Varnish Adhesion Performance of Densified and Thermally Post-treated Beech and Pine Wood

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The adhesion strength was studied for water-based nano-varnishes applied to densified and thermally post-treated beech (*Fagus orientalis* L.) and pine (*Pinus sylvestris* L.) woods. Specimens were thermo-mechanically densified at different compression ratios (20% and 40%) and temperatures (110 °C and 150 °C), and subsequently thermally treated at 190, 200, and 210 °C. One-component (OWB) and two-component (TWB) nano-varnishes were applied, and adhesion strength was evaluated using the pull-off test. Results revealed that the modification processes greatly influenced adhesion, with distinct effects depending on wood species. For untreated beech, densification improved adhesion strength, whereas for pine, it either reduced or did not cause a pronounced change. A primary finding was that thermal treatment decreased adhesion strength for all specimens in a temperature-dependent manner; higher temperatures led to progressively lower adhesion. This decline was more pronounced in densified specimens (especially beech wood). The reason was attributed to the cohesive failure within the weakened wood substrate rather than adhesive failure at the varnish-wood interface. Across all treatment conditions, TWB varnish exhibited superior adhesion compared to OWB. The study concluded that densification may have a species-specific effect, while thermal treatment fundamentally reduces wood surface strength and, consequently, varnish adhesion.

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INTRODUCTION

Wood is a natural, renewable, recyclable, lightweight, aesthetic, and versatile material preferred in construction, furniture, decoration, transportation, energy, and many industrial fields throughout human history. However, despite its many advantages, its hygroscopic, anisotropic, and biologically sensitive nature leads to issues such as dimensional instability, decay, cracking, and surface degradation, which limit its service life and application potential. Many modification methods have been developed to reduce these natural disadvantages of wood and improve its performance (Hill 2006; Sandberg *et al.* 2021; Zelinka *et al*. 2022).

One of these modifications, thermal treatment, is a process carried out in an oxygen-free environment and usually at temperatures between 160 and 220 °C (Esteves and Pereira 2009; Boonstra 2016; Hill *et al.* 2021). Thermal treatment induces permanent alterations in the chemical composition of wood, leading to reduced equilibrium moisture content, enhanced dimensional stability, improved biological resistance, and a decreased susceptibility to natural decay (Militz 2002; Bekhta and Niemz 2003; Esteves *et al.* 2007; Korkut and Guller 2008; Dubey *et al.* 2012; Pelit 2014; Yalçın and Şahin 2015). However, the chemical changes that occur as a result of thermal treatment cause some reduction in the mechanical properties of the wood, which is a constraint that should be carefully considered, especially in applications requiring load-bearing (Bekhta and Niemz 2003; Yıldız *et al.* 2006; Korkut *et al.* 2008; Esteves and Pereira 2009; Percin *et al.* 2015; Pelit *et al.* 2018).

Another common modification method preferred to increase the mechanical and structural performance of wood and to increase the economic and technical value of low-density wood species is densification (Sandberg *et al.* 2013; Song *et al.* 2018; Cabral *et al.* 2022; Luan *et al.* 2022). In this process, the wood material is subjected to mechanical pressure, usually under heat and/or steam, to increase its density, hardness, abrasion resistance, and mechanical strength (Seborg *et al.* 1956; Inoue *et al.* 1993; Navi and Girardet 2000; Kamke and Sizemore 2008). However, one of the most common disadvantages of densified wood is that the material tends to return to its initial dimensions (set-recovery) when exposed to liquid water or humidity (Rautkari *et al.* 2010; Kutnar and Kamke 2012; Cabral *et al.* 2022). This negatively affects the long-term stability of the material, limiting its industrial production and widespread use.

Recently, the combined application of densification and thermal treatment processes (integrated modification) has been reported to have highly beneficial effects on the mechanical properties, biological resistance, dimensional stability, or aesthetic performance of wood (Welzbacher *et al.* 2008; Gong *et al.* 2010; Fang *et al.* 2011; Pelit 2014; Dubey *et al.* 2016; Kariz *et al.* 2017; Pelit and Yalçın 2017; Pelit *et al.* 2018). Although the combined process improves stability and reduces set-recovery, its influence on surface-coating interactions has been largely overlooked. In particular, there has been limited knowledge regarding how integrated modification affects the adhesion behavior of modern water-based varnishes.

