

**INFLUENCE OF TWO COLD IMMERSION TREATMENTS UPON THE CONSOLIDANT
UPTAKE AND ITS DISTRIBUTION INTO THE WOODEN STRUCTURE**

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Abstract

Consolidation of old wooden objects by cold immersion into consolidation polymer solutions is one fundamental treatment in the conservation of wooden cultural objects. Its main purpose is to ensure structural integrity and thus the preservation of object authenticity. The efficiency of a consolidation treatment is mainly given by the quantity and distribution of the consolidation product which penetrates into wood. The present paper presents an experimental study upon the weight percentage gain and distribution of Paraloid B 72 consolidant into the structure of poplar wood. The consolidants used were prepared at a concentration of 10% in two different solvents, toluene and a mixture of ethanol and acetone 1:1. The consolidation products were applied on sound poplar samples (10x10x15) mm by two different cold immersion treatments, several times of exposure being applied. The distribution of the consolidant inside the wood structure as well as its uptake was investigated by means of Scanning Electron Microscopy.

The aim of this research was to establish the optimum recipe considering the two viewpoints. This represents the first stage within a wider research which envisages the addition of nano-insertions into the present consolidant Paraloid B72 in order to increase several properties of the consolidant material: fire retardancy, resistance to biological attack, mechanical strength.

Key words: cold immersion, wood consolidation, Paraloid B72, consolidant distribution, consolidant retention

1. INTRODUCTION

Wood was and still remains a favourite material in human life, especially in the field of furniture, works of art, religious items and other applications. Its conservation therefore has been an aim for the last decades and still plays an important role nowadays, current research being undertaken. One of the most important aspects when conserving old wooden artefacts, besides the preservation treatment, is consolidation, a treatment usually necessary when the object is seriously altered. The purpose of the consolidation treatment is to impart sufficient strength to the object while maintaining its integrity which has been lost in time because of biological attack, chemical and physical degradation factors (Wang and Schniewind 1985, Timar et al. 2010, Clausi et al. 2010).

Consolidation with soluble thermoplastic resins is the most preferred treatment in conservation practice of porous materials, including wood, due to the advantages such a treatment offers: ease of application, increased physical and mechanical properties of the treated object (Wang and Schniewind 1985, Crisci et al. 2010), potential reversibility of the consolidation product (Appelbaum 1987), adhesive properties (Sakuno and Schniewind 1990, Podani et al. 2001), adhesion and cohesion, two fundamental properties required to consolidants and possible resistance to biological attack (Clausi et al. 2010).

On the other hand, any treatment in conservation, consolidation treatments included, should keep to several criteria based upon internationally established principles (Appelbaum 1987, Oberlander et al. 1989, Unger and Unger 1994, Unger et al. 2001, Farmakalidis 2006, Unger 2009):

- Any material in conservation must not alter the integrity and authenticity of the object
- Any material or treatment must present potential reversibility and allow further interventions
- Any material or treatment must be compatible with all the materials present on the object in question, the artistic materials being treated as a whole and not separately.

Additionally, the general requirements of consolidation materials, apart from reversibility, re-treatability and compatibility, include uniformity of distribution and consolidant retention, two properties that ultimately define the efficiency of a consolidation treatment (Farmakalidis 2006, Unger 2009, Timar et al. 2010).

The current research is an experimental assessment of two consolidation treatments with soluble thermoplastic acrylic resin Paraloid B72 by cold immersion in terms of consolidant retention and its distribution in the wooden structure. The final obtained data stand for a key starting point in the development of new consolidation materials with nanoparticle insertions.

2. MATERIALS AND METHODS

2.1. Synthetic resin consolidants

Paraloid B72 is a well-known and studied consolidation material (Schniewind and Eastman 1994, Podany et al. 2001, Chapman et al. 2001, Favaro et al. 2007, Vaz et al. 2008) which is extensively used in the consolidation of degraded wooden works of art. 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 as a consolidant. It has also been used as an interface barrier that, at least in theory, lends greater reversibility to joints made with non-reversible adhesives such as epoxies or polyesters and limits the penetration of these materials into the adherend substrate (Podany et al. 2001, Ellis and Heginbotham 2002).

The consolidants used were prepared at a concentration of 10%, frequently used in conservation practice, employing two different solvents, toluene and a mixture of ethanol and acetone 1/1. These solvent types were used to better understand the influence of the type of solvent in terms of polarity on the efficiency of a consolidation treatment: polar solvents, ethanol and acetone and a non-polar solvent, toluene due to their properties. Since ethanol and acetone are polar solvents, they are compatible to wooden supports, which would mean a higher retention and possible better distribution of the consolidant due to higher attraction forces, while toluene, an aromatic non-polar solvent, would theoretically lead to a better penetration of the consolidant (Schniewind and Eastman 1994, Timar et al. 2010).

The prepared consolidation products were coded C₁ for Paraloid B72 in toluene, and C₂ for Paraloid B72 in ethanol and acetone mixture 1/1.

