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ORIGINAL SCIENTIFIC PAPERS

REVIEW PAPER / Pregledni rad

Meltem Cengiz Önal[1], Ahmet Gençer[2], Tülin Gürkan Polat[2], Abdullah Sütçü[1]

Improving Surface Quality of Resin Impregnated Paper Used for Laminated Flooring Overlaid

Poboljšanje kvalitete smolom impregniranog papira za pokrivni sloj laminata

ORIGINAL SCIENTIFIC PAPER
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ABSTRACT • *This study aimed to improve the quality properties of impregnated papers used as a coating material in wood-based composites, especially for high-density fiberboard (HDF) floorings, using the Taguchi method and parameter design. Three factors were evaluated in determining the interactions as they are considered the most important in affecting the product quality characteristics such as particle size of Al_2O_3, surface modification agent and melamine raw material. Experiments were conducted in three replications using the L18 ($2^1 \times 3^2$) orthogonal array. Color and gloss measurements were evaluated together with the sensory analysis method, which takes into account the number of the spots in the sample as quality characteristics. The best values obtained as a result of statistical analysis were A_2 for the melamine raw material, 30 to 60 µm for the particle size and N-methyl-3-(trimethoxysilyl) propylamine for the surface modifier agent. Whether the experimental factors have a meaningful effect on the quality characteristics was also tested separately by analysis of variance. Only 58 % of the effect of the evaluated factors on the outcome could be expressed with the linear model (R^2_{adj} = 0.5785).*

KEYWORDS: *melamine-faced laminate; surface modification; experiment design; functional coating*

SAŽETAK • *Cilj ovog istraživanja bio je poboljšati kvalitetu impregniranih papira koji se upotrebljavaju kao pre-krivni materijal za drvne kompozite, posebice za podove od tvrde ploče vlaknatice (HDF) primjenom Taguchijeve metode i projektiranja parametara. Za proučavanje interakcija odabrana su tri čimbenika za koja se smatra da najviše utječu na kvalitetu proizvoda. To su veličina čestica Al_2O_3, sredstvo za modifikaciju površine i melamin. Eksperimenti su provedeni na tri identična uzorka primjenom ortogonalnog niza L18 ($2^1 \times 3^2$). Rezultati boje i sjaja proučavani su zajedno metodom senzorske analize, koja kao odrednicu kvalitete uzima u obzir broj točaka na uzorku. Statistički najbolji rezultati dobiveni su za melamin A2, veličinu čestica 30 do 60 mm i za sredstvo za modifikaciju površine N-metil-3-(trimetoksilil) propilamin. Također, analizom varijance testirana je smislenost učinka eksperimentalnih čimbenika na kvalitetu papira. Samo 58 % evaluiranih čimbenika moglo se izraziti linearnim modelom (R^2_{adj} = 0,5785).*

KLJUČNE RIJEČI: *melaminom obložen laminat; modifikacija površine; projektiranje eksperimenta; funkcionalna prevlaka*

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1 INTRODUCTION
1. UVOD

The wood-based board (WBP) industry has shown a rapid upward trend since the beginning of the century. The industrial WBP production amount was 178 million tons in 2000 on a global scale and reached 387 million tons in 2015 (Morland and Schier, 2020). In China, the biggest industrial producer, plywood production increased from 826,900 m^3 to 171,952,300 m^3, fiberboard production increased from 1,481,000 m^3 to 62,970,000 m^3, and particleboard production increased from 483,100 m^3 to 2,777,700 m^3 in the period from 1988 to 2017 (Ke *et al.,* 2019). Similarly, Türkiye has become an important leader, rising to number one in Europe with 4.7 million m^3 of medium-density fiberboard (MDF) production (compound annual growth rate, CAGR, 12 %), and second with 92 million m^2 of laminate flooring production after Germany, which produced 227 million m^2 (İleri, 2020). In addition, it is also stated that to be consistent with the desired change towards a sustainable bio-economy, the consumption of the round wood should shift from its use as firewood or conventional paper to more efficient productions of wood-based panels or wood cellulose-based materials (Morland and Schier, 2020). Therefore, competition in existing markets is becoming increasingly aggressive as countries with rapid economic growth emerge as new competitors and significantly increase their pressure on the market (Kandelbauer *et al.,* 2010).

Around 70 % of the wood-based panels produced are covered with resin-impregnated paper sheets for decorative and protective purposes. As a result, it is inevitable for manufacturers of impregnated paper for WBP coating to produce innovative and quality products with less cost and higher productivity.

A typical laminate flooring product is composed of a high-density fiberboard (HDF) core, a balancing backing paper bonded underside of the core, a decorative paper that represents the wood grain on the top, and a wear-resistant overlay paper. The HDF core is produced using synthetic binders, mostly urea-formaldehyde, with lignocellulosic fibers. The main purpose of balancing backing paper is to provide strength to the core material and is generally made of low-grade papers. The decorative paper is composed of a thin layer of high-grade cellulose and represents the photographic illustration of the desired wood grain. A clear overlay paper carrying both the melamine resin and aluminum oxide particles (Al_2O_3, corundum) is on the top of the decor paper to provide the protection and stain resistance (Kim and Kim, 2006). Fine grounded particle-shaped aluminum oxide is spread onto the wet overlay paper during impregnation process. The durability of laminate flooring is determined by the AC (abrasion class) level. AC ratings are divided into five levels ranging from AC1 (moderate residential use) to AC6 (heavy commercial use).

