

Effect of the Mechanical Densification Process in Wood Material on the Surface Adhesion Strength of Varnishes

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This research aimed to determine the impact of the mechanical densifying process of wood material on the varnish surface adhesion strength. Specimens from black pine (*Pinus nigra*) and Uludag fir (*Abies bornmuelleriana* Mattf.) were subjected to densification in a hydraulic press at 140 °C to the extent of 25% and 50% in the radial direction. While densification increased the surface adhesion strength of the varnish layer in black pine, the value decreased in fir. Regarding the interaction between densification ratio, surface treatment, and wood type, the highest surface adhesion strength of the varnish layer was found in black pine + unsanded surface + 25% densification, and the lowest was in Uludag fir + unsanded surface + 50% densification. It can be stated that the densification process creates high adhesion values for the polyurethane varnish in the black pine wood type. The sanding process has an intensifying effect on these values, and the products that were obtained from the polyurethane varnished samples do not require sanding. Considering these situations can provide significant advantages in projects with wood materials subjected to the densification process.

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INTRODUCTION

Wood material is one of the most essential natural raw material resources used by human beings. The factors that give the tree this essential place as a renewable resource can be listed as its widespread availability, its physical-mechanical properties suitable for its usage areas, and its ability to improve these properties with some constructive and physical effects (Bozkurt and Goker 1996).

The organic, hygroscopic, anisotropic, and heterogeneous structure of the wood material can make it susceptible to biotic and abiotic effects, including fungi, insects, and dimensional problems in many areas of usage unless precautions are taken for these (Yasar *et al.* 2016; Atilgan *et al.* 2022). Processes conducted to improve some properties of wood material can be called “wood modification methods”. In usage areas in buildings where high durability, resistance, and hardness are required and other usage areas, the properties of wood material may be insufficient. Increasing the density is a commonly used modification method to reinforce those properties (Homan *et al.* 2000; Blomberg and Persson 2004; Kutnar and Šernek 2007; Rautkari *et al.* 2009; Laine *et al.* 2013; Laine 2014; Onduran *et al.* 2017; Sandberg *et al.* 2017; Yasar and Altunok 2023). Wood of lower

density can be transformed into high strength material, which can be made into valuable products by compacting low density.

In this study, black pine (*Pinus nigra*) and Uludag fir (*Abies nordmanniana* subsp. *bornmuelleriana*) tree species were modified using an environmentally friendly modification method. It was densified by using the Thermo-mechanical (TM) method at 140 °C and by compressing it at two different percentage levels (radial direction) of 25% and 50%. After densification, the springback amount was determined. According to the results, tree type was found to affect the extent of springback. Volumetric recovery (spring-back) has been determined as 15.44% in Uludag fir and 19.40% in larch (Atilgan 2023). The idea of compressing wood material by heating and applying pressure has been known for more than a century. In addition, the process of compacting wood in the radial direction has been available in the literature since 1886 (Kollmann and Côté 1968; Heger *et al.* 2004).

With the increase in solid wood material density, a decrease in the empty volume and an increase in the strength properties occurs (Ulker *et al.* 2012). Densification improves the mechanical properties of wood material and decreases its hygroscopicity (Arruda and Menezzi 2013). Some properties of wood material can be enhanced with the processes of densifying (Kutnar and Sernek 2007). In the densification process through compression, the wood material's cell wall collapses, and densification occurs with the empty volume becoming smaller (Kutnar *et al.* 2009). In densification through compression, the density value increases, roughness occurring in surface and wetting capability lessens, roughness rises, both mechanical and physical features enhance, and some spring-back may occur (Tosun and Sofuoğlu 2021). When it is considered that high temperature has some heat treatment effect in thermo-mechanical densification, it can be stated that it contributes to dimensional stability (Percin *et al.* 2016). The densification through compression has a drawback that the wood returns to its previous proportions when it is subjected to a high relative humidity or submerged in water (Seborg *et al.* 1956; Kollmann *et al.* 1975; Kultikova 1999; Morsing 2000; Blomberg *et al.* 2006; Gong and Lamason 2007; Pelit 2014; Pelit *et al.* 2014).

The comprehension of wood adhesive bonds requires both an understanding of the uniqueness of the wood structure for bond formation and an understanding of the modes of energy dissipation. To understand the adhesive interaction with the wood, one needs to consider in more detail the aspects of surface preparation, types of wood surfaces, and spatial scales of wood surfaces (Frihart 2005). Most observations of adhesive interaction with wood are concentrated on scales of millimeter or larger (Marra 1992).