2.2. Wooden samples

The wooden samples used in the research were sound poplar test pieces (*Populus tremula*) with the dimensions of 10x10x15 mm (longitudinal x tangential x radial). Poplar wood had historically been used in wood artefacts especially for painted and gilded objects, being a wood species easy to work. On the other hand, the structural properties of poplar, like porosity and permeability, made it an ideal material in this research whose main objective was to study the flow of consolidants through the

wooden structure in terms of distribution and consolidant retention. Finally, the susceptibility of poplar wood to microbiological attack, often posing serious problems of loss of structural integrity, cohesion and mechanical resistance, are several other aspects to be analysed in the current research, its natural low durability having a direct influence upon the faith of artefacts.

Before applying any immersion treatment, the wooden samples were sanded on all surfaces with H240 sandpaper grit and conditioned at 20°C and RH= 55% until constant weight, these being in fact the ambient conditions that wooden objects are usually kept in.

2.3. Consolidation treatments

In the present study, two types of cold immersion treatments were used: total immersion and vertical immersion. The wooden samples were immersed for different periods of time: 15- 30- 60 -120 minutes; for each treatment a batch of 5 wood test pieces was used. Moreover, during the immersion treatments the samples were kept in airtight boxes to prevent solvent evaporation.

For the *total immersion* treatments special devices were manufactured to prevent samples from floating and to ensure permanent contact of all the surfaces to the consolidation liquid. The samples were fixed in the device in vertical position (Fig. 1), placed in glass vessels and covered in solution entirely (the consolidant solution exceeding the samples with one centimetre). After the treatment, the samples were air dried in the laboratory for two days and then conditioned in a climatic chamber, T = 20°C and RH= 55%, until constant weight.

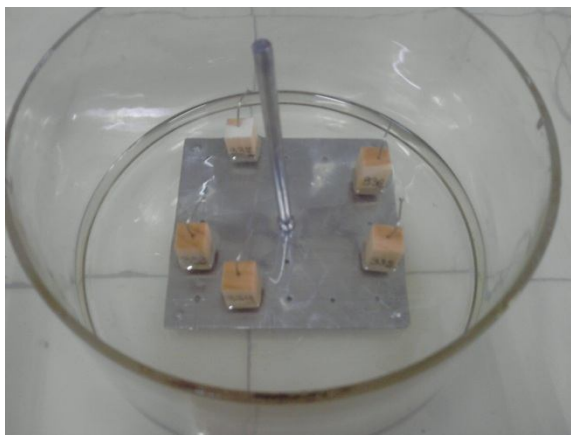


Fig. 1. Immersion treatments: the wooden samples fixed into the device, vertical immersion.

The second applied treatment, called *vertical immersion*, was meant to test a new consolidation method for immersion treatments in terms of impregnation efficiency. This procedure was used by Kucerova and Ohlidalova in 2009 to monitor the penetration of acrylic consolidants into wood, but it may also prove a valuable technique of immersion in conservation practice. During this treatment, the samples were fixed in the manufactured device in vertical position and placed in glass vessels into which the consolidant solution was then added so that only 5 mm from the bottom side of the samples was immersed into the liquid.

2.4. Characterisation and experimental testing

After the two immersion treatments, the samples were conditioned and weighed periodically until reaching constant weight, the weight percentage gain (WPG %) resulted from the consolidant retention being computed at this point.

The characteristics of the consolidants and of the consolidated samples (the uniformity of distribution) were reflected by means of electron microscopy SEM. For the SEM observations, small examination samples were prepared as follows. The treated samples were first immersed entirely into water for two days, the transversal surface straightened by a microtome and then small pieces of approximately 3x3x3 mm on the three fundamental directions (longitudinal, radial, tangential) were cut. These small samples were dehydrated by gradual drying at temperature progressively increasing from 20°C up to 60°C in an electric oven and then mounted on metal bases. Finally, the mounted samples were metallised and kept in proper storage conditions for further SEM examination.

3. EXPERIMENTAL RESULTS AND DISCUSSIONS

3.1. Samples experimental testing

The treatment of poplar test pieces by *total immersion* with the consolidation products employed C₁ and C₂ resulted in a weight increase as reflected by the weight percentage gain values. In the case of Paraloid B72 10% in toluene (C₁) the samples gain less in weight (0,85-1,24 %) than in the case of Paraloid B72 10% in mixture of ethanol and acetone 1/1 (C₂), where the values ranged between 1,09% and 1,81% (Fig. 2).