In literature, parameters affecting the end product quality of the overlay paper production and melamine impregnation processes, such as resin type (urea-formaldehyde (UF), melamine-formaldehyde (MF), melamine-urea-formaldehyde (MUF), and phenol-formaldehyde (PF)), paper quality, and impregnation steps, have been extensively studied and their physical, chemical, and mechanical processes have been revealed in detail (Roberts and Evans, 2005; Nemli, 2008; Kandelbauer *et al.,* 2010; Thébault *et al.,* 2017). Most recent research has shown that the production of customer-specific products stands out. Researchers are now working for the discovery of innovative products that are more resistant to scratch, dirt, fungi, biological pests, and more considerate of the environment and human health while revealing ways to produce higher quality products at lower costs (Kim and Kim, 2006; Kandelbauer *et al.,* 2010; Badila *et al.,* 2014; Kohlmayr *et al.,* 2014; Nosál and Reinprecht, 2017). Thus, effective analysis processes are needed to produce quality products that can meet user demands, optimize the parameters affecting the process, and provide them on short notice. This study aimed to design the parameters to prevent heterogeneous color changes (blurring on the surface) that are especially visible on the surfaces of dark-colored HDF flooring products and white staining problem on the surface related to the size of the aluminum oxide particles. For this purpose, studies have been conducted to obtain laminate flooring products with improved quality properties by analyzing the effective parameters (melamine powder raw material, Al_2O_3 particle size, type of surface modifier agent) in overlay paper production and melamine resin impregnation with the Taguchi experimental design.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

Raw overlay papers, melamine formaldehyde resins, and aluminum oxides (Al_2O_3) with different particle sizes used in the study were supplied by AGT Wood Industry and Trade Co. (Antalya, Türkiye). The general properties of the overlay paper are given in Table 1. N-methyl-3-(trimethoxy silyl) propylamine (MAMMO), 3-aminopropyl trimethoxysilane (AMMO) and 3-glycidyloxypropyl trimethoxysilane (GLYMO) were obtained from Merck. Hydrochloric acid and ethyl acetoacetate were obtained from Sigma-Aldrich. Melamine types belonging to two different companies (A1 and A2) were used in the MF resin synthesis.

Table 1 Properties of overlay paper
Tablica 1. Svojstva pokrivnog papira

Grammage / *Gramaža*	g/m²	25 ± 2.0
Wet tensile strength *Vlažna vlačna čvrstoća*	N/15 mm	> 5.5
Porosity / *Poroznost*	1/m²s	> 100
Capillary rise *Kapilarna upojnost*	mm/10 min	< 70
pH value / *pH-vrijednost*		6.2 ± 0.5

2.2 Preparation of resin impregnated paper
2.2. Priprema papira impregniranoga smolom

The pre-hydrolysis process, which is the same with N-methyl-3-(trimethoxy silyl) propylamine (MAMMO), is exemplified by the use of 3-aminopropyl trimethoxysilane (AMMO). A total of 20 g (0.1 mol) of AMMO was weighed into a container and to perform the pre-hydrolysis reaction of the ethoxy groups (0.3 mol), sufficient amounts of 2.7 g (0.15 mol) distilled water and 0.5 g 1 M HCl were added. The prepared solution was left to stir at room temperature for 2 h.

In the pre-hydrolysis of the systems prepared by using 3-glycidyloxypropyl trimethoxysilane (GLYMO), aluminum ethyl acetoacetate (Al(1:1) HacacOEt) (%30 by mole) was also used with hydrochloric acid to catalyze and open the epoxy ring. The prepared solution was stirred at room temperature for 4 h.

It is important to note that the alumina particles need to be pre-treated so that they will be more compatible with the MF resin, thus providing higher bonding and strength. In this regard, the above mentioned silane compounds were chosen because of their low cost, availability and ease of use in the hydrolization process.

With the preparation of the modifier solutions, the surface modification phase of the Al_2O_3 particles was conducted. Corundum samples with different particle sizes (30 to 60 µm, 60 to 80 µm, and 80 to 125 µm) were weighed into separate containers as 9 g each and homogeneously mixed with the modifier silane solutions. The modifier ratio was taken as 3 wt% in all systems to observe the effect of silane compounds with different functional groups. Surface-modified corundum systems were pre-dried in an oven at 70 °C for 1 h, adjusted for moisture content and stored until used.