In some low specific weight trees, the thermo-mechanical method, which is an environmentally friendly treatment, improves their physical and mechanical properties. Coating wood with varnishes can reduce the spring-back feature that may occur when exposed to moisture and it can also increase water repellency and dimensional stabilization. Wood material begins to deform when exposed to factors that may lead it to decompose. Coating wood with varnishes is one of the most common methods to eliminate this situation. Adhesion strength in densified and surface-treated wood is widely utilized to determine layer performance. Varnish type is one of the most significant reasons for the difference in the surface adhesion strength of varnishes. The surface adhesion strength of a varnish indicates the effectiveness of the varnish. The cross-cut test, tape-peel test, and pull-off test methods can be utilized to determine adhesion strength (Vitosytė *et al.* 2012). Adhesion is among the most complex factors that are used to specify the performance of protective layers in a long term (Williams *et al.* 1987, 1990).

It has been determined that the increase in the film layer of the varnishes (in polymer-based varnishes) is parallel with the increase in the surface adhesion strength (Budakci 1997). In the literature, it has been stated that adhesion in polyurethane varnish gives better results than other varnishes (Sonmez 1988). In another research, Yang *et al.* concluded that high values have been determined for adhesion strength on sanded surfaces (Yang *et al.* 2012). Significant increases from 12% to 25% were achieved in varnish adhesion strength values of beech (*Fagus orientalis* L.) wood samples with nano-graphene modified water-based varnish application (Pelit and Korkmaz 2019). In their study, Atar and Peker (2010) studied the adhesion resistance properties of varnishes applied to different wood species impregnated with boron compounds. As a result, they stated that as the impregnation solution intensified, the adhesion resistance properties increased. In the study conducted by Kilic and Sogutlu (2023), polyurethane, acrylic and water-based varnishes were applied to the surfaces of oak (*Quercus petraea* Liebl.), chestnut (*Castanea sativa* Mill.) and Scots pine (*Pinus sylvestris* L.) woods. It was determined that the age period did not affect the varnish adhesion strength. The highest adhesion strength at the wood species level was obtained in chestnut (3.4 N/mm²), followed by oak (3.2 N/mm²) and Scots pine (2.2 N/mm²). At the varnish type level, the highest adhesion strength was obtained in polyurethane varnish (4 N/mm²), followed by acrylic varnish (3.7 N/mm²) and water-based varnish (1.3 N/mm²), respectively. In a study, polyurethane, water-based, acrylic, and cellulosic varnishes were put on with different thicknesses in layers of coniferous and broad-leaved tree wood materials (Budakci and Sonmez 2010). As a result, higher adhesion strength values were determined in broad-leaved tree wood and lower adhesion strength values were determined in coniferous woods. With respect to the varnish type, the adhesion strength was highest in acrylic and polyurethane varnishes.

The increase in the layer thickness of polymer-based varnishes has been stated to have an impact on increasing the surface adhesion strength (Budakci 1997), and that the roughness, gloss and surface adhesion strength of various kinds of water-based varnishes, which are applied with varied methods on different types of wood, have lower values than solvent-based varnishes (Yakin 2001). It has been reported that reducing the surface roughness of the wood material and removing the air at the junction of the protective layer and the wood material from the adhesion surface is essential for high adhesion strength (Payne 1965; Kurtoglu 2000; Sonmez 2000).

Cellulosic (one component) and polyurethane (double component) varnishes form a protective layer. They are preferred in the industry because they are economical and quick drying, and they have easy sanding and high-performance resistance. This study aimed to reveal the effects of cellulosic and polyurethane varnishes on the surface adhesion strength after the densification process conducted on black pine (*Pinus nigra*) and Uludag fir (*Abies nordmanniana* subsp. *bornmuelleriana*), which are partially low-density coniferous trees.

EXPERIMENTAL

In this study, black pine (*Pinus nigra*) and Uludag fir woods (*Abies nordmanniana* subsp. *bornmuelleriana*) that are commonly preferred in the Turkey woodworking industry with their lower densities, were chosen as the test samples. Wood materials were obtained from the local timber management Avcioglu Kereste Ind. Trade from the city of Afyonkarahisar as lath from the timbers, with random selection method in accordance with ISO 3129 (2019) principles. Attention was paid to ensure that the wood material was free

of rot, knots, and gaps and had smooth fibers. Afterwards, these timbers were dried in an automatically managed drying oven until an average of 12% moisture was reached.