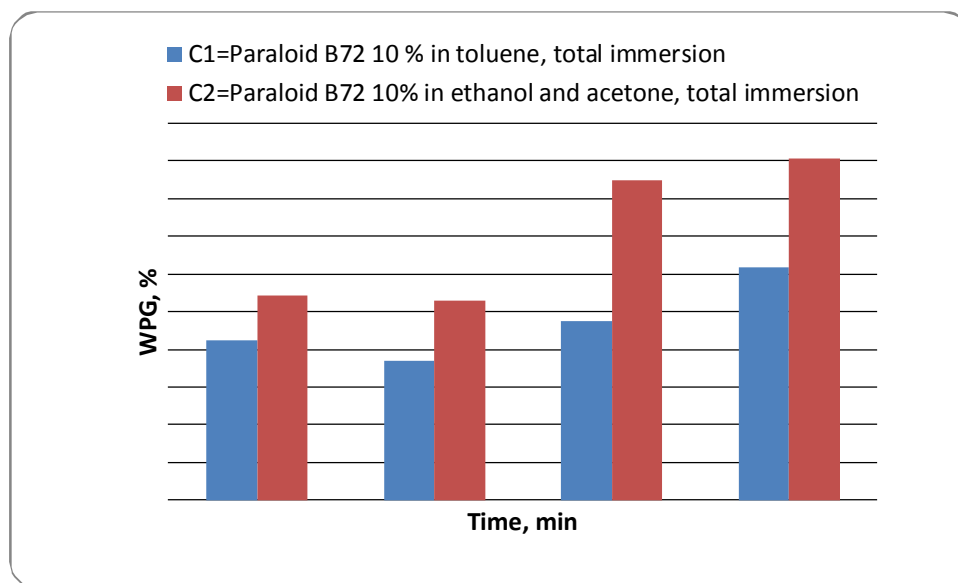


Fig. 2. Comparative date of the weight percentage gain of poplar samples treated by total immersion in the two consolidation products C1 and C2.

In the case of *vertical immersion*, the same phenomenon occurs, the samples gaining much more in weight when immersed in the C₂ consolidant, with values between 3,16-5,54 %, whilst samples treated in consolidant C₁ gained less, the WPG values ranging between 1,52% and 2,88% (Fig. 3).

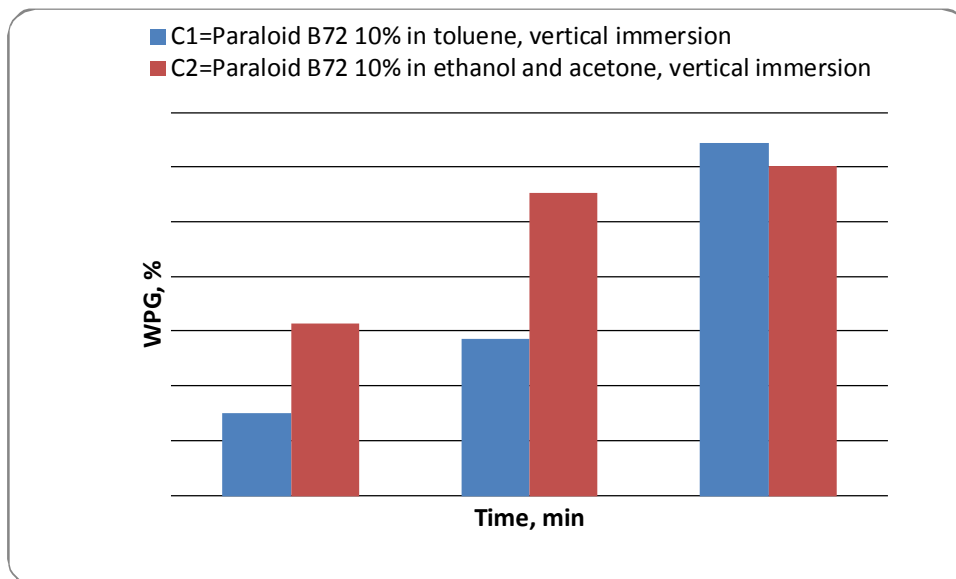


Fig. 3. Comparative data of the weight percentage gain of poplar samples treated by vertical immersion in the two consolidation products C1 and C2.

This higher absorption obtained with the C₂ consolidant may be the result of the influence of polar solvents (mixture ethanol-acetone), similar results being noticed by other researchers (Schniewind and Wang 1985, Timar et al. 2010).

On the other hand, differences of weight gain are also noticed between the two treating procedures: the samples treated with C₁ by total immersion gain less in weight (0,85-1,24 %) than those consolidated in the same product by vertical immersion (1,52-2,88 %). Similarly, the test pieces immersed totally in C₂ solution had a weight gain between 1,09-1,81%, while during the vertical immersion the consolidant retention was higher 3,16-5,54% (Fig. 4).

