Adhesive Type's Effects on Adhesive Strength of Densified Reinforced Laminated Wood Obtained from Black Poplar (*Populus nigra* L.)

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Wood material is the most critical indoor and outdoor building element that has not changed since ancient times. Previous studies have determined that the mechanical properties of tree species with low industrial importance, such as poplar wood, can be improved when they are subjected to the densification process. In addition, it has been determined in studies that the lamination process has a positive effect on the mechanical properties of the wood material. This study aimed to assess the impact of the glue type on the bonding strength during the lamination process of the densified black poplar *(Populus nigra* L.*)* using reinforcement material. Wood materials were subjected to densification at 140 °C for 10 min. Then, the densified boards were laminated in 3 layers with a reinforcement element (Kevlar®®, fiberglass, and carbon fiber) between the two wooden boards. It was determined that the best result was obtained with the combination of Akfix polyurethane resin type and carbon fiber reinforcement material (8.49 N/mm^2).

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INTRODUCTION

As a traditional material, the tree is obtained from nature in pure form. It is widely used because of its easy processing, strength properties, and cost advantages (Altun *et al.* 2008; Efe and Bal 2016; Görgün *et al.* 2016; Bal and Bektaş 2018; İlkuçar *et al.* 2018). The high demand for wood material has caused the decreased availability of wood having superior properties. For this reason, the need to find new methods to use wood material more effectively has arisen (Percin *et al.* 2009; Kasal *et al.* 2010; Şenol *et al.* 2011; Şenol and Budakçı 2016). Many studies have been conducted to discover new methods that will eliminate or minimize the harmful properties of wood material and improve its properties. The methods that emerged from these studies are generally called "Wood Modification Methods" (Doruk *et al.* 2010; Demirel Köse and Temiz 2015).

Poplar wood material is generally considered too soft and weak for structural applications requiring high strength, stiffness, and durability. The mechanical properties of wood materials are related mainly to their density. Because the densification of wood material increases its mechanical properties and hardness, many attempts have been made to develop a suitable method. The low-density wood material is commercially converted into a high-value product through the densification process. High-density wood material types can also be made more durable by densification (Dizman Tomak and Yıldız 2010;

Doruk *et al.* 2010; Dizman Tomak and Ali 2014; Demirel Köse and Temiz 2015; Hill 2011; Şenol and Budakçı 2016; Sandberg *et al.* 2017; Balfas 2019; Fang *et al.* 2019). The most crucial disadvantage of the densification process is that when the compressed wood material comes into contact with moisture or water, the cell walls tend to expand and regain the attributes of the initial wood. Various studies have been conducted to almost eliminate the tendency to return by chemical modification and thermal hydromechanical treatment (Neyses and Sandberg 2016).

Many studies have been conducted to eliminate the negative properties of wood material. To use wood more efficiently, wood-based composite materials are produced with different techniques (Bal *et al.* 2012). Lamination technology can be briefly expressed as the bonding of many small cross-section boards to each other in layers for a more economical use of the raw material wood, and the improvement of the physical and mechanical properties of building elements (Karayılmazlar *et al.* 2008). In lamination, different wood species, varied number of layers, various sizes, shapes, and layer thicknesses can be applied (Percin *et al.* 2009).

In many academic studies, polyurethane, epoxy, and polyester adhesives have been used in lamination method, densification and bonding strength properties (Percin *et al.* 2009; Rahmani *et al.* 2014; İlhan and Feyzullahoğlu 2019; Karaman *et al.* 2021).

There have been many studies focused on the mechanical properties of densified wood material and laminated wood material. However, there has been a lack of studies on the properties of laminated and reinforced laminated material obtained from densified wood material. For this purpose, the authors aimed to determine the glue type with the highest adhesion between the reinforcement layer and the wood material in reinforced, laminated materials produced from densified wood material. The study produced laminated materials using woven Kevlar®, woven carbon fiber, woven fiberglass as reinforcement, polyurethane, epoxy, and polyester as adhesives.

EXPERIMENTAL

Materials

Black poplar (*Populus nigra* L.) wood was used to prepare the test specimens. Timber was obtained from Safranbolu industrial zone (Karabük, Turkey). During the selection of wood, the following qualities were taken into consideration as per TS EN 384+A2 (2022) and TS ISO 3129 (2021) standards: dry, solid, natural colored, flawless, parallel to each other, no fiber curl, and no damaged by insects and fungi.