The corundum amount on the surfaces of the AC4 group (≥ 4000 revolutions according to Taber abraser, EN 438-2) laminate flooring products used in the studies should be at least 22 g/m² according to the standard TS EN 13329 A1 (2017). According to this scale, the amount of Al_2O_3 to be used on 40 cm x 40 cm overlay paper was determined as 3.68 g. Selge model decor paper and Timberland model (HUECK, Lüdenscheid, Germany) press sheet were used in all studies. Decor papers are impregnated with 60 g of two different melamine (A_1 and A_2) formaldehyde resins. Al_2O_3 particles were homogeneously dispersed on impregnated overlay paper by using a series of 40 to 63 to 75 µm sieves for 30 to 60 µm-sized particles, 63 to 75 µm sieves for 60 to 80 µm-sized particles, and 75 to 90 to 106 to 125 µm sieves for 80 to 125 µm-sized particles.

Overlay papers carrying the corundum particles were combined with decor papers, placed on HDF panels and pressed for 35 s at 220 N/m² pressure and 185 °C press (ImalPal PL100, Imal Group, Modena, Italy) temperature. A total of 18 × 3 sample panels were prepared in accordance with the experimental design. In the preparation phase of the samples, it was important to focus on randomizing to ensure the homogeneous distribution of the possible effects of the uncontrollable factors across all samples that may affect the test results. The samples coded with random numbers, created in accordance with the uniform distribution of the Excel RAND function (Microsoft Excel, Microsoft Corporation, Version 2013, Redmond, WA, USA), were produced by considering this order.

2.3 Experimental design
2.3. Postavke eksperimenta

Taguchi experimental design is an experimental design method that tries to minimize the uncontrollable factors that create the variability in the product and process by choosing the most appropriate combination of controllable factor levels with performing fewer experiments than the traditional experimental design (Taguchi *et al.,* 2005). It is a direct replacement of traditional One-Factor-At-A-Time or the 'Hit or Miss' approach to experimentation (Antony, 2003).

The goal of the experimental design is to find a link between various variables and an objective characteristic (response). It is critical to identify the proper connection (model) accurately and effectively while designing experiments. These words must be included when there are substantial interactions between variables (causes). The signal to noise ratio (S/N), a measure of functionality, is introduced through parameter design, and orthogonal arrays are used to test the importance of interactions between control elements. If these interactions are substantial, the repeatability of conclusions is disputable. The S/N ratios indicate how a control factor, a signal factor (S), and noise factors (N) interact. The use of the S/N ratio allows for the avoidance of control factor interactions. However, as it is unknown whether or not the interactions between control factors are noticeable simply by adding the S/N ratio, orthogonal arrays are employed to screen for significant interactions (Taguchi *et al.,* 2005).

In the study, the quality performance characteristic is considered as the production of a HDF laminate flooring product that minimizes the formation of white spots on its surfaces. The production methods in the factory were observed to determine the factors and in-

Table 2 Experimental Factors and Levels
Tablica 2. Čimbenici i razine eksperimenta

Factors / *Čimbenici*	**Levels** / *Razine*		
	1	2	3
Melamine powder, raw material / *melaminski prah, sirovina*	A_1	A_2	-
Particle size of Al_2O_3 / *veličina čestica* Al_2O_3	30 to 60 µm	60 to 80 µm	80 to 125 µm
Surface modification agent *sredstvo za modifikaciju površine*	3-aminopropyl trimethoxysilane	3-glycidyloxy-propyl trimethox-ysilane	N-methyl-3-(trimethoxysilyl) propylamine

teractions and three factors affecting the quality characteristics that were selected within the study framework (Table 2).

The L18 ($2^1 \times 3^2$) orthogonal index for factors and levels was selected and necessary analyses were performed using Minitab software (Version 16, Minitab Inc., State College, PA, USA). The selected array indicated that 18 experiments should be performed. Each experiment was repeated three times to reduce variation. Thus, measurements were made on 18 × 3 = 54 samples. L18 arrays are highly recommended because the interactions are distributed uniformly to all columns (Taguchi *et al.,* 2005; Nas and Altan Ozbek, 2019).

2.4 Color and gloss measurements
2.4. Mjerenje boje i sjaja

The purpose of the color measurements is not to make precise color definitions on laminate flooring surfaces, but to calculate the total color difference (metric) (ΔE^*) occurring in the samples compared to the control group using the CIE Lab 1976 (Commission Internationale d'Eclairage) standard (Sahin and Mantanis, 2011). ΔE is defined as the difference between two colors in an $L^*a^*b^*$ color space. ΔE^* is total color difference and is calculated based on delta L^*, a^*, b^* color differences (Eq.1):

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \qquad (1)$$

Ten measurements were made on each specimen and the average of the color values were obtained. All color measurements were made with a portable X-Rite SP 968 spectrophotometer (X-Rite Inc., Grandville, MI, USA), which automatically calculated. A D65 illuminant and a 10-degree standard observer were used to make the measurements.

In the study, surface gloss measurements were made with a Pacific Scientific Glossgard II 60° gloss meter according to ASTM D523-14 (2018) standard. Measurements were repeated ten times and the average values were calculated to evaluate the color and gloss of each sample.

2.5 Sensory analysis method
2.5. Metoda senzorske analize

Visual control was carried out by the sensory analysis method to determine the intensity of white staining on laminate flooring surfaces. A template with a 10 cm × 30 cm size was prepared for this purpose. Then, 30 cells of 4 cm^2 area were created in the template and then 10 of them were randomly selected (Figure 1). The number of cells with the staining on this template was noted and evaluated on a scale of 1 to 10.