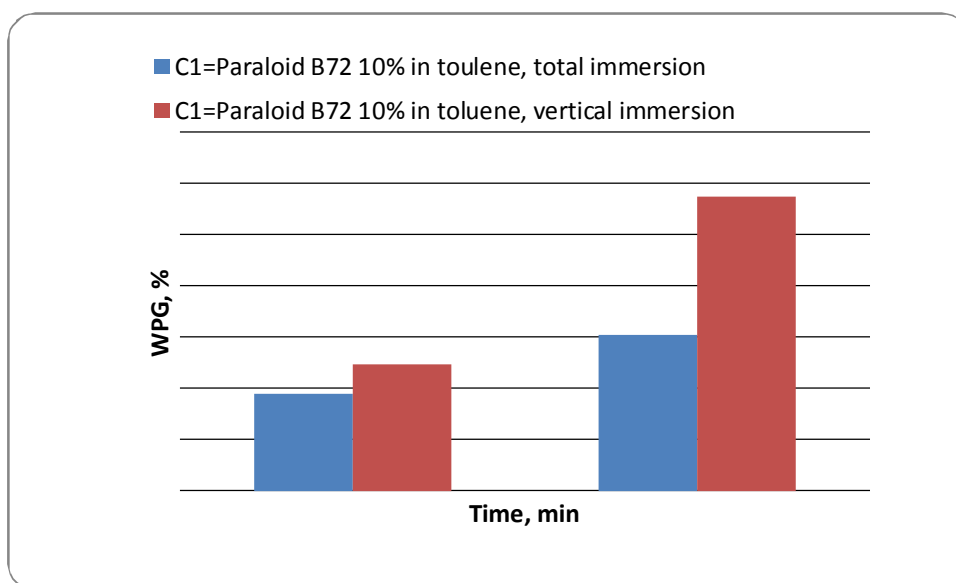


Fig. 4. Comparative data of the weight percentage gain of poplar samples treated by total immersion and vertical immersion in the consolidation product C1.

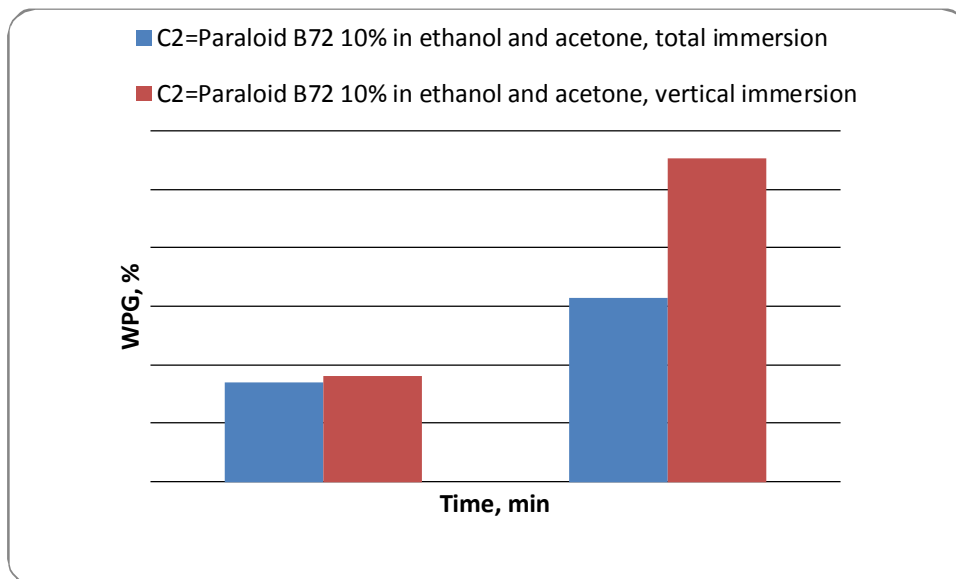


Fig. 5. Comparative data of the weight percentage gain of poplar samples treated by total immersion and vertical immersion in the consolidation product C₂.

As a conclusion to this point, the experimental results reflect that the vertical immersion is more efficient than total immersion in terms of weight percentage gain, a target of all consolidation treatments. The efficiency of such a treatment is even supported by the conservation practice, conservators and restorers consolidating fragile and degraded wooden objects by injecting the consolidant along the grain (in longitudinal direction). Moreover, ever better results should be obtained when using polar solvents such as ethanol and acetone.

3.2. Microscopic characterisation

The microscopic analysis concerning the efficiency of the consolidation treatments in terms of consolidant distribution was performed on a SEM microscope model VEGA II LSH. The investigation was undertaken by comparison between a control group of untreated poplar samples in all three grain directions (Fig. 6) and groups of consolidated samples by total and vertical immersion (Fig. 7- Fig.11).

The consolidated samples by total immersion were investigated on all three directions while the test pieces consolidated by vertical immersion were finally analysed on their cross-section, this section characterising best the efficiency of the treatment. In addition, the investigation of cross-sections was also meant to somewhat characterise the penetration depth of the consolidants during the vertical immersion treatment. In this respect, both cross-sections, the bottom cross-section soaked in the liquid and the upper cross-section, were analysed by electron microscopy. The presence of the consolidant on the upper cross-sections which were not soaked in the consolidant confirmed its penetration. A better penetration was noticed after the 2 h vertical immersion treatment (the amount of the consolidant was greater) than in the case of the 1 h vertical immersion treatment.