Methods

Moisture determination

From the moisture measurements of the wood materials, the average moisture values were determined by the principles specified in TS ISO 13061-1 (2021). The test samples were prepared in the form of a square prism with a length of 30 mm in the fiber direction and a cross-sectional dimension of 20 mm by TS ISO 3129 (2021). The prepared samples were conditioned with an air-conditioning cabinet conditioned to 20 °C and 65% relative moisture content (RH). Weight changes during waiting in the air-conditioning cabinet were regularly monitored. The first 9 measurements were made every 24 h, and the subsequent measurements were made every 72 h. Values were recorded when the samples reached the constant weight (W_S, g). Then, the moisture samples were dehydrated (W_0, g) in a drying cabinet operating at 103 ± 2 °C. Equilibrium moisture content (EMC, g) was calculated using Eq. 1:

$$EMC = (W_s - W_0) / W_0 \quad (1)$$

Impregnation

The plates were prepared in 170 × 300 × 10 mm^3 dimensions and were impregnated with melamine formaldehyde resin for 10 min with 6 bar pressure (Fig. 1). Technical data on melamine formaldehyde resin is given in Table 1.

Table 1. Technical Data of Melamine Formaldehyde Resin

Ph (20 °C)	Viscosity (20 °C)	Solid Content (120 °C 2 h)	Flow Time (FC4, 20 °C, s)
9.5	20 cPs	52%	14

To calculate the retention amount (R%), the plates were weighed before impregnation (M_O) (g) and after the hot-press process (M_1) (g). The amount of solid matter penetrating the leaves was determined using Eq 2:

$$R\% = (M_1 - M_0) / M_o \times 100 \quad (2)$$

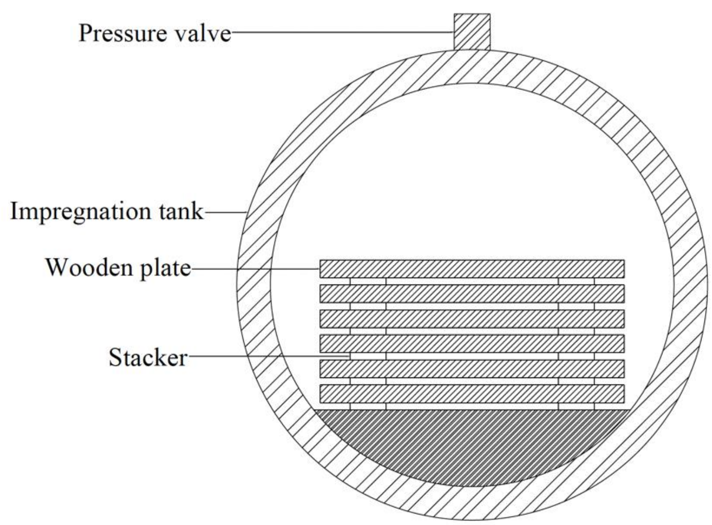

Fig. 1. Impregnation process

Densification Process

The impregnated plates were pressed at 140 °C for 10 min, and their thickness was reduced to 5 mm. A Cemil Usta SSP 180-T 60 × 60 cm^2 laboratory-type press (Cemil Usta Ağaç Makinaları Sanayi Ticaret A.Ş, İstanbul, Turkey) was used for the pressing.

Lamination Process

The densified sheets were laminated in 3 layers with a layer of reinforcement element between every two sheets. Lamination was made at 20±3 °C and 65±5% relative humidity. Woven fiberglass, carbon fiber, and Kevlar® fabrics with a density of 200 g/m^2 were used as reinforcement elements in the study. The study used two different brands of glue from polyester, polyurethane, and epoxy glue types. Technical data on the adhesives used are given in Table 2.

