Effects of Nanosilver-Impregnation and Heat Treatment on Coating Pull-off Adhesion Strength on Solid Wood

Učinci impregnacije nanočesticama srebra i toplinske obrade na čvrstoću prianjanja premaza na drvo

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ABSTRACT • *Effects of impregnation with a silver nano-suspension, as well as of heat-treatment, on pull-off adhesion strengths of the coating system on three commercial solid wood species were studied. The wood species included beech, poplar, and fir. The size range of silver nanoparticles was 30 – 80 nm. The specimens were coated with an un-pigmented sealer and a clear finish on the basis of an organic solvent. The results showed that the highest and the lowest pull-off strengths were found in beech specimens heat-treated at 145 °C (5.7 MPa) and in nanosilver-impregnated poplar specimens heat-treated at 185 °C (2.5 MPa), respectively. Impregnation with nanosilver decreased pull-off strength in the case of all species as a result of formation of micro checks in the cell walls caused by the impregnation under high pressure in vessel. Heat-treatment at the temperature lower than 145 °C increased pull-off strength as to the irreversible hydrogen bonding in the course of water movements within the pore system of the cell walls, resulting in extra bonds among cell wall components and higher mechanical properties. However, heat-treatment at the temperature higher than 185 °C significantly decreased the strength as the degradation of hemicellulose and cell wall wood components caused significant decrease in mechanical strength and cell wall thinning. High thermal conductivity coefficient of silver intensified the impact of heat-treatment by rapid absorption of heat on the surface of the specimens.*

Keywords: *coating, nanotechnology, heat treatment, permeability, porous structure, solid wood.*

SAŽETAK • *U radu su istraživani učinci impregnacije drva srebrnom nanosuspenzijom, kao i učinci toplinske obrade na čvrstoću prianjanja (adhezije) sustava premaza na tri komercijalne vrste masivnog drva. U istraživanjima je analiziran sustav premaza na drvu bukve, topole i jele. Raspon veličine srebrnih nanočestica bio je 30 – 80 nm. Uzorci su obrađeni nepigmentiranim punilom i završnim premazom na osnovi organskog otapala. Rezultati su pokazali da je najveća čvrstoća prianjanja izmjerena na uzorcima od toplinski obrađene bukovine pri 145 °C (5,7 MPa), a najmanja na uzorcima od drva topole impregniranima srebrnim nanočesticama i toplinski obrađenima*

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pri 185 °C (2,5 MPa). Impregnacija srebrnim nanočesticama smanjila je čvrstoću adhezije na svim vrstama drva, što je posljedica stvaranja mikropukotina na staničnim stijenkama uzrokovanima impregnacijom pod visokim tlakom u posudi. Toplinska obrada na temperaturi nižoj od 145 °C povećala je čvrstoću adhezije s obzirom na ireverzibilne vodikove veze u toku kretanja vode u sustavu pora staničnih stijenki, što rezultira dodatnim vezama između komponenti stanične stijenke i boljim mehaničkim svojstvima. Međutim, toplinska obrada na temperaturi višoj od 185 °C bitno smanjuje čvrstoću adhezije jer propadanje hemiceluloze i komponenti stanične stijenke drva uzrokuje znatno smanjenje mehaničke čvrstoće i stanjivanje stanične stijenke. Visoki koeficijent toplinske vodljivosti srebra pojačava učinak toplinske obrade brzom apsorpcijom topline na površini uzorka.

Ključne riječi: *premazivanje, nanotehnologija, toplinska obrada, propusnost, porozna struktura, masivno drvo*

1 INTRODUCTION
1. UVOD

The porous structure in different wood species is one of the root causes of difference in physical and mechanical properties of the wood produced by each species. The size of the pores, the way they are inter-connected or isolated from the neighboring pores, and even the quality of the surface of the pores would all influence the properties. Many factors influence the formation of wood and thereof its structure and porous system; factors such as initial spacing, intercropping with different plants, drying procedures (Oltean *et al*., 2007), growing season, extractive content, and moisture content and hygroscopicity of wood (Borrega and Karenlampi, 2010; Hering *et al*., 2012). Wood is frequently modified by engineering processes to get stiffness or homogeneous mechanical properties because only few species exhibit radial and axial uniformity. Moreover, some surface preparation techniques were used for better adhesion of wood surface (Militz and Viöl, 2008; Militz *et al*., 2013).