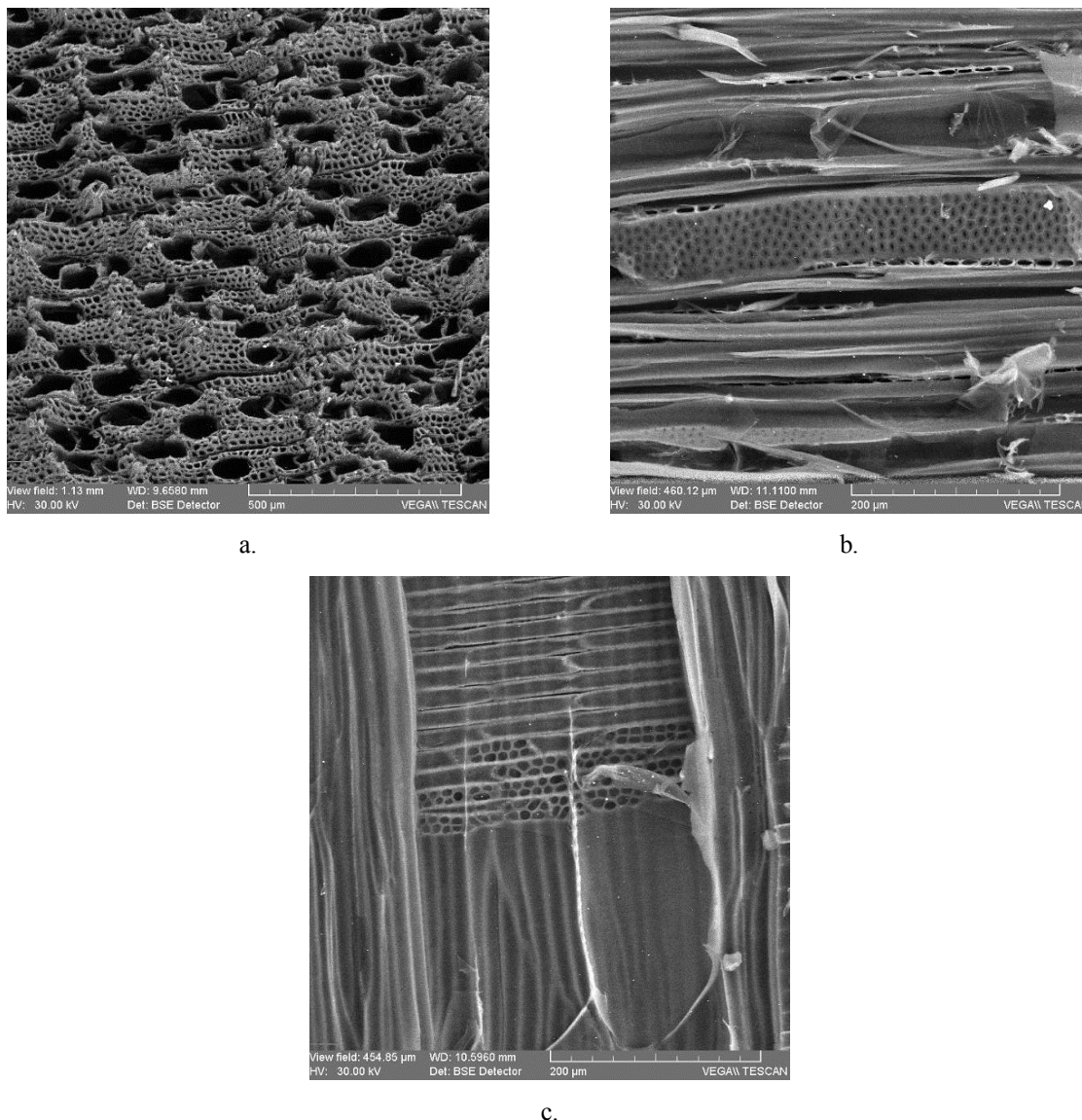


Fig. 6. SEM micrographs of control (untreated) poplar samples, magnification 500X:

a. Cross-section; b: tangential section; c. radial section.

An expected effect of the consolidation treatment is the filling of voids so that this particular aspect was a relevant property investigated for characterising the efficiency of consolidants. The investigated sections (Fig. 7) outlined the path ways of the consolidant through the wooden structure: the main anatomical elements being the vessels and rays, as well as the interconnecting pits, observations confirmed by previous research regarding the distribution of consolidants (Schniewind and Eastman 1994, Timar et al. 2010).