Table 2. Technical Data of Resins and Press Process

	AP	SP	VP	KP	VE	TE
Press Duration (h)	12	12	12	12	12	12
Condition	20±3 °C, 65±5%	20±3 °C, 65±5%	20±3 °C, 65±5%	20±3 °C, 65±5%	20±3 °C, 65±5%	20±3 °C, 65±5%
Pressure (bar)	150	150	150	150	150	150
Glue Amount (g/m^2)	180 to 220	180 to 220	180 to 220	180 to 220	180 to 220	180 to 220
Viscosity (cP) (20 °C)	5.000 to 15.000	6.500 to 8.500	600	700	1000 to 1250	900 to 1000
Amount of Curing Agent	-	-	2%	2%	20%	33%

AP: Akfix polyurethane, SP: Selsil polyurethane, VP: Verpol polyester, KP: Kompozitsan polyester, VE: Verpol epoxy, and TE: Teknomarin epoxy

Set Recovery

To determine the tendency to return, the thickness of the samples in air-dry moisture was measured before the densification process (T_1). Then, their thickness was measured after the densification process (T_2). The samples were kept in water for 4 days, and their thickness at the end of the 4th day was also measured (T_3). Spring back (SB) of densified laminated materials were calculated with Eq. 3:

$$SB\ (\%) = [(T_3 - T_2) / (T_1 - T_2)] \times 100 \tag{3}$$

Bonding Strength

Bonding strength tests of laminated materials obtained from black poplar boards subjected to 140 °C and 50% densification process, obtained with combinations of three reinforcing materials and three types of glue, were completed using the principles of TS EN205 (2017). An example of a bonding strength test sample is given in Fig. 2.

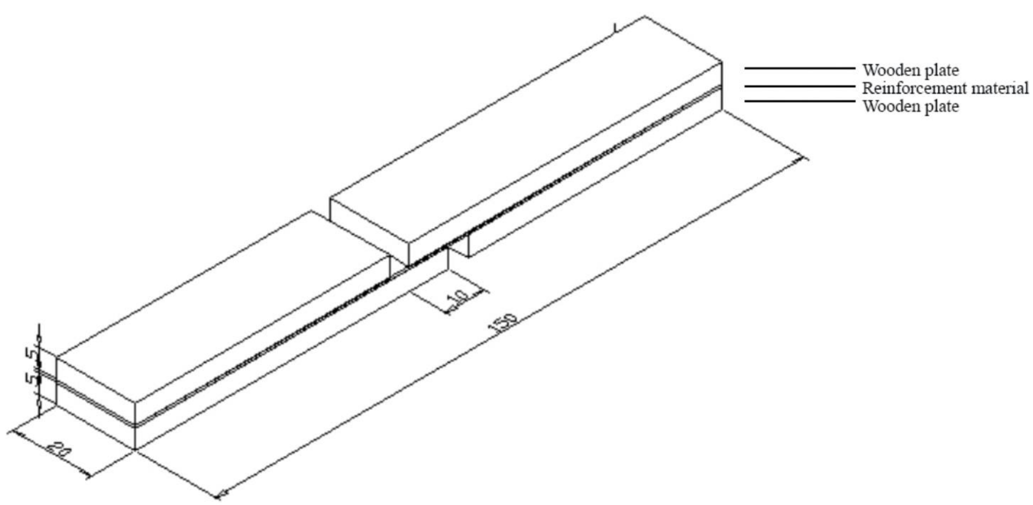

Fig. 2. Bonding strength test sample (Esen and Özcan 2012; CC By 4.0 International License)

The loading speed was kept constant at 1.5 mm/min. The maximum force at break (F_{max}) was determined, and the adhesion strength was calculated with Eq. 4,

$$\sigma_y = F_{max} / A \text{ (N/mm}^2\text{)} \tag{4}$$

where F_{max} is the maximum force at break (N), σ_y is the bonding strength (N/mm²), and A is the (a × b) = adhesion area (xs²).

Statistical Methods

Statistical calculations of the results were performed with SPSS software (IBM Corp., v.21, Armonk, NY, USA). The "Explore" test was applied to determine whether the data showed normal distribution or not. Analysis of variance was performed to determine the effect of glue type on adhesion strength. Duncan's test was applied to determine which homogeneity groups caused the significant differences.

RESULTS AND DISCUSSION

The average densities of black poplar samples before impregnation were calculated as 0.45 g/cm^3 and 1.18 g/cm^3 after the densification process. The average moisture content values of the samples before the densification process were calculated as 10.37%. The average plate thickness before densification was calculated as 9.94 mm, and the average plate thickness after densification was calculated as 4.87 mm. Retention amount (R%) was calculated as 22%.