Thermal modification is by far the most commercially-advanced wood modification method. The thermal modification of wood has long been recognized as a potentially useful method to improve the dimensional stabilization of wood and increase its decay resistance (Hill, 2006). Although it has negative effects on the strength properties of wood, there are some techniques for mitigating these effects (Awoyemi, 2007). Thermal modification is invariably performed between the temperatures of 180 °C and 260 °C. The temperatures lower than 140 °C result only in slight changes of material properties and higher temperatures result in unacceptable degradation of the substrate (Hill, 2006). Studies of the thermal treatment of wood above 300 °C are of a limited value, due to severe degradation of the material. Modern thermal modification processes are limited to temperatures not higher than 260 °C; in practice, the temperature ranges from 150 °C to 230 °C are generally used, because hydrolysis is very slow at lower temperatures, whereas cellulose degradation begins to occur in the region 210-220 °C (Hill, 2006). Cellulose degradation becomes predominant at 270 °C. A sharp increase in the free-radical content of wood was also found when it was heated to temperatures above 200 °C (Hill, 2006). The reduction of wood swelling with increasing temperature and duration of thermal treatment was often attributed to hemicelluloses destruction (Hill, 2006; Taghiyari *et al.*, 2013). However, structural modifications and chemical changes of lignin were suggested to be also involved in the process (Repellin and Guyonnet, 2005). Furthermore, Borrega and Karenlampi (2010) indicated that reduction in hygroscopicity was not only due to mass loss, but another mechanism also existed. They suggested that this mechanism might be related to irreversible hydrogen bonding in the course of water movements within the pore system of the cell walls.

High thermal conductivity coefficients of metal nanoparticles (Pati, 2012) were used to improve some of the properties of solid woods and wood-composite materials (Taghiyari *et al*., 2013). Both impregnation with nanosilver suspension and heat-treatment were also reported to alter the porous structure of solid woods, significantly changing the gas and liquid permeability (Taghiyari, 2013), and possibly the penetration of coatings and paints into the porous structure, altering their adhesion strength. The effects of different thermal modifications on pull-off adhesion strength of paints were investigated in some studies (Meijer, 2004; De Moura *et al*., 2013; Nejad *et al*., 2013); however, in the literature, the authors could not find any reports on the effects of nanosilver-impregnation and heat-treatment on adhesion strength of paints and finishes on solid woods. The present study was, therefore, aimed at finding the effects of heat-treatment on pull-off adhesion strength in solid woods. In the meantime, considering the previous experience in which changes in physical and mechanical properties of solid wood species were reported by their increased thermal conductivity due to the impregnation with metal nano-suspension (Taghiyari, 2011; Taghiyari *et al*., 2013; Taghiyari, 2013), a separate set of specimens were impregnated with nanosilver suspension to increase the thermal conductivity of the specimens and find out its effects on pull-off adhesion.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Specimen preparation
2.1. Priprema uzoraka

Two hardwoods and one softwood were chosen based on their commercial importance in various industrial and musical applications in Iran - beech (*Fagus orientalis* Lipsky), poplar (*Populus nigra* L.), and fir (*Abies alba* Mill.). From each species, 30 tangential specimens were prepared; the size of each specimen was 250 mm × 150 mm × 10 mm; the specimens were

Table 1 Specifications of the sealer and clear coating used in the present study
Tablica 1. Specifikacija punila i premaza upotrijebljenih u istraživanju

Coating / *Premaz*	**Solids, %** *Čvrsta tvar, %*	**Viscosity (25 °C)** *Viskoznost pri 25 °C* cP	**Density, g/cm³** *Gustoća, g/cm³*	**Appearance** *Izgled*
Sealer / *punilo*	38±1	120±10	0.97	Clear liquid
Clear finish (un-pigmented coating) *završni premaz (nepigmentirani premaz)*	39±1	80±10	0.97	Clear liquid