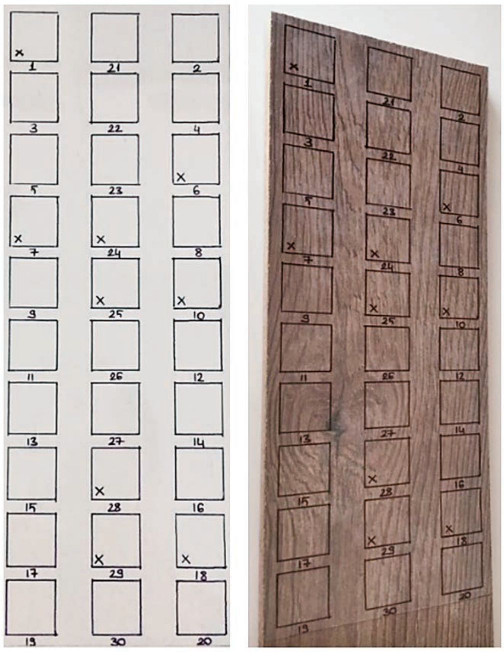

Figure 1 Template used to determine stain density
Slika 1. Predložak za određivanje intenziteta obojenja

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Experimental results
3.1. Eksperimentalni rezultati

The effect on the three quality characteristics (stain, color, and gloss) was evaluated separately and tested by analysis of variance (ANOVA) to determine whether the experimental factors had a significant effect on the stated quality characteristics.

It was observed in analyses made for the stain quality characteristics that all factors and interactions had a significant effect on the results at the 95 % confidence level, except for the Factor_1*Factor_3 (M*A) interaction ($P < 0.05$) (Table 3).

Table 3 ANOVA results for color, gloss, and stain count
Tablica 3. ANNOVA rezultati za boju, sjaj i intenzitet obojenja

Variance source *Izvor varijance*	**Degree of freedom (*DoF*)** *Stupanj slobode (DoF)*	**Sum of squares (*SS*)** *Zbroj kvadrata (SS)*	**Mean squares (*MS*)** *Srednji kvadrati (MS)*	***F* value** *F-vrijednost*	***P* value** *P-vrijednost*	**Contribution rate** *Doprinos*
Color (ΔE)						
M	1	0.0413	0.04133	0.05	0.825	11.14 %
P	2	4.7107	2.35533	2.82	0.073	2.72 %
A	2	3.3318	1.66590	2.00	0.150	5.94 %
M*P	2	5.4046	2.70231	3.24	0.051	35.44 %
M*A	2	0.1846	0.09230	0.11	0.896	0.31 %
P*A	4	3.7055	0.92639	1.11	0.366	6.21 %
M*P*A	4	1.2694	0.31735	0.38	0.821	1.55 %
Error	36	30.0172	0.83381			36.69 %
Total	53	81.8075				100 %
	R^2	R^2_{adj}				
	63.31%	45.98%				
Gloss Unit (*GU*)						
M	1	9.728	9.7283	17.37	0.000	17.50 %
P	2	9.935	4.9674	8.87	0.001	3.91 %
A	2	12.369	6.1844	11.04	0.000	16.29 %
M*P	2	8.372	4.1858	7.47	0.002	0.71 %
M*A	2	7.737	3.8686	6.91	0.003	3.46 %
P*A	4	17.845	4.4613	7.96	0.000	7.68 %
M*P*A	4	12.789	3.1972	5.71	0.001	15.69 %
Error	36	20.168	0.5602			24.75 %
Total	53	81.492				100 %
	R^2	R^2_{adj}				
	75.25%	63.57%				
Stain Count (*SC*)						
M	1	10.667	10.667	5.14	0.029	16.36 %
P	2	22.889	11.444	5.52	0.008	14.58 %
A	2	13.556	6.778	3.27	0.050	13.65 %
M*P	2	18.778	9.389	4.53	0.018	0.55 %
M*A	2	4.333	2.167	1.04	0.362	3.88 %
P*A	4	57.259	14.315	6.90	0.000	11.45 %
M*P*A	4	28.444	7.111	3.43	0.018	10.91 %
Error	36	74.667	2.074			28.63 %
Total	53	260.815				100 %
	R^2	R^2_{adj}				
	71.37%	57.85%				

M – Melamine powder raw material, P – Al_2O_3 particle size, A – Surface modification agent / *M – melaminski prah, sirovina, P – veličina čestica Al_2O_3, A – sredstvo za modifikaciju površine*

The detailed analysis conducted for the Stain Count quality characteristic was also performed for the color values, but it was observed that the factors and their interactions did not have a significant effect ($P > 0.05$) (Table 3).

In the analysis made for the Gloss quality characteristic, it was determined that the factors and their interactions had a significant effect at the 95 % confidence level (Table 3). However, this high interaction occurs naturally due to low gloss (high opacity) in stained areas and high gloss in unstained areas. This situation, which cannot be avoided during random measurements, makes it impossible to monitor the effect of the test condition. For this reason, although all response values were included in the Taguchi analysis, in the calculation of *S*/*N* ratios and in drawing the graphs, detailed analyses were made only for the "Stain Count" quality characteristic considering that the significant relationship may be misleading.