The samples were kept in water for 4 days to determine the reversion tendency. On the first and fourth days, the samples were measured from three places, and the average values are given in Table 3.

Table 3. Set Recovery Values

Sample Group	Thickness 1st Measurement (mm)		Thickness After 4 Days (mm)		Set Recovery
	Mean	Std. Dev.	Mean	Std. Dev.	
Densified wood	5.01	0.316	5.26	0.267	3%
Control	5.39	0.611	6.94	0.255	23%

The set recovery was calculated using Eq. 5,

$$SR = [(T_3 - T_2) / (T_1 - T_2)] \times 100 \text{ [\%]} \tag{5}$$

where T_1 (mm) is the thickness in dry air moisture before compression (20 ± 2 °C / 65% RH ± 3 RH); T_2 (mm) is the thickness in dry air moisture after compression (20 ± 2 °C / 65% ± 3 RH); and T_3 is the thickness (mm) after soaking.

According to Table 3. It is apparent that the impregnation with melamine formaldehyde resin before densification reduced the set recovery rate from 23% to 3% after densification.

The "Explore" test was applied to determine whether the data showed a normal distribution or not. Test results are given in Table 4.

Table 4. Normal Distribution Test Results

	Statistics	Standard Error
Mean	4.8037	0.27703
Variance	8.442	-
Standard Deviation	2.90553	-
Minimum	0.55	-
Maximum	13.93	-
Skewness	0.907	0.230
Kurtosis	0.167	0.457

According to the results in Table 4, because the Skewness and Kurtosis values were between -1.5 and +1.5, the data showed a normal distribution (Tabachnick and Fidell 2013). The average adhesion strength data of the sheets laminated with three different types of glue using reinforcing material are given in Table 5.

Table 5. Average Adhesion Strength Data of Laminated Boards

Adhesive	Mean	N	Standard Deviation	Minimum	Maximum
SPC	5.1658	12	1.60469	2.39	7.66
SPF	4.8300	11	3.65026	1.29	11.79
SPK	1.9600	9	.96457	1.03	3.71
APC	8.4923	13	2.63191	4.06	13.93
APF	7.8362	13	2.39888	3.51	11.72
APK	2.9780	10	.48905	2.35	3.67
VPK	2.7729	7	1.81516	1.12	6.31
VPF	3.4880	10	1.44996	2.29	7.14
KPC	3.8880	5	0.56539	2.92	4.34
KPF	1.2067	3	1.12873	0.55	2.51
VEC	4.1071	7	1.44616	1.66	5.64
VEF	4.7500	10	2.23729	2.22	8.38
Total	4.8037	110	2.90553	0.55	13.93

SPC: Selfix polyurethane carbon fiber, SPF: Selfix polyurethane fiberglass, SPK: Selifx polyurethane Kevlar®, APC: Akfix polyurethane carbon fiber, APF: Akfix polyurethane fiberglass, APK: Akfix polyurethane Kevlar®, VPK: Verpol polyester carbon, VOF: Verpol polyester fiberglass, KPC: Kompozitsan polyester carbon fiber, KPF: Kompozitsan polyester fiberglass, VEC: Verpol epopxy carbon fiber, VEF: Verpol epoxy fiberglass

According to the data obtained from Table 5, the highest adhesion strength was obtained in the bonding strength samples of carbon fiber reinforced laminated materials (8.49 N/mm^2) made using Akfix polyurethane. Bonding strength tests could not be performed because the desired adhesion could not be achieved in some laminated materials using Kevlar® as a reinforcement material. When the results are compared with the studies, it can be seen that the reinforcement material layer and impregnation process had a negative effect on the bonding strength in the lamination process (Şeker 2011; Keskin *et al.* 2016)

Analysis of variance was performed to determine the effect of glue type on adhesion strength. Analysis of variance results are given in Table 6.