free from any knots, fissures, and checks; they were kept in conditioning room (25±2) °C, and (40±3) % relative humidity for four weeks. The surface of the specimens was sanded with 100-grit sandpaper; they were then wind-blown to remove the dust and wood floor. All specimens (including the control, the heat-treated, nanosilver-impregnated, and heat-treated + nanosilver-impregnated specimens) were equally coated with an un-pigmented sealer-clear resin with an organic solvent and produced by Pars-Eshen Co., as to its great popularity in the local market; technical specifications of the resins are in Table 1. Nitrocellulose was used as the binder of the clear finish. The coating was applied by spraying in two separate runs by a professional workman. The thickness of the coating was measured to be 150 μm. Four dollies of 20 mm in diameter were fixed to each specimen for testing the coating-adhesion strength. The moisture content of specimens was (8±0.5) % in all treatments, where the pull-off tests were carried out because wood has a thermo-hygromechanical behavior and its deformation properties depend on the combined action of temperature, relative humidity, and mechanical local variations (ASTM D4541-02; Figueroa *et al*., 2012).

2.2 Nanosilver impregnation
2.2. Impregnacija nanočesticama srebra

A 400 ppm aqueous dispersion of silver nanoparticles was produced and applied to the specimens using electrochemical technique (Khaydarov *et al*., 2009; Taghiyari and Norton, 2014). The size range of silver nanoparticles was 30-80 nm. The formation and size of the silver nanoparticles was monitored by transmission electron microscopy, for which samples were prepared by drop-coating on to carbon-coated copper grids. The pH of the suspension was measured to be 6-7; two kinds of surfactants (anionic and cationic) were used in the suspension as stabilizer; the concentration of the surfactants was three times the nano-silver particles. Empty-cell impregnation process (Rueping method) was carried out in pressure vessel under 3 bars of pressure by Mehrabadi Machinery Mfg. Co. (Tehran, Iran). Before and after the impregnation process, all specimens were weighted with a digital scale with 0.01 g precision, and their dimensions were measured by a digital caliper with 0.01 mm precision, to measure the density and the amount of nano-suspension absorption. After the impregnation, all specimens (including the control, heat-treated, nanosilver-impregnated, and heat-treated + nanosilver-impregnated specimens) were kept at room temperature for three months. Once the NS-impregnated specimens were dried under the conditions mentioned in the Specimens Preparation section, they were heat treated.

2.3 Heat-treatment process
2.3. Proces toplinske obrade

All specimens for heat-treatment were randomly arranged in an oven. They were heated at 145 °C for 12 hours. Heat-treated specimens were marked with HT; and specimens impregnated with nanosilver were coded by NS. Then, all HT-145 and NS-HT-145 specimens were taken out of the oven. HT-185 and NS-HT-185 specimens were heated at 185 °C for another four hours for comparison purposes of two temperature levels (145 and 185 °C). Explanation on the abbreviations and the specimen coding are in Table 2.

2.4 Pull-off adhesion strength testing
2.4. Određivanje čvrstoće adhezije

Adhesion strength testing provides the force required to pull a specified test diameter of a coating away from its substrate, using hydraulic pressure. Adhesion tests were carried out in accordance with the ASTM D4541-02. In the present study, an automatic PosiTest® pull-off adhesion tester was used; it is a self-

Table 2 Definition of the abbreviations and coding of the specimens
Tablica 2. Definiranje i skraćeno označivanje uzoraka

Code of the treatment *Oznaka obrade*	**Description of the treatment** / *Opis obrade*
Control	Specimens without any treatment / *uzorak bez obrade*
Control-NSI	Specimens impregnated with silver nano-suspension *uzorak impregniran suspenzijom srebrnih nanočestica*
HT145	Heat-treated specimens at 145 °C / *uzorci toplinski obrađeni pri 145 °C*
NSI-HT145	Nanosilver-impregnated specimens heat-treated at 145°C *uzorci impregnirani srebrnim česticama i toplinski obrađeni pri 145 °C*
HT185	Heat-treated specimens at 185°C / *uzorci toplinski obrađeni pri 185 °C*
NSI-HT185	Nanosilver-impregnated specimens heat-treated at 185°C *uzorci impregnirani srebrnim česticama i toplinski obrađeni pri 185 °C*

Table 3 Density of wood species before the impregnation process and the amount of nano-silver absorption in each species
Tablica 3. Gustoća drvnih uzoraka prije procesa impregnacije i količina nanočestica srebra u uzorcima svake vrste drva

Species / *Vrsta drva*	***Fagus orientalis*** / *Bukva*	***Populus nigra*** / *Topola*	***Abies alba*** / *Jela*
Nano-silver absorption, g/cm^3 *Apsorbcija nanočestica srebra, g/cm^3*	0.37	0.28	0.08
Density, g/cm^3 / *Gustoća, g/cm^3*	0.56	0.34	0.43

aligning spherical dolly-head tester (Type V according to the ASTM standard).