Cellulosic (one-component) and polyurethane (two-component) varnishes, which are the most widely used in the industry, were preferred for varnishing the test samples. Varnish types were obtained from the Polisan Company.

Sapwood was utilized to prepare the experiment samples in accordance with ISO 3129 (2019) standards, which state that wood is not damaged by insects and fungi, the wood is free of knots, cracks and arcs, and any discoloration, but has smooth fiber structure and annual rings parallel to the surface. To reach a sample thickness of 10 mm after densification, the test samples were cut into rough thicknesses of 13.33 mm and 20 mm. The measurements and data belonging to the densification process of the experiment samples are given in Table 1.

Table 1. Information on the Densification Process

Press Temperature	Compression Ratio	Press Duration	Dimension (mm)			
			Length (Longitudinal Direction)	Width (Radial Direction)	Thickness (Tangential Direction)	Targeted Thickness
140 °C	Control	-	500	100	10	
	25%	Heating +15 min	500	100	13.33	10
	50%	Heating +15 min	500	100	20	10



Fig. 1. Densification press table

The densification process was carried out on test materials using a specially designed resistance hydraulic press equipped with pressure and temperature controllers. The press had a capacity of 100 tons and could reach up to 250 atm, with a loading speed of 60 mm/min. The thermo-mechanical (TM) method was employed, and the materials were densified by compressing them in the radial direction at controlled rates of 25% and 50%. This was done in a press set to a table temperature of 140 ± 5 °C (Fig. 1).

To achieve the targeted compression ratios, 10-mm-thick metal stopper profiles were placed on the press table at regular intervals. The compressed test samples were held

under pressure for a duration of 15 min. After that, the samples were removed from the device and left to cool down to room temperature with an average pressure of 5 kg/cm² to reduce the effect of spring-back. The trial design determined for the test and its variations is shown in Table 2.

Table 2. Experiment Trial Design

Wood Type	Compression Ratio (%)	Varnish Type	Sanded (n = 10) Repeated	Unsanded (n = 10) Repeated	Measurements (mm)	Total (pcs)
Black Pine	%0 (control)	Cellulosic	10	10	100 × 100 × 10	4 × 10 = 40
		Polyurethane	10	10		
	% 25	Cellulosic	10	10	100 × 10 × 13.33	4 × 10 = 40
		Polyurethane	10	10		
	% 50	Cellulosic	10	10	100 × 100 × 20	4 × 10 = 40
		Polyurethane	10	10		
Uludag Fir	%0 (control)	Cellulosic	10	10	100 × 100 × 10	4 × 10 = 40
		Polyurethane	10	10		
	% 25	Cellulosic	10	10	100 × 10 × 13.33	4 × 10 = 40
		Polyurethane	10	10		
	% 50	Cellulosic	10	10	100 × 100 × 20	4 × 10 = 40
		Polyurethane	10	10		

After the densification process, the samples that would be sanded were sanded with 80 and 120 grit sandpaper. In the varnishing of the samples, three layers of filler varnish were applied at a calculation of 125 g/m² at 1-h intervals following the manufacturer's recommendations. After the 24-h wait time, it was sanded with 220-grit sandpaper to remove fiber bubbles and ensure surface smoothness. Then, dust was removed with a soft bristle brush, and a topcoat varnish was applied. The varnishes were applied at a height of approximately 30 cm from the sample surfaces with a spray gun at room temperature (~20 °C) and the sprayed amount on the surface was determined by weighing with an analytical scale that has a precision of ± 0.01 g. The varnished test samples were left to dry in a position parallel to the ground under laboratory conditions at a room temperature of 20 ± 2 °C. The ASTM-D 3023 (2017) principles were followed in the varnishing process.

The surface adhesion strength was elucidated in the adhesion test device for varnish layers following the principles of TS EN ISO 4624 (2016). Thus, “Penloc-GTI” brand adhesive, which has two components and high adhesion strength, and is acrylic-based, non-solvent, was applied to varnished samples and Ø 20 mm drawing rollers, on varnish layers in a ratio of 150 ± 10 g/m². It was applied at a temperature of (20 ± 2 °C) using a special apparatus and left to dry for 24 h (Fig. 2). The perimeter of the test cylinder was cut up to the wood material surface with the help of a cutting apparatus, and only the layer on the adhesion surface of the cylinder was forced to be removed. During the tests, care was taken to increase the tensile stress at a constant rate of not more than 1 MPa/s and to complete the test within 90 s, the varnish layer was removed and the force at break was recorded.

