experiments

From the point of view of the weighed mass, the degraded wood without treatments will double its mass by absorbing water during immersion, *i.e.*, the wood without treatment

absorbs more than 110%. Regarding water absorption, a noticeable improvement was found in the hydrophobicization of wood subjected to consolidation treatment with synthetic resins, regardless of the type of treatment and solvent used, compared to untreated wood. Therefore, the treatments with Paraloid B72 (T1 and T2 types) had the best hydrophobic action, the treatment with Regalrez 1126 being weaker from this point of view, with 52.5% weaker than the treatment with Paraloid B72 in mixture of solvents, and with 44.8% weaker than treatment with Paraloid B72 in acetone.

Analyzing the three values of water absorption, it can be seen that the treatment with B72 consolidant in acetone was the most effective in terms of absorption, whereas the T2 treatment type had the lowest value of absorption. The treatment that was the least effective, from the point of view of hydrophobicing, was the T3 type, where the Regalrez consolidant solubilized in white spirit D40 reduced only 32.2% the water absorption, although it had the highest retention of the consolidant. It followed that the consolidant Regalrez 1126 produced a weaker hydrophobicing of degraded wood.

#### Mark Hardness Results (HM)

Table 3 shows the average values of Mark hardness by stages and types of treatment and their effectiveness (calculated with Eq. 4) due to the increases in hardness recorded after the consolidation of the degraded specimens. If the efficacy analysis is performed at the first consolidation stage, the most effective treatment was that the consolidation with Paraloid B72 solubilized in a mixture of ethyl acetate and toluene, with a Mark hardness increase of 23.1%, and the least effective was the treatment with B72 in acetone. Efficacy after the three consolidation treatments was changed, the treatment with Regalrez 1126 being the most effective, with an increase in Mark hardness of 53.7% (with a noticeable increase in stages 2 and 3 of 30.6%), and the B72 treatment dissolved in a mixture of acetate of ethyl and toluene again preceding the treatment with B72 dissolved in acetone (T2).

Therefore, for the T1 treatment (Table 3) and consolidation step 1, the hardness was improved 23.16%, increasing from 8.55 to 10.53 N/mm<sup>2</sup>. In stages 2 and 3 the efficiency was improved 13.39% compared to the previous stage, due to the Mark hardness values of 11.94 N/mm<sup>2</sup> compared to 10.53 N/mm<sup>2</sup>. For the multiple consolidation produced in all 3 stages, the  $H_M$  hardness increases were 39.65% and 11.94 N/mm<sup>2</sup>, respectively, compared to 8.55 N/mm<sup>2</sup> for untreated wood. Therefore, it was found that the effectiveness of the treatment is revealed in increasing the Mark hardness from one stage to another.

	Treatment Stage	Нм	Treatment Effectiveness (%)			
Туре		(N/mm²)	Between 0 and	Between 1 and	Between 0 and	
			1	3	3	
T1	Untreated (0)	8.55				
	1 Aplication (1)	10.53	23.16	13.39	39.65	
	3 Aplications (3)	11.94				
	Untreated (0)	12.87				
T2	1 Aplication (1)	14.85	15.38	13.33	30.77	
	3 Aplications (3)	16.83				
	Untreated (0)	8.08				
Т3	1 Aplication (1)	9.49	17.45	30.66	53.47	
	3 Aplications (3)	12.40				

**Table 3.** Mean Values of  $H_M$  Hardness and Treatment Efficacy in the Three Stages for Medium Degraded Lime

For the T2 treatment in stage 1, the increase was 15.38% compared to the untreated wood, in treatment stages 2 and 3 the increase was 13.33% and the growth difference between the untreated wood and the one with multiple treatments was 30.77%. However, if analyzing the two reference specimens T1 and T2 that have the same number of treatments and the same type of consolidant B72 but solubilized in different solvents, in stage 1 between the two specimens T1 and T2, the T1 specimens have a higher hardness by 31.08% (4.78 N/mm<sup>2</sup>) compared to the T2 specimens. In stages 2 and 3 the values were substantially equal, and if a comparison is made between the wood without treatment with that with multiple treatment, it found that the T1 specimens have increased hardness compared to those in T2 by 28.86%, a noticeable increase was highlighted mainly in stage 1 of treatment.

A brief analysis of the standard deviations of the Mark hardness values obtained after one or more applications of the consolidates (Table 3), shows that the 95% confidence interval is respected, and the normality of the values is confirmed. Using Minitab 18 software, a test for equal variances was made. It was determined that the variances of standard deviation of the 3 groups did not differ noticeably.

Regarding the determination of Mark hardness, a comparison between the hardness values of the untreated wood with those with multiple treatment were made. The results obtained from the difference between the final and initial values show that the specimens of T2 treatment increased the hardness by 3.96% after first treatment compared to the T1 samples, resulting that the total hardness improvement was 16.81%. However, from the point of view of the restoration of heritage objects with wooden support, it is considered that the solution of the solvent mixture (ethyl acetate and toluene 1:1) solubilized Paraloid B72 in a way that did not affect the dimensional stability and flatness. The permeability and penetrability of the solubilized consolidant in a mixture of ethyl acetate and toluene is at least as good as for the acetone solvent, and in addition, the hardness is noticeably improved.

It is observed that from one stage to another the Mark hardness was improved noticeably and almost constantly. These tests confirm and agree with a previous study (Crisci *et al.* 2010), which shows that multi-stage treatment improves efficiency, as well as that impregnation can produce increases in hardness. In absolute values it can be stated that the hardness of the T3 series of specimens increases to  $4.32 \text{ N/mm}^2$ , compared to  $3.96 \text{ N/mm}^2$  at T2 and  $3.39 \text{ N/mm}^2$  for T1.

# CONCLUSIONS

- 1. Through the three types of treatments performed (T1, T2, and T3), it was confirmed that the hardness of the wood, the retention of the consolidant, the depth of penetration of the consolidant, and the reduction of water absorption had good increases, especially if 2 to 3 successive treatments were performed.
- 2. In addition to the fact that the consolidation substances used (Paraloid B72 and Regalrez 1126) increased the hardness properties of degraded wood, they also led to dimensional stabilization and hydrophobicization of degraded wood.
- 3. The study on the absorption and swelling of degraded wood in solvents highlighted the disadvantages of acetone and the advantages of using a mixture of ethyl acetate and toluene. Therefore, if a ranking were to be made of the 3 treatments used in the research

from the point of view of efficiency and other collateral consequences, the first place would be the consolidation treatment with Regalrez (T3), the second place would be the treatment with Paraloid B72 solubilized in mixture of ethyl acetate and toluene (T2), and in the last place would be the treatment with Paraloid B72 solubilized in acetone.

4. As a general conclusion, the use of Paraloid B72 and Regalrez 1126 provides viable options for the restoration of heritage objects.

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