Diameter of the dolly used was 20 mm. The greatest tensile pull-off strength X that the coating could adhere to the substrate was evaluated in MPa (Equation 1). The breaking points, demonstrated by fractured surfaces, occurred along the weakest plane within the system consisting of the dolly, adhesive, coating layers and substrate.

$$X = \frac{4 \cdot F}{\pi \cdot d^2} \quad (1)$$

Where F is the rupture force (N), and d is the diameter of the experiment cylinder (mm) (ASTM D4541-02).

The moisture content of the specimens at the time of the pull-off adhesion tests was (8±0.5) %, and the temperature was (25±3) °C.

2.5 SEM Imaging
2.5. SEM fotografije

Scanning Electron Microscope (SEM) imaging was carried out in the thin-film laboratory, FE-SEM lab (Field Emission), School of Electrical & Computer Engineering, University of Tehran; a field-emission cathode in the electron gun of a scanning electron microscope provides narrower probing beams at low as well as high electron energy, resulting in both improved spatial resolution and minimized sample charging and damage.

2.6 Statistical analysis
2.6. Statističke analize

One-way analysis of variance (ANOVA) was conducted to discern significant difference at 95 % level of confidence, using SAS software program (version 9.2) (2010). Grouping was then made between treatments using the Duncan test. Regression and hierarchical cluster analyses, including dendrogram and using Ward methods with squared Euclidean distance intervals, were carried out by SPSS/20 (2011). Cluster analysis was performed to find similarities and dissimilarities between treatments based on more than one property simultaneously (Ada 2013).

3 RESULTS
3. REZULTATI

Results from weighing and measuring the specimens before and after the impregnation process showed that the highest nano-suspension absorption was in beech specimens (0.37 g/cm^3), and the lowest in fir (0.08 g/cm^3) (Table 3).

Visual observation showed that, in nearly all cases, the failure occurred in the damaged woody substrate, not in the coating or the adhesive layers. In fact, the pulled-off dollies showed a great deal of fibril and woody substrate stuck to it. Similar results were reported for eucalyptus and pine woods (De Moura *et al*., 2013). This indicated that the coating and adhesion procedures were carried out correctly; that is, the surface of the substrate provided suitable anchoring for the finish to be stuck to. The maximum pull-off adhesion strength was observed in beech specimens heat-treated at 145 °C (5.68 MPa); the minimum adhesion strength was seen in nanosilver-impregnated poplar specimens heat-treated at 185 °C (2.46 MPa) (Fig. 1). Impregnation with nanosilver suspension decreased