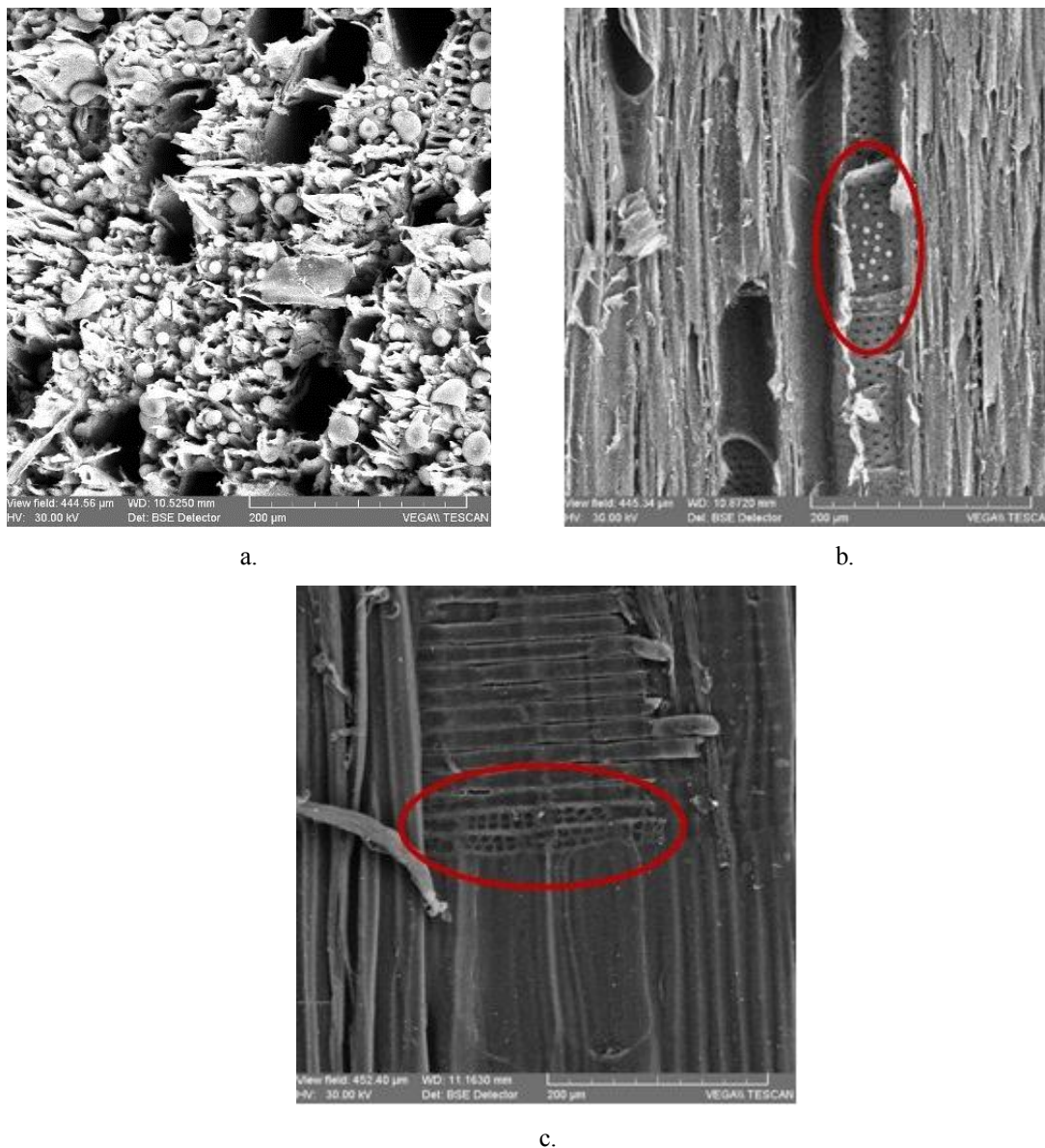


Fig. 7. SEM micrographs of consolidated pieces by *total immersion* in Paraloid B72 in ethanol and acetone mixture, for 30 min, consolidant (C_2), magnification 500X:

- a. Cross-section: the consolidant is visible through vessels and fibres as bubbles;
- b. Tangential section: the consolidant deposition is confirmed by the white spots in the pits;
- c. Radial section: the consolidant deposition in ray / vessels interconnecting pits.

The microscopic analysis of the total immersion treatments (a batch of 5 samples) led to the conclusion that the most relevant section that outlines the distribution of the consolidation products is the cross-section. Accordingly, for the vertical immersion treatments were analysed only those sections (Fig. 8-11), respectively two cross-sections for each sample.