Adhesion strength (X) measurements were calculated according to Eq. 1,

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

where F is the force at the break (Newton) and d is the test cylinders diameter (mm).

The results were studied with a computerized MSTAT-C 2.1 statistical program (Michigan State University, East Lansing, MI, USA) that included the analysis of variance

(ANOVA) and the Duncan test applied at a 95% confidence level. Statistical evaluations were made on homogeneity groups (HG) according to the least significant difference (LSD) critical value, where different letters reflect statistical significance.

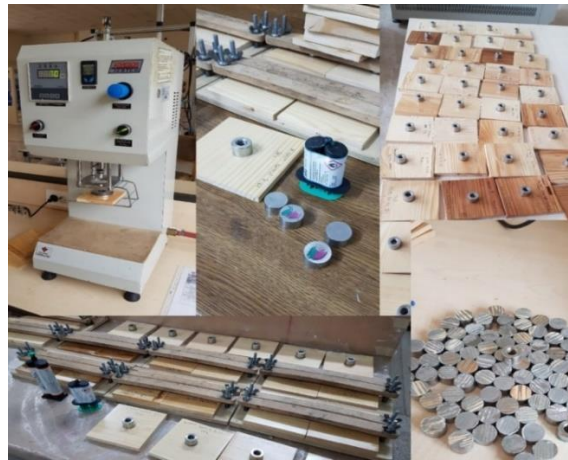


Fig. 2. Adhesion test device and the process of sticking the cylinder to the layer (Atilgan 2023)

RESULTS AND DISCUSSION

An analysis of multiple variances was performed to find the impact of densification ratio, wood type, sanded and unsanded surfaces, varnish type, and their interactions on the surface adhesion strength of varnishes (Table 3).

Table 3. Multiple Variance Analysis for Adhesion Strength of Varnishes

Source	Degrees of Freedom	Sum of Squares	Mean Squares	F-value	Sig.
Wood Type (A)	1	2520.833	2520.833	17.4982	0.0001*
Densification Ratio (B)	2	45.017	22.508	0.1562	0.0000*
Interaction (AB)	2	4188.217	2094.108	14.5361	0.0000*
Varnish Type(C)	1	6840.300	6840.300	47.4815	0.0000*
Interaction (AC)	1	616.533	616.533	4.2796	0.0413*
Interaction (BC)	2	657.950	328.975	2.2836	0.1074
Interaction (ABC)	2	740.017	370.008	2.5684	0.0819
Surface Treatment (D)	1	580.800	580.800	4.0316	0.0475*
Interaction (AD)	1	80.033	80.033	0.5555	0.0000*
Interaction (BD)	2	1234.350	617.175	4.2841	0.0165*
Interaction (ABD)	2	2013.217	1006.608	6.9873	0.0015*
Interaction (CD)	1	2201.633	2201.633	15.2825	0.0002*
Interaction (ACD)	1	433.200	433.200	3.0070	0.0861
Interaction (BCD)	2	2091.817	1045.908	7.2601	0.0012*
Interaction (ABCD)	2	505.550	252.775	1.7546	0.1785
Error	96	13830.000	144.063		
Total	119				

*: Statistically significant difference $\alpha \leq 0.05$

When the results of the multivariate analysis are analyzed, the adhesion strength of the varnish layers was found insignificant in the densification process and varnish type

interaction, the wood type-densification-varnish type trio (ABC) interaction, the wood type-varnish type-surface treatment (ACD) trio interaction, and the quaternary factors (ABCD) interaction; others and their relations were determined as significant ($P \leq 0.05$). The values belonging to the effects of varnishes on adhesion strength with respect to surface treatment, wood type, densification ratio, and varnish type are given in Table 4.