The integration of wood with protective coating materials, such as varnish and paint, is of critical importance in terms of expanding the area of use of the material, preserving its surface quality, increasing its aesthetic properties and providing a long service life. The long-term effectiveness of these coatings depends largely on their adhesion strength to the wood substrate. Poor adhesion leads to peeling, cracking, and premature failure of the protective layer (Sönmez and Budakçı 2004; Rowell 2012). Adhesion strength refers to the tensile strength acting perpendicular to the surface plane of the coated wood specimens. This property is influenced by numerous factors, including the porosity and anatomical structure of the wood, surface wettability, capillary action, surface roughness, process history (thermal, plasma, chemical modification, *etc.*), characteristics of the applied coating and the application technique used, and environmental effects (Ozdemir and Hiziroglu 2007; Budakçı and Sönmez 2010; Söğütlü *et al.* 2016; Ghani 2021; Hubbe and Laleicke 2025).

Investigating the performance of protective coatings on densified and thermally treated wood surfaces can contribute to the design and production of higher performance wood products for both structural and decorative uses. This study aims to investigate the effect of different densification and thermal post-treatment conditions on the adhesion performance of water-based nano varnishes applied to beech and pine samples. The study also examined how these modified surfaces interact with coating formulations, comparing the performance of one-component (OWB) and two-component (TWB) varnish systems. The results of the study are expected to optimize the protective coating performance, contribute to the development of sustainable wood products, and fill the current gap in the literature.

EXPERIMENTAL

Wood Material

Eastern beech (*Fagus orientalis* Lipsky) and Scots pine (*Pinus sylvestris* L.) woods were preferred for this study because they are widely used in the woodworking and furniture industry. Beech and pine trees were obtained from Akkuş and Mesudiye Forest Management Directorates in Ordu, Turkey, respectively. Round woods were cut into rough-sized timbers from the sapwood parts on a band sawing machine, taking into account the study methodology. The specimens were dried to approximately 12% moisture content by technical drying and then cut to the dimensions of 450 × 95 mm^2 (longitudinal direction × tangential direction) and three different thicknesses 10 mm (for undensified specimens), 12.5 mm, and 16.7 mm (radial direction). Prior to densification, the wood specimens were conditioned in a chamber (ID 501; Nüve Ind. Mater. Mfg. and Trading Inc., Ankara, Turkey) maintained at 65 ± 3% relative humidity (RH) and 20 ± 2 °C until a constant weight was achieved. Air-dry density values were measured at 688 kg/m^3 for beech and 561 kg/m^3 for pine.

Densification

The wood specimens were densified using the thermo-mechanical (TM) method with a specially designed hydraulic test press (100 T; Hürsan Presser Ind. Inc., Konya, Turkey). Densification was performed at compression ratios of 20 and 40%, with temperatures of 110 and 150 °C. These parameters were selected based on values commonly reported in previous densification studies and widely accepted in industrial applications. The specimens placed in the press machine were kept in this position for a while under slight pressure until the target temperature was reached. The temperature of the specimens was monitored with a digital thermometer. Heated specimens were compressed in the radial direction with a loading speed of 30 mm/min. The targeted compression thickness (10 mm) was achieved using the metal stopping sticks placed at certain intervals on the press tray. Densified specimens were kept under pressure for 10 min and then removed from the press. To minimize the spring-back effect, these specimens were cooled to room temperature under an average pressure of 0.5 MPa.

Thermal Treatment

Thermal treatment of densified and control (undensified) specimens was carried out in three stages: (I) drying, (II) thermal treatment, and (III) cooling/conditioning; according to the procedures described in the *ThermoWood Handbook* (Finnish ThermoWood Association 2003). Wood specimens were thermally treated at three different targeted temperatures (190, 200, and 210 °C) and under the protection of hot water vapor. The thermal treatment duration at the target temperature was 2 h and the total thermal treatment duration was 38 h (Fig. 1). Common industrial applications were influential in determining thermal treatment duration.