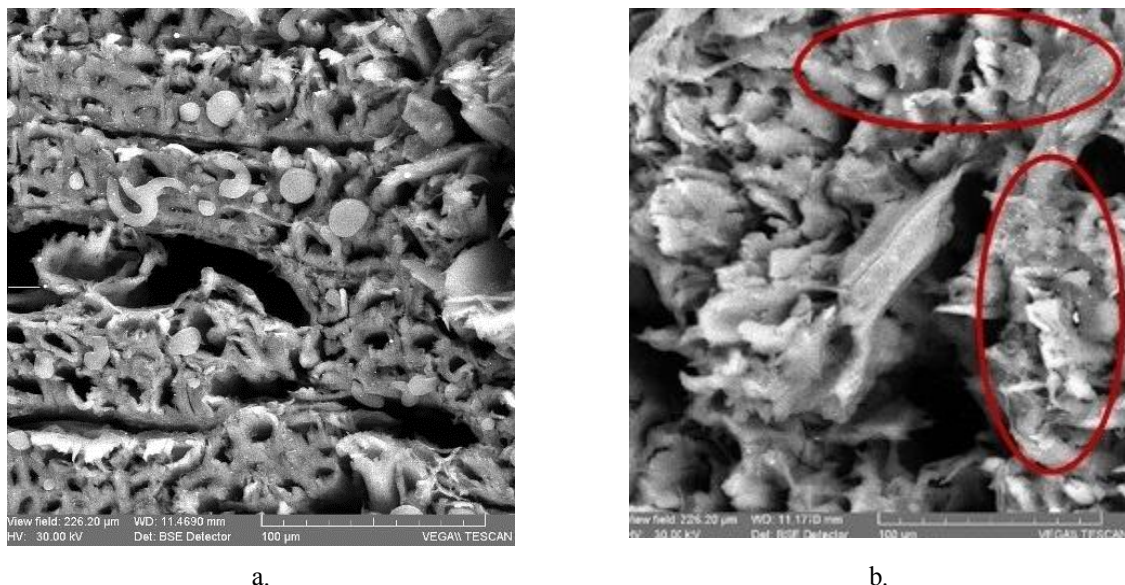


Fig. 8. SEM micrographs of consolidated samples by vertical immersion with Paraloid B72 10% in toluene (C_1) for 1 h, 1000X:

- a. Cross-section 1: soaked in the consolidation product;
- b. Cross-section 2: the upper section not soaked in the consolidant, consolidant deposits visible.

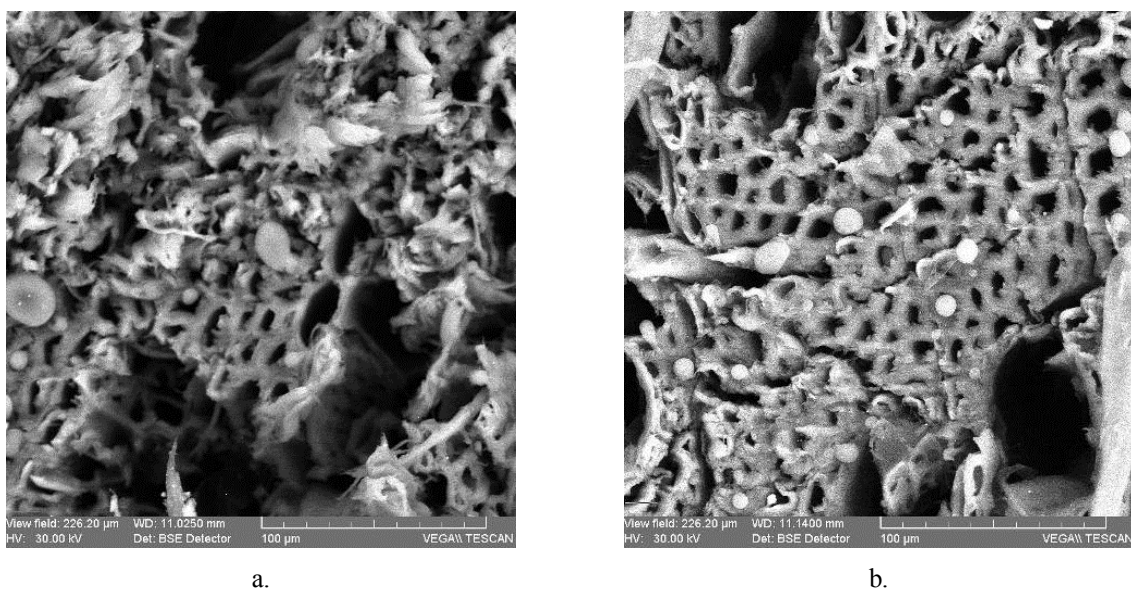


Fig. 9. SEM micrographs of consolidated samples by vertical immersion with Paraloid B72 10% in toluene (C_1) for 2 h, 1000X:

- a. Cross-section 1: the section soaked in the product;
- b. Cross-section 2: the upper section not soaked in the consolidant, consolidant deposits clearly visible.