Table 6. Analysis of Variance Results

(I) Adhesive		Mean Difference (I-J)	Standard Error	Sig.
APC	SPC	3.32647*	0.83215	0.000
	SPF	3.66231*	0.85159	0.000
	SPK	6.53231*	0.90139	0.000
	APF	0.65615	0.81533	0.423
	APK	5.51431*	0.87435	0.000
	VPC	5.71945*	0.97451	0.000
	VPF	5.00431*	0.87435	0.000
	KPC	4.60431*	1.09389	0.000
	KPF	7.28564*	1.33144	0.000
	VEC	4.38516*	0.97451	0.000
	VEF	3.74231*	0.87435	0.000

SPC: Selfix polyurethane carbon fiber, SPF: Selfix polyurethane fiberglass, SPK: Selifx polyurethane Kevlar®, APC: Akfix polyurethane carbon fiber, APF: Akfix polyurethane fiberglass, APK: Akfix polyurethane Kevlar®, VPK: Verpol polyester carbon, VOF: Verpol polyester fiberglass, KPC: Kompozitsan polyester carbon fiber, KPF: Kompozitsan polyester fiberglass, VEC: Verpol epoxy carbon fiber, VEF: Verpol epoxy fiberglass

According to Table 6, the bonding strength of glass wool and carbon fiber-reinforced laminated materials prepared using Akfix brand polyurethane adhesive was significantly higher than all other groups. Considering the results, it has been determined that the glue type affects the wood material's bonding strength during lamination (Percin *et al.* 2009; Yörür *et al.* 2010).

Duncan's test was applied to determine which homogeneity groups caused the significant differences. Duncan's test results are given in Table 7.

Table 7. Duncan's Test Results

Adhesive	N	Mean	Homogenous Group
KPF	3	1.2067	D
SPK	9	1.9600	C-D
VPC	7	2.7729	C-D
APK	10	2.9780	C-D
VPF	10	3.4880	C-D
KPC	5	3.8880	B-C
VEC	7	4.1071	B-C
VEF	10	4.7500	B
SPF	11	4.8300	B
SPC	12	5.1658	B
APF	13	7.8362	A
APC	13	8.4923	A

SPC: Selfix polyurethane carbon fiber, SPF: Selfix polyurethane fiberglass, SPK: Selifx polyurethane Kevlar®, APC: Akfix polyurethane carbon fiber, APF: Akfix polyurethane fiberglass, APK: Akfix polyurethane Kevlar®, VPK: Verpol polyester carbon, VOF: Verpol polyester fiberglass, KPC: Kompozitsan polyester carbon fiber, KPF: Kompozitsan polyester fiberglass, FVEC: Verpol epoxy carbon fiber, VEF: Verpol epoxy fiberglass

According to Table 7, the highest adhesion strength was obtained from the samples cut from the laminated sheets produced using Akfix polyurethane adhesive and carbon fiber reinforcement material. The bonding strength of samples obtained from Akfix polyurethane adhesive and glass wool reinforced laminated sheets was also in the same homogeneity group as carbon fiber-reinforced laminated materials using Akfix polyurethane adhesive.

In line with these results, it was concluded that the highest bonding strength was obtained using Akfix polyurethane adhesive. The desired bonding strength results could not be achieved using polyester and epoxy in the lamination processes. When the results found in the literature are compared, it can be seen that the bonding strength results for wood materials laminated with polyurethane and epoxy resins were close to each other.

CONCLUSIONS

It is essential to use low-density wood materials that are low cost and have limited use in the industry through applying the densification process. Considering that all mechanical properties of laminated materials reinforced with densified laminated materials can affect bonding strength,

1. The set recovery value was reduced to 3% by impregnating melamine formaldehyde in the densification process.
2. The highest adhesion strength was obtained from the test samples produced using carbon fiber reinforcement material and polyurethane adhesive. This is because of the strong mechanical bond between the reinforcement element and the wood material.
3. The lowest adhesion strength was obtained in the test samples using polyester adhesive and fiberglass reinforcement material. This is because of the glue penetration and the inability of the glue to establish a mechanical bond between the reinforcement material and the wood material.
4. It is recommended not to use carbon fiber reinforcement elements and polyurethane glue when producing reinforced, laminated materials of low-density wood materials. In applications where adhesion is essential, cheap wood materials with low density can be preferred through increasing their density and strengthening with carbon fiber reinforcement elements.

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