Table 4. Mean Values of Surface Adhesion Strength

Tree Type and Abbreviations ¹	\bar{x}	HG	Varnish Type ³	\bar{x}	HG
Black Pine (BP)	1.63	A	Cellulosic (Cl)	1.34	B
Fir (F)	1.34	B	Polyurethane (Py)	1.63	A
Densification Ratio ²			Surface Treatment ⁴		
% 25 (I)	1.50	A	Sanded (Sn)	1.53	A
% 50 (II)	1.47	A	Unsanded (Un)	1.40	B
LSD ¹ : ± 0.0138 , LSD ² ± 0.0169 , LSD ³ ± 0.0138 , LSD ⁴ : ± 0.0138					

\bar{x} : Arithmetic mean, HG: Homogeneity group

The surface adhesion strength with regard to the factors was as follows; the highest values were found in black pine, polyurethane varnish, sanded surface, whereas the lowest values were found in fir and cellulosic varnish. According to the single factors, the surface adhesion strength of varnishes was 18% higher in Black Pine than in fir and in polyurethane varnish than cellulosic varnish. Varnishes, except oil-based ones, increased the hardness of the wood surface. The greater penetration of cellulosic varnish into the wood surface also reduces the layering rate, hardness, and adhesion value of the varnish. The reason why polyurethane varnish provides higher adhesion strength may be its smaller molecule size, high layering feature, and pH level being closer to the wood material (Yakin 2001; Budakci and Sonmez 2010; Ceylan, 2016; Sogutlu *et al* 2016; Yalcin and Ceylan 2017; Pelit *et al* 2023). Sanding the wood material increased the wetting and adhesion (Sakata *et al.* 1993; Frihart 2005). Values were found to be approximately equal in the densification process. The values of the surface adhesion strength regarding the bilateral interactions of the wood type, the varnish type, the densification ratio, and the surface treatment are shown in Table 5.

Table 5. Adhesion Strength Values Concerning the Bilateral Interactions

Factors			Factors		
Wood + densification1 (AB)	\bar{x}	HG	Densification + surface treat. 4 (BD)	\bar{x}	HG
Bp+I	1.75	A	I+ Sn	1.43	AB
Bp+II	1.78	A	I+ Un	1.56	A
F+I	1.24	C	II+ Sn	1.59	A
F+II	1.18	C	II+ Un	1.37	AB
Wood + varnish2 (AC)			Varnish + surface treat. 5 (CD)		
Bp+Cl	1.31	BC	Cl + Sn	1.18	C
Bp+Py	1.94	A	Cl + Un	1.31	C
F+Cl	1.30	BC	Py + Sn	1.91	A
F+Py	1.50	B	Py + Un	1.50	B
Wood + surface treat. 3 (AD)					
Bp+Sn	1.66	A	F+Sn	1.43	BC
Bp+ Un	1.56	AB	F+ Un	1.24	C
LSD1 : ± 0.0239 , LSD2: ± 0.0195 , LSD3: ± 0.0195 , LSD4: ± 0.0239 , LSD5: ± 0.0195					

Regarding the interaction of wood type and densification ratio, the highest adhesion strength of the varnish layer was found in Bp+II (1.78) and the lowest value was F+II (1.18). While the densification process increased the surface adhesion strength of the varnish layer in Black pine, it decreased in fir. The interaction of varnish type and wood type was highest in Bp+Py (1.94) and the lowest values, Bp+Cl and F+Cl (1.30, 1.031), were approximately equal. Thus, it can be stated that the varnish type has a first-degree effect, and the wood type has a second-degree effect on the interaction of the factors.

Concerning the interaction of wood type and surface treatment, the highest surface adhesion strength of the varnishes was found in Bp+Sn (1.63) and the lowest was in F+Un (1.24). Thus, it can be stated that the wood type had a first-degree effect, and the surface treatment had a second-degree effect on the interaction of the factors. Hence, when the obtained results are studied, both high surface adhesion values were obtained in black pine.

Regarding the interaction of densification and surface treatment, the highest surface adhesion strength was found in II+Sn (1.59) in the varnishes and the lowest was in II+Un (1.37). Thus, different results were obtained in the interaction of factors. While sanding increased the adhesion strength, the opposite occurred for unsanded surfaces.

Regarding the interaction of varnish type and surface treatment, the highest surface adhesion strength in varnishes was found in Py +Sn (1.91) and the lowest was in Cl+Un (1.18). Polyurethane varnish created lower surface roughness than water-based and cellulosic varnish (Togay *et al.* 2009; Yalcin and Ceylan 2017). Thus, it can be stated that the varnish type has a first-degree effect on the interaction of the factors and the surface treatment has a second-degree effect. The triple interaction values belonging to the effects of densification ratio, surface treatment, and wood type on the adhesion strength of varnishes are shown in Table 6.