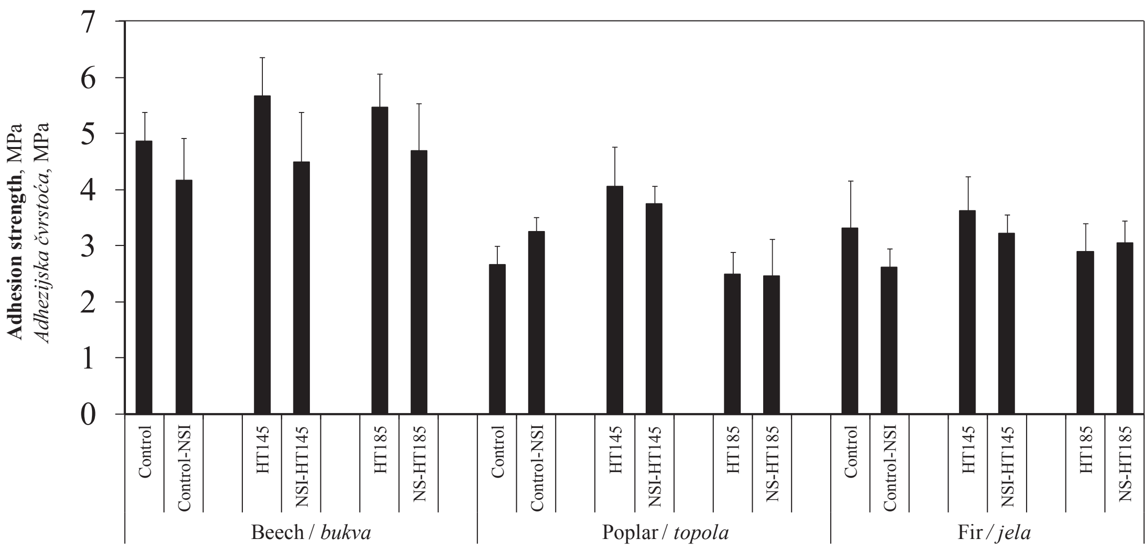

Figure 1. Pull-off adhesion strength (MPa) of three species coated with sealer-clearer
Slika 1. Čvrstoća prianjanja premaza (MPa) izmjerena na uzorcima triju vrsta drva obrađenima sustavom punilo – završni premaz

the pull-off adhesion strength in the case of all treatments of beech specimens; in poplar and fir specimens, however, decrease or increase of the adhesion strength depended on the treatments and species.

Heat-treatment at 145 °C resulted in an increase of the adhesion strength in all three species; in beech and poplar, the increase was significant. Effect of heat-treatment at 185 °C was different according to the species; in beech specimens, it significantly increased the adhesion strength; in fir specimens, it decreased the strength, although not significantly. As to the poplar specimens, the amount of decrease in the adhesion strength was significant.

4 DISCUSSION
4. RASPRAVA

The highest nano-suspension absorption was found in the species with the highest density and permeability (Taghiyari, 2011; 2013). As to the fact that empty-cell process was used for nanosilver impregnation of the specimens, the suspension was absorbed by the cell wall; that is, the cell cavities were ultimately empty at the end of the impregnation process. Therefore, the highest NS-absorption occurred in the case of species that had the highest portion of woody material.

The pull-off adhesion strength of the coating was significantly higher in all treatments of beech specimens. Having significantly higher density in comparison to the other two species, it is shown that the mechanical properties of the substrate significantly affected the pull-off adhesion strength.

Impregnating solid wood species with liquids in pressure vessels was reported to break the cell walls and cause the formation of micro cracks in the cell walls (Taghiyari, 2013; Taghiyari *et al*., 2014), ultimately decreasing the mechanical strength (Taghiyari, 2011). The broken cell parts and distorted perforation plates are shown in Figure 2. The decrease in the pull-off adhesion strengths (statistically significant $P < 0.021$) was related to these micro checks that were formed in the cell walls. On the other hand, the formation of micro checks results in a significant increase in permeability of solid woods (Taghiyari *et al*., 2013); coating pull-off strength was, therefore, expected to increase because coating can more easily penetrate into the texture of the substrate with more permeability, providing mechanical anchoring (Ekstedt, 2002). In fact, two mechanisms were involved in this case. Firstly, the micro checks decreased the mechanical strength of the substrate; secondly, more permeability resulted in possibly better penetration of the coating into the substrate, increasing pull-off strength (Meijer *et al*., 2001a,b). However, in the present case, the decrease in the mechanical properties put the increase in permeability in perspective, eventually decreasing the overall pull-off strength. However, in order to come to a final conclusion, further studies on the depth of penetration of coating into the wood texture of nanosilver-impregnated specimens should be carried out.

Heat-treatment at 145 °C increased the pull-off adhesion strengths in all treatments. This increase was related to the decrease in moisture content (Sönmez *et al*., 2011; Hering *et al*., 2012; Goli *et al*., 2014) as well as the irreversible hydrogen bonding in the course of water movements within the pore system of the cell walls (Borrega and Karenlampi, 2010; Taghiyari *et al*., 2013). To be specific, when water molecules are withdrawn from the cell walls, irreversible bonds are formed among the molecules in the cell wall; these bonds resulted in higher mechanical properties (Taghiyari, 2011). The increase in the mechanical strength of the substrate eventually increased the pull-off adhesion strength due to the fact that all failures actually occurred in the substrate. However, heat-treatment at higher temperatures (higher than 170 °C) was repeatedly reported to decrease the mechanical properties as a result of degradation of hemicelluloses and cell wall components; cell walls are also thinned due to high temperatures (Hill, 2006; Taghiyari, 2011; Taghiyari *et al*., 2013; Taghiyari and Moradi, 2014). The significant decrease of the pull-off adhesion strength of the coating in all three species was related to the degradation of hemicelluloses and the consequent decrease in the mechanical properties of the substrate (Fig. 1).