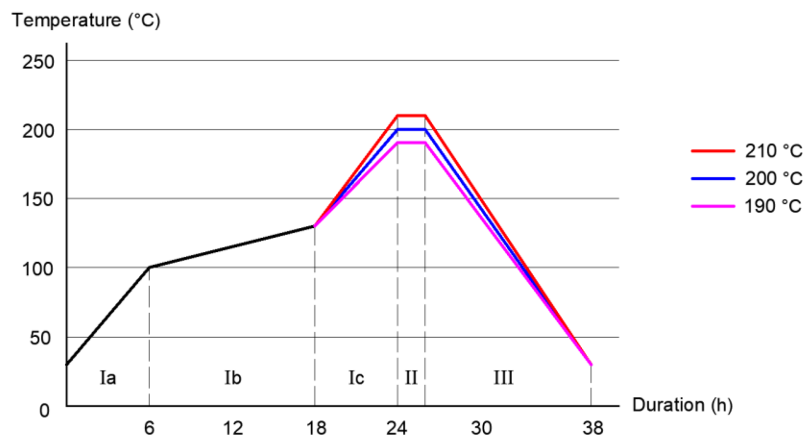

Fig. 1. Thermal treatment processes

After thermal treatment, the wood specimens were kept under normal room conditions for 3 to 4 weeks. The specimens were then cut into pieces measuring 80 × 80 × 10 mm^3 (longitudinal direction × tangential direction × radial direction). For each experimental variable, eight replicates (n = 8) were prepared. In the study, 320 pieces for each wood species and 640 pieces in total were prepared. Then, the specimens were kept at RH 65% ± 3% and 20 °C ± 2 °C until they reached a stable weight. Subsequently, the specimen surfaces were sanded with 150- and 180-grit sandpapers, respectively. Dust was removed using compressed air and a cloth, and the specimens were prepared for varnishing.

Application of Varnishes

One-component (OWB) and two-component (TWB) water-based wood varnishes with glossy properties produced with nanotechnology were used for varnishing the densified and thermal post-treated wood specimens. The varnishes were supplied by Kimetsan company (Ankara, Turkey). Some technical properties of the varnishes are given in Table 1. Varnishing was performed using a spray gun with a 0.8-mm nozzle, at an air pressure of 1 to 1.5 bar, and a spray distance of approximately 20 cm from the specimen surfaces.

Table 1. Some Properties of Water-based Varnishes

Type of Water-based Varnish	pH	Density (g/cm³)	Application Viscosity (sn/DIN Cup 4 mm/20 °C)	Amount of Varnish Applied (g/m²)	Solid Content (%)	Resin	Research Code
Filling	8.1	1.11	18	70	34.2	Acryl copolymer	WBF
One-component (topcoat)	8.1	1.13	18	65	26.9	Acryl copolymer	OWB
Two-component (topcoat)	8.2	1.15	18	75	34.1	Acryl modified polyurethane	TWB

The surfaces of the wood specimens were coated with water-based filling varnish (WBF) three times, with an interval of 1 h between each application. Following a 24-h waiting period, the specimens were sanded with 280-grit sandpaper to smooth out fiber swelling, and the resulting dust was removed using a soft-bristle brush. Then, OWB topcoat varnish was applied to half of the specimens and TWB topcoat varnish to the other half. The OWB application was carried out three times with 1-h intervals, considering the solids content, and TWB application was carried out two times with 1-h intervals. To ensure complete drying of the applied varnishes, all specimens were kept parallel to the ground plane and at room temperature for three weeks.

Determination of Adhesion Strength

The adhesion strength of the varnishes applied to the wood specimens was determined with a pneumatic adhesion tester in accordance with TS EN ISO 4624 (2016). Using a mold, pull cylinders with a 20-mm diameter were bonded to the specimen surfaces at room temperature (20 ± 2 °C). The specimens were maintained in this condition for 24 h. In the bonding process, a two-component acrylic-based adhesive (Penloc-GTI) was applied at a calculated level of 150 ± 10 g/m², which did not dissolve the varnish layer. In the densified but non-thermal treated pine wood specimens, an irregular (wavy) surface was formed after varnishing (Fig. 2). This effect was attributed to the greater spring-back of earlywood compared to latewood following the application of water-based varnishes, particularly in specimens subjected to higher compression (40%). For these specimens that had a wavy surface and were disrupted in terms of smoothness, the amount of adhesive applied to the surface of the pull cylinders was necessarily increased slightly.