Table 6. Values for the Adhesion Strength Concerning the Triple Interactions

Factors			Factors		
Wood type + surface treatment + densification ratio ¹	\bar{x}	HG	Varnish type + densification ratio + surface treatment ²	\bar{x}	HG
Bp + Sn	1.66	ABCD	Cl + Sn	1.02	F
Bp + Un	1.02	FG	Cl + Un	1.34	DEF
Bp + Sn + I	1.53	BCDE	Py + Sn	2.23	A
Bp + Un + I	1.98	A	Py + Un	1.24	DEF
Bp + Sn + II	1.82	AB	Cl +I+ Sn	1.05	EF
Bp + Un + II	1.75	ABC	Cl +I+ Un	1.31	DEF
F+ Sn	1.59	BCD	Py +I+ Sn	1.79	B
F + Un	1.56	BCD	Py +I+ Un	1.82	B
F + Sn +I	1.34	DEFG	Cl +II+ Sn	1.43	CDE
F + Un +I	1.18	EFG	Cl +II+ Un	1.24	DEF
F + Sn +II	1.37	CDEF	Py +II+ Sn	1.75	BC
F + Un +II	0.99	G	Py +II+ Un	1.47	BCD

LSD¹: ± 0.0338, LSD²: ± 0.0338

Regarding the wood type, density ratio, and surface treatment interaction, the highest surface of the varnish layer was found in Bp+Un+I (1.98) and the lowest was in F+Un+II (0.99). While densification and sanding increased the surface adhesion strength of the varnish layer in Black Pine, it decreased in fir wood. Regarding the interaction of varnish type, densification ratio, and surface treatment, the highest surface adhesion strength of the varnish layer was found in Py + Sn (2.23), and the lowest was in Cl + Sn (1.02).

CONCLUSIONS

This study considered the effect of cellulosic and polyurethane varnishes on the adhesion strength of the sanded and unsanded samples prepared from black pine and fir. Densification levels of 25 and 50% by thermo-mechanical method were investigated. The results show that the densification process significantly affected the varnish adhesion strength, and remarkable differences are observed between wood types and the varnish process.

1. The surface adhesion strength of varnishes was higher in Black Pine than fir, in polyurethane varnish than cellulosic varnish. This may be due to black pine forming a smoother surface. The sanded surface gave 8.5% higher results than the unsanded surface. It is assumed that this originates from the fact that the sanding process creates smoother surfaces, thus increasing the strength of adhesion between the varnish and wood. It was observed that no statistically significant difference occurred in terms of varnish adhesion strength with respect to the densification ratio. In the densification process, the formation of a more covered surface in the wood material can create a composition that prevents mechanical adhesion between the varnish and the wood. In this respect, scientific studies are considered essential for other materials and processes (glue, paint, lacquer, *etc.*) that are believed to provide specific and mechanical adhesion with wood material.
2. While densification increased the surface adhesion strength of the varnish layer in black pine, it decreased in fir. Thus, it can be stated that the wood type had a first-degree effect, and the densification had a second-degree effect on the interaction of the factors. In addition, lower values occurred in densified fir samples when they were compared with the control samples, and higher values in black pine were assessed as important results that should be considered. These may have originated from the smoother surface, which occurs after densification in black pine wood, increasing the specific adhesion.
3. It can be stated that the wood type had a first-degree effect, and the surface treatment had a second-degree effect on the interaction of the factors. When the obtained results were studied, both high surface adhesion values were obtained in black pine. This result, which emerged in the densification process, was found to be significant.
4. While densification and sanding increased the surface adhesion strength of the varnish layer in black pine samples, they decreased it in fir wood. This result in fir was regarded worth considering in terms of production.
5. Regarding the triple interaction, the surface adhesion strength of the varnish layer was found to be lower than the control samples with polyurethane. However, it gave higher values than the unsanded control sample with polyurethane. It has been observed that sanding and densification processes have shown an increasing effect in cellulosic varnish samples, and it can be essential to consider this situation in wood-based projects where cellulosic varnish will be used.
6. It is stated that the densification process creates positive high adhesion values for polyurethane varnish in Black Pine wood and the sanding process also increases

these values. The results obtained from the polyurethane samples show that sanding is not needed.

7. In this study, it was observed that the wettability properties of wood are an important parameter especially in terms of hot pressing and sanding. In future studies, it is recommended to conduct a new study with different parameters.

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