The *S*/*N* ratio of the Taguchi method has three quality characteristics as nominal-the-best, smaller-the-better, and larger-the-better (Taguchi *et al.,* 2005). In this study, the characteristics defined were smaller-the-better for the ΔE and the Stain Count and larger-the-better for the Gloss. Table 4 shows the *S*/*N* ratios and the experimental Color (ΔE^*), the Gloss Unit (*GU*), and the Stain Count (*SC*) results obtained *via* the Taguchi L18 experimental design.

Table 4 Experimental results and *S*/*N* ratios
Tablica 4. Eksperimentalni rezultati i *S*/*N* omjeri

	Control factors *Kontrolni čimbenici*								
Exp. No	M	P	A	Color (ΔE)	Gloss (*GU*)	Stain (*SC*)	*S*/*N* for Color	*S*/*N* for Gloss	*S*/*N* for Stain
1	A_1	30 to 60 µm	AMMO	1.876	13.25	5	-6.2905	23.4577	-12.9373
2	A_1	30 to 60µm	GYLMO	3.547	16.81	3	-9.3477	24.7145	-8.6530
3	A_1	30 to 60µm	MAMMO	3.348	18.00	2	-11.0398	25.0109	-3.0103
4	A_1	60 to 80µm	AMMO	3.894	11.45	10	-11.8801	22.5667	-17.6343
5	A_1	60 to 80 µm	GYLMO	3.802	16.98	3	-14.0874	24.4287	-10.5436
6	A_1	60 to 80 µm	MAMMO	6.197	17.45	2	-14.2477	24.8838	-6.0206
7	A_1	80 to 125 µm	AMMO	2.743	16.04	3	-9.3087	24.2591	-12.1307
8	A_1	80 to 125 µm	GYLMO	2.100	13.56	10	-8.0339	23.3820	-18.5733
9	A_1	80 to 125 µm	MAMMO	2.608	16.12	3	-7.6843	24.4757	-11.5635
10	A_2	30 to 60 µm	AMMO	1.611	17.88	2	-5.5969	24.9217	-4.7712
11	A_2	30 to 60 µm	GYLMO	2.186	17.34	1	-7.3454	24.8520	-4.7712
12	A_2	30 to 60 µm	MAMMO	3.160	17.35	0	-11.8520	24.7366	-4.7712
13	A_2	60 to 80 µm	AMMO	1.825	17.28	2	-4.5161	24.8022	-6.0206
14	A_2	60 to 80 µm	GYLMO	1.477	16.92	3	-5.7872	24.5930	-8.6530
15	A_2	60 to 80 µm	MAMMO	1.733	17.52	0	-5.2552	24.8006	-6.3682
16	A_2	80 to 125 µm	AMMO	2.368	17.11	4	-10.3494	24.4199	-10.7918
17	A_2	80 to 125 µm	GYLMO	2.401	17.04	6	-9.9562	24.6681	-12.5527
18	A_2	80 to 125 µm	MAMMO	3.561	17.13	2	-10.8483	24.8045	-7.5333

M – Melamine powder raw material, P – Al_2O_3 particle size, A – Surface modification agent (AMMO: 3-aminopropyl trimethoxysilane, MAMMO: N-methyl-3-(trimethoxylyl) propylamine and GLYMO: 3-glycidyloxypropyl trimethoxysilane) / *M – melaminski prah, sirovina, P – veličina čestica* Al_2O_3*, A – sredstvo za modifikaciju površine (AMMO: 3-aminopropil trimetoksilan, MAMMO: N-metil-3-(trimetoksilil) propilamin i GLYMO: 3-glicidiloksipropil trimetoksilan)*