Fig. 2. Irregular (wavy) surface formed after varnishing in densified pine specimens

According to the study methodology, because the tangential section of the wood specimens was used, attention was paid to the balancing of early and late wood in bonding the pull cylinders to the varnished specimen surfaces in terms of sensitivity of the tests (Fig. 3). To ensure the rupture originated from the bonded area, the varnish layer surrounding the glued cylinders was cut using a specialized apparatus. In the testing machine, the tensile stress was increased at a constant rate of not more than 1 MPa/s, and the tests were completed within 90 s. The process was repeated for specimens where the pull cylinder did not adhere well in the tests. The adhesion strength of varnished specimens was calculated using Eq. 1,

$$\text{Adhesion strength (MPa)} = 4F / \pi . d^2 \qquad (1)$$

where F is the maximum force at rupture (N) and d is the diameter of the pull cylinder (mm).

Fig. 3. Experimental process of the study

Statistical Analysis

The data were analyzed using MSTAT-C 2.1 statistical software (Michigan State University, East Lansing, MI, USA). Analysis of variance (ANOVA) was realized to determine the effects of densification and thermal post-treatments on adhesion strength of water-based nano-varnishes applied to beech and pine specimens at the 0.05 significance level. After ANOVA, when a significant difference was found between groups, Duncan's one-way tests were applied to determine which groups differed.

RESULTS AND DISCUSSION

The ANOVA results for the adhesion strength of water-based nano-varnishes applied to densified and thermally post-treated beech and pine specimens are presented in Table 2. Table results show that the effects of densification condition, thermal treatment temperature, and varnish type on adhesion strength of wood specimens were statistically significant ($p \leq 0.05$). However, in beech specimens the effect of densification condition was insignificant.

Table 2. ANOVA Results for Adhesion Strength of Water-Based Nano-Varnishes

Source	Beech		Pine	
	F-ratio	p-value	F-ratio	p-value
Densification condition (A)	0.34	NS	7.43	0.000*
Thermal treatment temperature (B)	472.48	0.000*	202.84	0.000*
Varnish type (C)	15.59	0.000*	5.55	0.019*
Interaction (AB)	7.51	0.000*	1.15	NS
Interaction (AC)	0.73	NS	3.31	0.011*
Interaction (BC)	10.59	0.000*	4.58	0.004*
Interaction (ABC)	1.58	NS	2.06	0.020*

* Significant at 95% confidence level; NS: not significant

Table 3. Duncan's One-Way Test Results for Means of Adhesion Strength

Factor	Beech		Pine	
	Mean (MPa)	SD	Mean (MPa)	SD
Densification Condition				
Undensified	2.66 a**	0,63	2.17 a	0.62
110 °C / 20%	2.64 a	0,91	2.02 b	0.60
110 °C / 40%	2.59 a	1,11	2.04 b	0.53
150 °C / 20%	2.62 a	1,01	1.85 c	0.52
150 °C / 40%	2.60 a	0,98	2.02 b	0.55
Thermal Treatment Temperature				
Untreated	3.77 a	0,53	2.58 a	0.34
190 °C	2.97 b	0,48	2.28 b	0.38
200 °C	2.10 c	0,47	1.81 c	0.37
210 °C	1.64 d	0,33	1.41 d	0.32
Varnish Type				
OWB	2.54 b	0,85	1.98 b	0.54
TWB	2.71 a	1,02	2.06 a	0.60