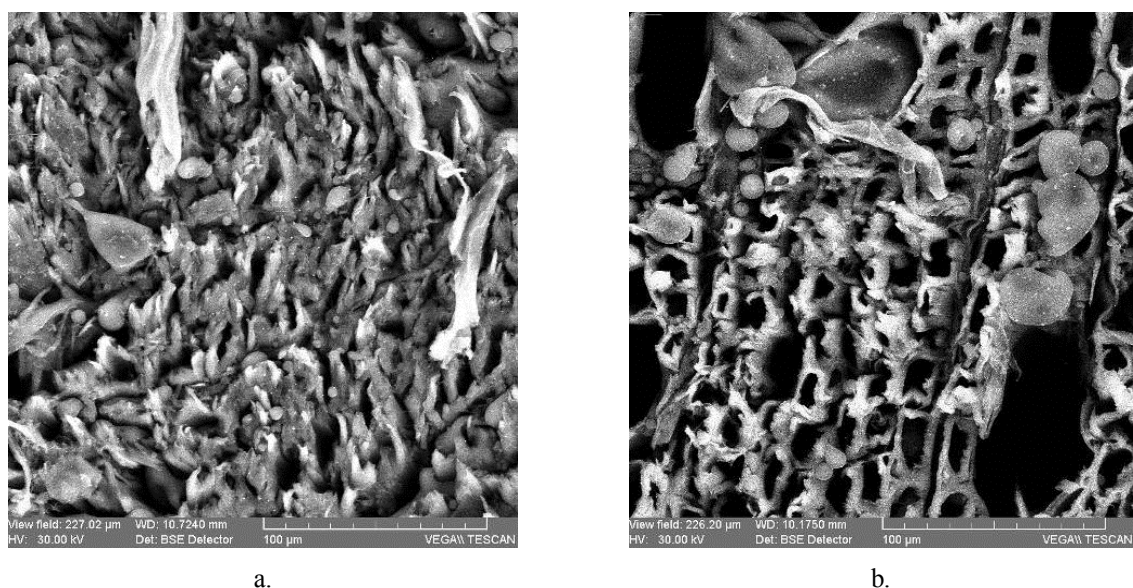


Fig. 10. SEM micrographs of consolidated samples by vertical immersion with Paraloid B72 10% in ethanol and acetone (C₂) for 1 h, 1000X:

- a. Cross-section 1: the section soaked in the product;
- b. Cross-section 2: the upper section not soaked in the consolidant.

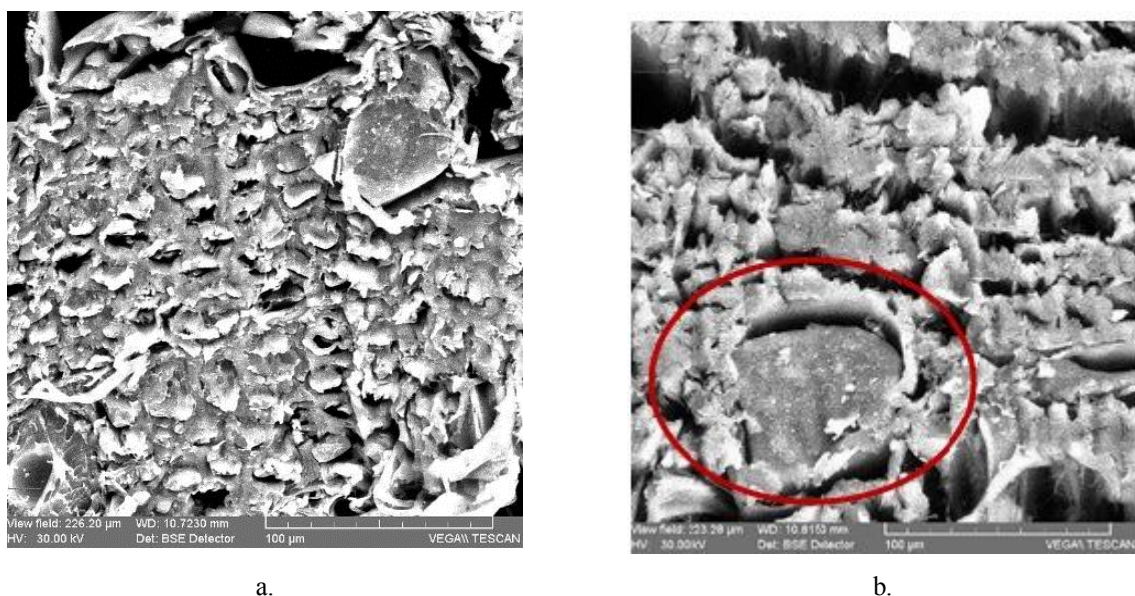


Fig. 11. SEM micrographs of consolidated samples by vertical immersion with Paraloid B72 10% in ethanol and acetone (C₂) for 2 h, 1000X:

- a. Cross-section 1: the section soaked in the product;
- a. Cross-section 2: the upper section not soaked in the consolidant, consolidant deposits into vessels and fibres; some of the vessels lumina are completely filled.

The deposited consolidant is clearly visible after each treatment as either spots (bubbles) lighter in colour than the wooden support (Fig. 7-10) or as reflective deposits that fill the lumina (Fig. 11). 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. A better filling of pores seems to be achieved by vertical immersion than by total immersion treatments (see Fig. 8-11 compared to Fig. 7). On the other hand, no visible difference concerning the influence of the solvent type on the consolidant distribution could be noticed, though the degree of lumina filling seems to be higher in the case of polar ethanol-acetone mixture, than in the case of toluene, supporting the quantitative data referring to consolidant retention (WPG values). However, a completely uniform treatment (distribution) and a total filling lumina seem not to be possible by immersion with Paraloid B72 solutions, as also concluded by Schniewind and Eastman (1994).

Last but not least, all the analysed micrographs of the consolidated samples revealed a current phenomenon that takes place during consolidation treatments with soluble resins: the migration of the consolidant during solvent evaporation towards the surface of the sample. This phenomenon, however, apparently unfortunate, may render opportune properties to the consolidated object. The migration to the surface of the consolidant may give better mechanical and physical strengths, better dimensional stability of the object, and ultimately, but of prime importance in conservation practice, reversibility, as also concluded by Schniewind and Eastman (1994).

4. CONCLUSION

As an overall conclusion, the weight percentage gain as well as 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 concentration of the consolidant Paraloid B72 is 10%, the most effective treatment is by vertical immersion and the most proper solvent for wooden supports is the mixture of ethanol and alcohol 1/1.

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