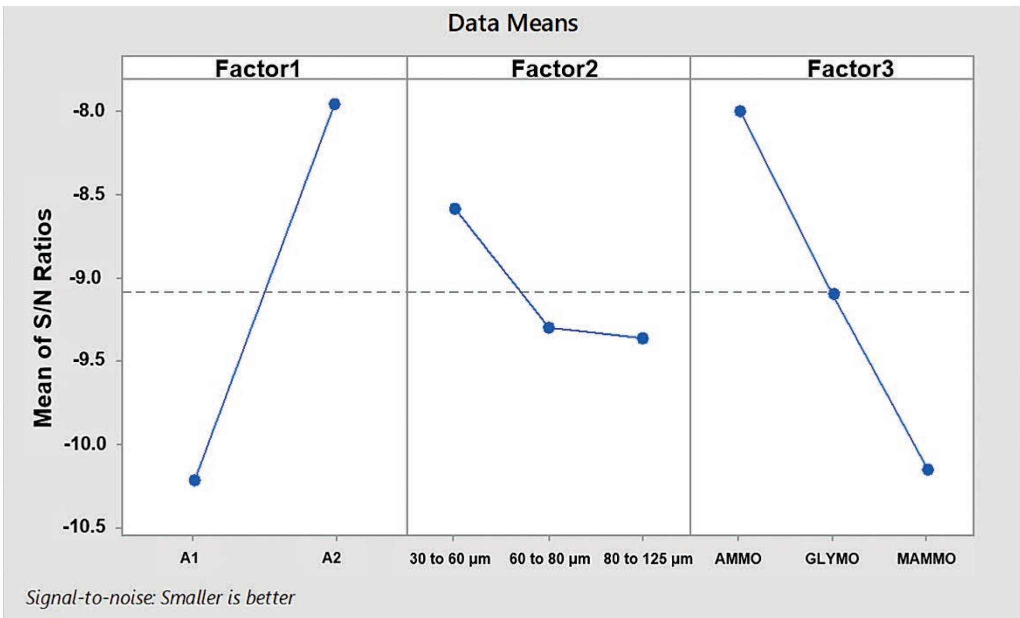

Figure 2 Process parameter effects on *S*/*N* ratio for color
Slika 2. Utjecaji procesnih parametara na *S*/*N* omjer za boju

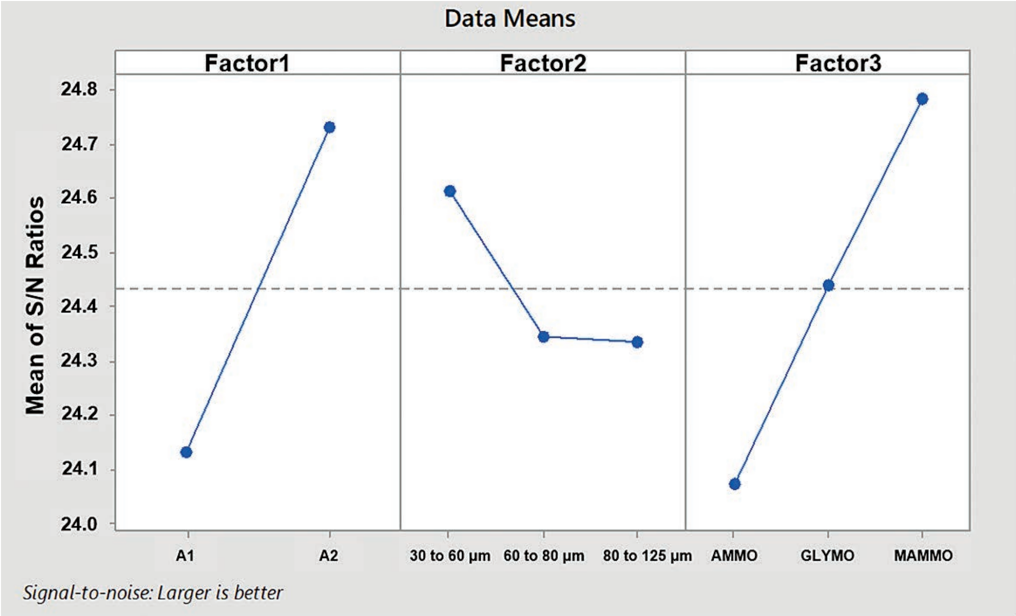

Figure 3 Process parameter effects on *S/N* ratio for stain
Slika 3. Utjecaji procesnih parametara na *S/N* omjer za obojenje

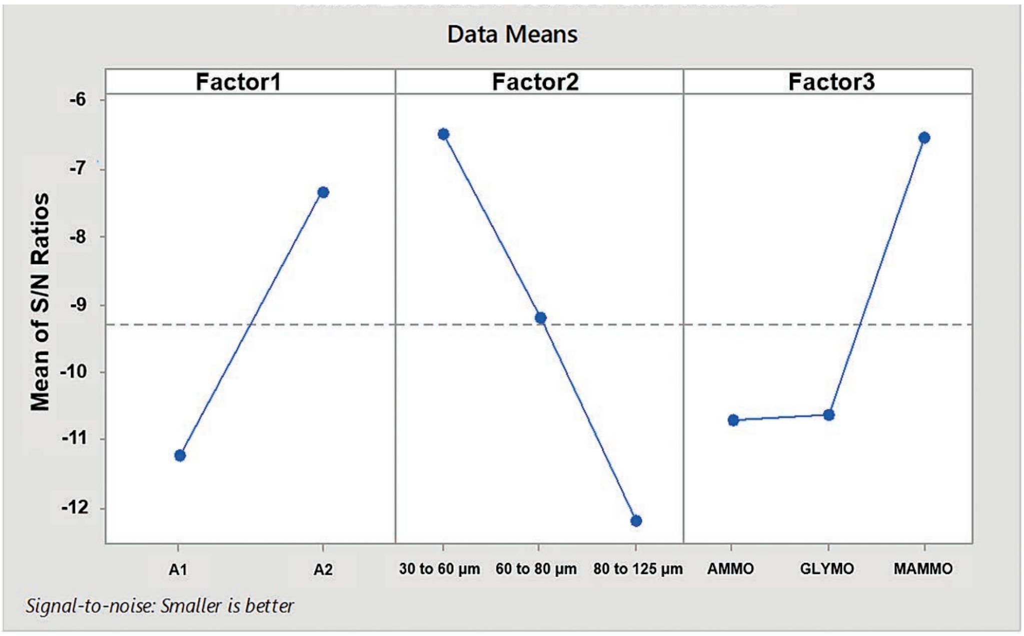

Figure 4 Process parameter effects on *S/N* ratio for stain
Slika 4. Utjecaji procesnih parametara na *S/N* omjer za obojenje

As a result of the analyses and the evaluation of the graphics, it was observed that the most suitable combination was obtained by using the A_2 melamine powder raw material, the Al_2O_3 with a particle size of 30 to 60 µm and the surface modification agent of N-methyl-3-(trimethoxy silyl) propyl amine (Figure 4).