**: statistical group (different letters denote significant differences); SD: standard deviations

With respect to densification condition, the maximum strength average for pine wood was determined in the undensified specimens (2.166 MPa), and the lowest was obtained in the specimens compressed at the ratio of 20% at 150 °C (1.855 MPa). For beech wood, the difference between densification conditions on strength averages was statistically insignificant (Table 3). After densification, the adhesion strength of untreated (without thermal treatment) beech specimens increased depending on the compression ratio. Higher strength values were obtained at higher compression ratio (40%). However, compression temperature had no significant effect on the same specimens (Fig. 4). The increase in adhesion strength of beech samples can be explained by the decrease in surface roughness values due to densification and the formation of strong specific adhesion bonds at the varnish-wood interface. In previous studies, it was reported that the surface roughness of wood decreased after densification processes (Arruda and Del Menezzi 2013; Bekhta *et al.* 2014; İmirzi *et al.* 2014; Pelit *et al.* 2015; Pelit and Arısüt 2023). After densification, the adhesion strength of the untreated pine specimens was found to be similar or lower than the control (undensified) specimens in contrast to the beech wood (Fig. 4). This may be due to insufficient mechanical adhesion between varnish and wood due to the decrease in surface wettability of densified pine specimens. In previous studies, it was emphasized that the wettability of wood decreased after densification (Rautkari *et al.* 2010; Diouf *et al.* 2011; Ünsal *et al.* 2011; Bekhta *et al.* 2017; Pelit and Arısüt 2023). In addition, as explained in detail in the "Determination of adhesion strength" section, densified but untreated (without thermal treatment) pine specimens formed a wavy surface (especially in specimens compressed with 40% ratio) after varnish applications. For these specimens, the amount of adhesive applied to the pull cylinder in adhesion tests may have been more than necessary, which may have affected the results. The contrasting behavior observed between the two species may also be related to inherent anatomical and chemical differences. Beech, as a diffuse-porous hardwood with a relatively uniform cellular structure and lower extractive content, may undergo more even compression, which could contribute to smoother surfaces and possibly facilitate better interaction with the varnish. In contrast, pine contains alternating earlywood–latewood bands and higher amounts of resinous extractives. During densification, earlywood sections may compress more than latewood, potentially leading to surface irregularities that limit consistent mechanical interlocking. Additionally, extractive migration toward the surface may reduce wettability and interfere with adhesion. These factors may help to account for the differing adhesion responses between beech and pine. The fact that only two types of wood were tested in the study limits the generalizability of the findings. In a previous study, Atilgan *et al.* (2024) evaluated the adhesion strength of polyurethane and cellulosic varnishes applied to thermo-mechanically densified black pine and fir wood. The findings indicated that densification enhanced varnish adhesion strength in black pine samples, whereas a reduction was observed in fir wood. Moreover, the influence of compression ratio (25% or 50%) on adhesion strength was reported to be statistically insignificant.

Regarding thermal treatment temperature, the highest adhesion strength average for both wood species was obtained in the untreated specimens (3.77 MPa for beech and 2.58 MPa for pine), while the lowest was determined in the specimens thermally treated at 210 °C (1.64 MPa for beech and 1.41 MPa for pine) (Table 3). Adhesion strength values decreased in both wood species and in all groups (densified and undensified) subjected to thermal treatment. Strength values also decreased with increasing thermal treatment temperature (Fig. 4). In the pull-off tests performed to determine adhesion strength, the ruptures in thermally treated beech and pine specimens were in the form of fiber rupture from the wood (Fig. 5). This can be attributed to damage to intermolecular bonds due to the thermal degradation of the chemical components of wood under the influence of high temperatures. Hemicelluloses are considered the most thermally sensitive polysaccharides in wood and have been reported to degrade at relatively low temperatures (Esteves and Pereira 2009; Hill *et al.* 2021). Hence, treated wood may have reduced surface flexibility and bonding capacity. At higher treatment temperatures, partial depolymerization of cellulose and condensation reactions in lignin were also reported (Tjeerdsma and Militz 2005; Hill 2006), which may further weakened internal cohesion. This situation indicates that the adhesion bonds between varnish and wood are higher than the internal cohesive bonds of the thermally treated wood. Similar results were also reported in a previous study (Pelit *et al.* 2023). In different studies examining varnish adhesion strength for thermally treated wood, it was also reported that adhesion strength decreases due to an increase in thermal treatment temperature or duration (Atar *et al.* 2015; Kesik and Akyıldız 2015; Gurleyen *et al.* 2019; Krystofiak *et al.* 2022; Pelit *et. al.* 2023).