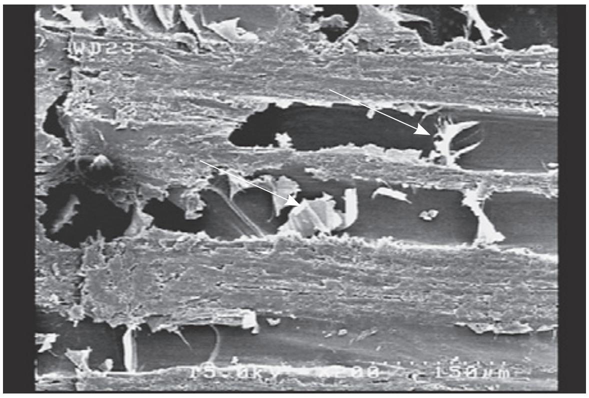

Figure 2 SEM image showing cell parts and perforation plates (↓) distorted along the vessel element
Slika 2. SEM fotografija prikazuje stanične dijelove i perforacije ploče (↓) iskrivljene duž traheje

The lowest pull-off adhesion strength was found in the NS-impregnated specimens heated at 185 °C; this showed the impact of high thermal conductivity coefficient of silver nanoparticles (Saber *et al*., 2013); that is, silver nanoparticles intensified the effects of heat-treatment by rapid absorption of heat. Therefore, the substrate was highly affected by heat-treatment process, especially its surface layer of the specimens to which the pull-off adhesion strength of the coating was most related.

Cluster analysis of the three species based on the six treatments studied in the present study (control, NS-impregnated, HT-145, NS-HT-145, HT-185, and NS-HT-185) showed that beech was clustered differently; poplar and fir species were clustered similarly (Fig. 3). Considering the significantly higher density of beech in comparison to the other two species (poplar and fir), it can be concluded that pull-off adhesion strength was significantly influenced by the density of the species; in fact, the high impact of density on other mechanical properties was also repeatedly reported in

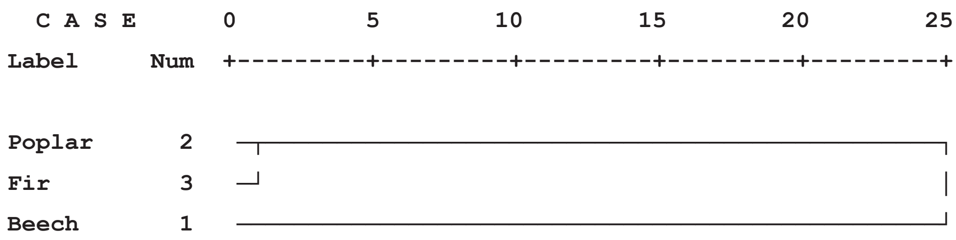

Figure 3 Cluster analysis of three species of beech, poplar, and fir based on pull-off adhesion strengths of six treatments of each species
Slika 3. Klasterska analiza uzoraka triju vrsta drva (bukovine, topolovine i jelovine) na temelju čvrstoće adhezije premaza pri šest različitih obrada tih vrsta drva

many research projects (Taghiyari, 2011; Taghiyari *et al*., 2013).

5 CONCLUSIONS
5. ZAKLJUČAK

1. The pull-off adhesion strength of the coating is closely correlated to the density and mechanical properties of the solid wood substrate.

2. Heat-treatment of solid woods at 145 °C generally tends to increase the pull-off adhesion strength as a result of lower moisture content and irreversible hydrogen bonding in the cell wall. However, heat-treatment at 185 °C significantly decreases the adhesion strength due to degradation of hemi-cellulose in the cell-wall structure of wood.

3. Impregnation with nanosilver suspension significantly decreases pull-off adhesion strength of control and heat-treated solid woods. This decrease is related to the formation of micro checks in cell wall as a result of high pressure in the impregnation vessel, significantly decreasing the mechanical properties of solid woods.

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