Confirmation tests were performed for the determined combination. The mean values of the analyses (0.156) and the confirmation tests (0.666) showed the compatibility of each other.

3.2 SEM Results
3.2. Rezultati SEM-a

Laminate flooring samples produced in accordance with the result of the most suitable combination were cut in 2 cm × 2 cm dimensions, analyzed with a scanning electron microscope (SEM-FEI Quanta FEG 250) and SEM images were taken.

Taguchi analysis revealed that the best combination was A_2 / 30 to 60 µm / N-methyl-3-(trimethoxy silyl) propyl amine, while the worst combination was

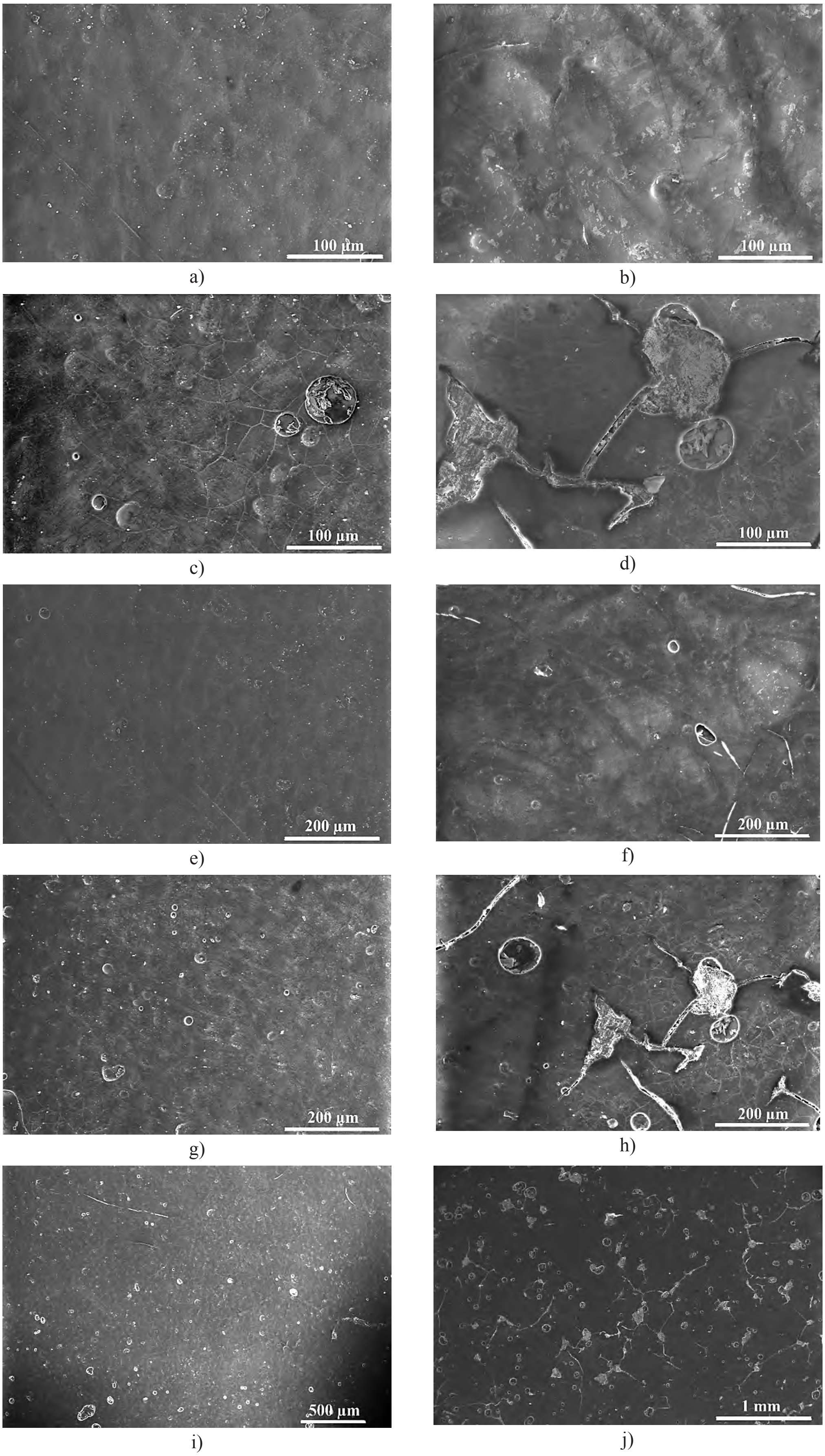

Figure 5 SEM images of: 1- best combination: a: 100 µm, c: 100 µm, e: 200 µm, g: 200 µm, i: 500 µm, and 2- worst combination: b: 100 µm, d: 100 µm, f: 200 µm, h: 200 µm, j: 1 mm)
Slika 5. SEM fotografije: 1 – najbolja kombinacija (a: 100 mm, c: 100 mm, e: 200 mm, g: 200 mm, i: 55 mm), 2 – najlošija kombinacija (b: 100 mm, d: 100 mm, f: 200 mm, h: 200 mm, j: 1 mm)