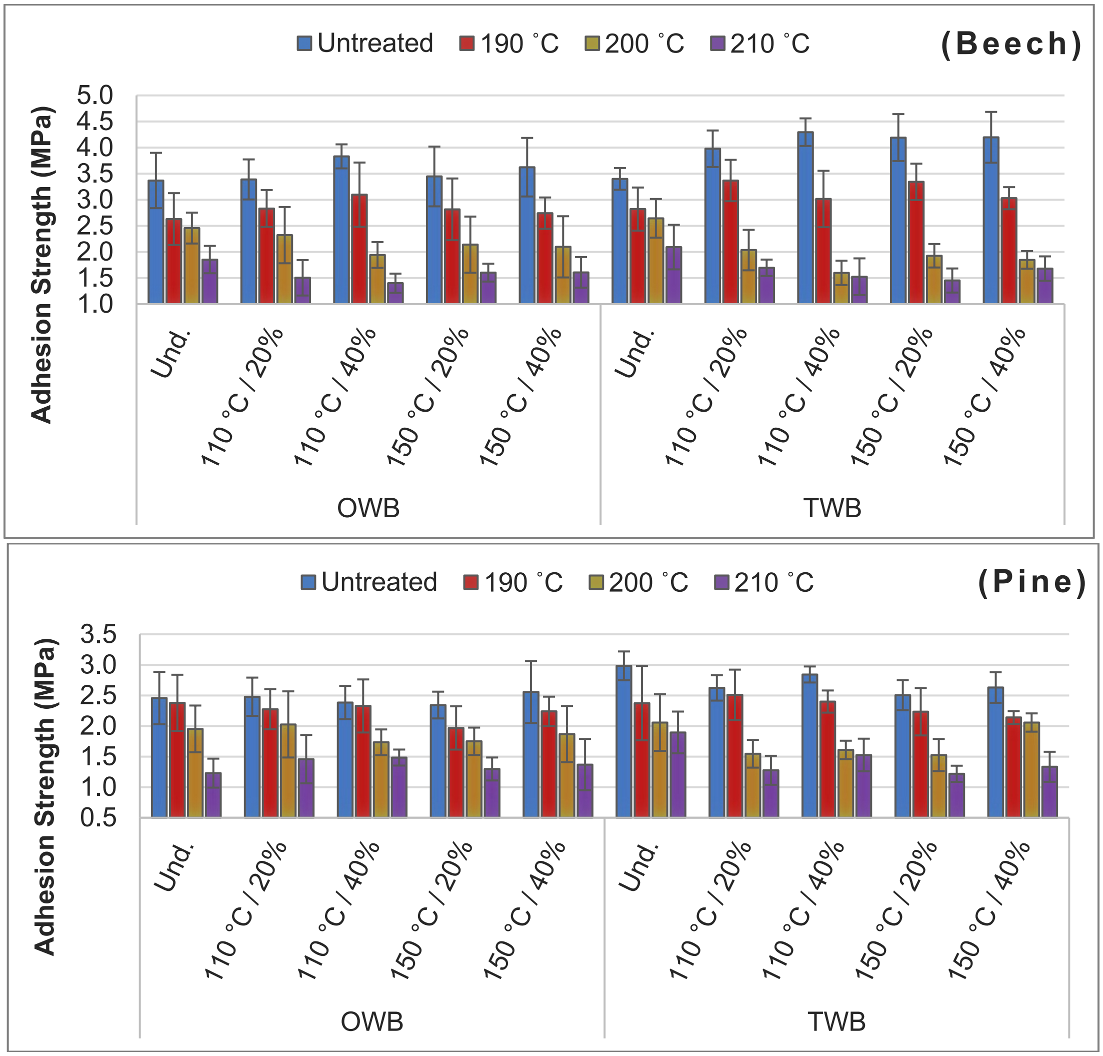

Fig. 4. Adhesion strength of water-based nano-varnishes applied to densified and thermally post-treated beech and pine specimens

The adhesion strength of the densified and thermally treated specimens at high temperatures (200 and 210 °C) was generally lower than that of the undensified specimens thermally treated at the same temperatures. This situation is especially evident in the beech specimens (Fig. 4). In addition to deformations, such as fractures and cracks, that occur in the cell structure of the wood after mechanical densification, thermal degradations in the polymeric components of the wood due to thermal treatment are thought to influence the results. It is stated in the literature that thermal treatment applied to densified wood causes more degradation of wood polymers than natural wood (Dwianto ve ak 1997; Hsu *et al.* 1988; Dubey 2010).

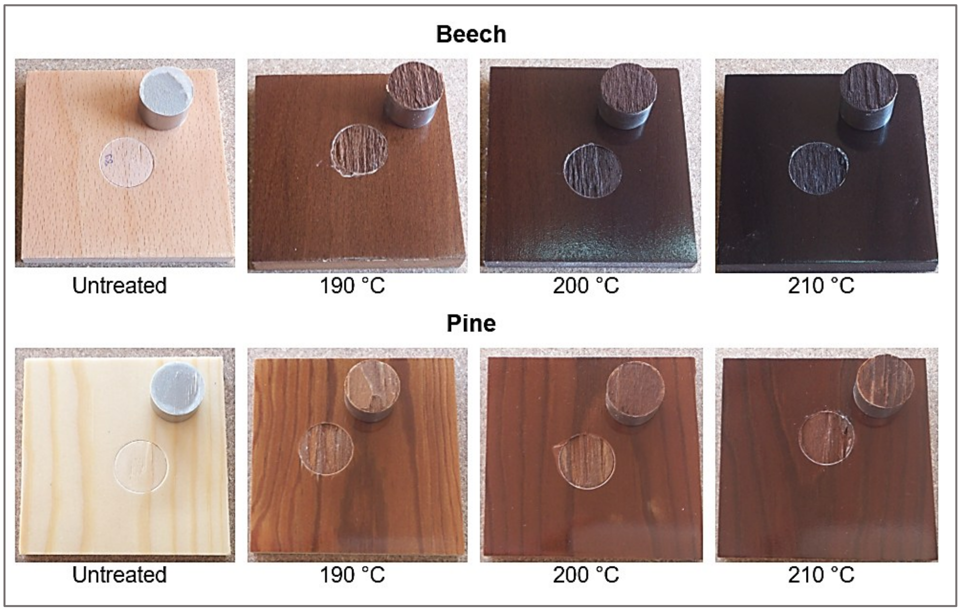

Fig. 5. The appearance of untreated and thermally treated beech and pine specimens after the adhesion strength test

The average adhesion resistance regarding varnish type was higher in TWB applied specimens compared to OWB applications for both wood species (Table 3 and Fig. 2). Different resin types and drying mechanisms of both varnish types may affect the results (Table 1). In addition, the results indicate that the adhesion bonds between the varnish layer and the wood material are stronger in TWB.

From a practical standpoint, these findings have significant implications for the use of densified and thermally modified wood in finished products. For example, the higher adhesion performance of TWB coatings, particularly on densified beech wood, suggests strong potential for use in furniture components and interior flooring where abrasion and delamination resistance are critical. Conversely, the marked decrease in adhesion strength, particularly at treatment temperatures of 200 °C and above, indicates a weakening of the wood substrate itself. For high-wear applications such as flooring, this compromised surface integrity could lead to premature failure of the protective varnish, diminishing the product's service life and aesthetic appeal. Similarly, in furniture manufacturing, where a flawless and durable finish is paramount, using wood treated at high temperatures could result in coating delamination or flaking. Therefore, it can be said that it is important to understand the interaction between modification level and coating formulation to design durable wood products in different service environments.

CONCLUSIONS

The adhesion strength of water-based nano-varnishes applied to densified and thermally post-treated beech and pine wood surfaces was investigated in the presented study. The findings were as follows:

1. In untreated (without thermal treatment) beech wood, the densification process improved the varnish adhesion strength. Additionally, higher compression ratio provided higher adhesion strength. However, no significant effect of compression

temperature on adhesion strength was observed. In untreated pine wood, densification either kept the adhesion strength at the same level or caused a decrease. This situation reveals that densification may have different effects depending on the wood species.

2. In both beech and pine wood, thermal treatment decreased the adhesion strength in all densified and undensified groups. Increasing the thermal treatment temperature made the decrease in adhesion strength even more pronounced.
3. The adhesion strength of densified and thermally treated specimens at high temperatures (200 °C and 210 °C) was generally lower than that of thermally treated but undensified specimens at the same temperatures. This effect was observed more clearly in beech wood.
4. In both wood species, two-component (TWB) coatings provided higher adhesion strength compared to one-component (OWB) coatings.
5. It was found that densification and thermal treatment processes significantly affected the adhesion strength of varnishes on wood materials. In particular, the adhesion strength-reducing effect of thermal treatment may limit the areas of use of such applications or require special precautions to be taken. Because the effect of densification varied depending on the type of wood, the properties of the material should be considered before application. In addition, the choice of varnish type stands out as an important factor in terms of adhesion performance.
6. In future studies, the effects of different densification parameters (*e.g.*, different compression ratios and times) and thermal treatment conditions (*e.g.*, different combinations of temperature and duration) can be investigated in more detail. Furthermore, the generalizability of these findings can be tested using different wood species and varnish types. Such studies will provide important information to optimize the performance of wood materials and make them suitable for different applications.

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