A_1 / 80 to 125 μm / 3-aminopropyl trimethoxysilane. In Figure 5, the images of the same magnification were compared according to the combinations. The combination of A_2 / 30 to 60 μm / N-methyl-3-(trimethoxysilyl) propyl amine, which had almost no white spots by sensory analysis, appeared to be homogeneously distributed when microscopic images were examined. In the microscopic images of the combination of A_1 / 80 to 125 μm / 3 to aminopropyl trimethoxysilane, which had intense white staining on the surface, there was no homogeneity, and cluster formations were observed. It was found that these clusters in the SEM images and the white staining that occur on the laminate surfaces overlap with each other (Figure 5).

4 CONCLUSIONS
4. ZAKLJUČAK

While no significant relationship was found for the color quality characteristic, the gloss characteristic provided a meaningful result. It was determined that the gloss was low in the areas with staining on the samples, while it was high in the areas where there were no spots.

In this study, the data belonging to the stain variable are qualitative data determined by sensory analysis. Qualitative data can also be obtained by using image processing techniques in future studies.

Only 57 % of the effects of the evaluated factors on the result (staining) could be expressed in the linear model (R^2_{adj} = 0.5785). The effect of other factors that were not evaluated by the model was also too high to be ignored. However, due to the working conditions and limitations, the study was completed with the existing evaluation factors.

The best values obtained by statistical analysis were estimated to be A_2 for the melamine raw material, 30 to 60 μm for the particle size, and N-methyl-3-(trimethoxysilyl) propyl amine for the surface modifier agent. The validation test result showed that the analysis result was consistent with the average of 0.156.

In addition, SEM images of the samples with different combinations predicted as the best and the worst by Taguchi analysis were compared. It was found that these clusters in the SEM images and the white staining that occur on the laminate surfaces overlap with each other.

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Production of Lightweight Three-Layered Particleboards Using Waste Tire Rubbers

Proizvodnja laganih troslojnih iverica iskorištenjem otpadnih autoguma

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ABSTRACT • *The aim of the study is to investigate the production possibilities of particleboard by using waste tire rubbers with different properties as fillers and find a solution to a serious environmental pollution problem caused by waste tires. For this purpose, waste summer and winter tires were ground, rubber powders were obtained by separating rubber materials from other materials, and particleboards with different properties were produced using this sawdust in different percentages. The oven-dry density, air-dry density, and equilibrium moisture content at (65±5) % RH and (20±2) °C, thickness swelling (TS, 2 hours and 24 hours), water absorption (WA, 2 hours and 24 hours) characteristics, bending strength, modulus of elasticity, and internal bond strength were determined according to the applicable European standards. The hydrophobic nature of the tire rubber enhanced the water absorption ratios of particleboards. In addition, mechanical performance of groups was affected by tire content, and it was mainly determined that the values decreased dramatically with increasing tire content. It was especially observed that this decrease was more pronounced when subtracting 30 % to 20 % waste tire content. The groups including winter waste tire rubber achieved better performance than summer waste tire groups. The use of waste rubber in boards resulted in a decrease in the equilibrium moisture content and mechanical strength of the samples.*

KEYWORDS: *mechanical properties; particleboard; physical properties; waste summer tire; waste winter tire*

SAŽETAK • *Cilj ovog rada bio je istražiti mogućnosti proizvodnje ploča iverica iskorištenjem otpadnih autoguma različitih svojstava kao punila te pronalaženje rješenja za ozbiljno onečišćenje okoliša otpadnim autogumama. Za potrebe eksperimenta samljevene su otpadne ljetne i zimske autogume, a odvajanjem gumenih od negumenih dijelova dobiven je prah gume, koji se u različitim omjerima upotrebljavao za proizvodnju ploča iverica različitih svojstava. Prema europskim standardima određena su ova svojstva ploča iverica: gustoća u apsolutno suhom i prosušenom stanju, relativni sadržaj vode pri (65±5) RH i (20±2) °C, debljinsko bubrenje (TS, nakon 2 sata i 24 sata) i upijanje vode (WA, nakon 2 sata i 24 sata). Zbog hidrofobne prirode praha autoguma povećani su omjeri upijanja vode ploča iverica. Osim toga, udjel dodane količine praha autoguma utjecao je na mehanička svojstva ploča iverica te je utvrđeno da se te vrijednosti drastično smanjuju s povećanjem količine praha autoguma u pločama. Posebno je naglašeno smanjenje mehaničkih svojstava ploča iverica kada udio praha autoguma prijeđe 20 %. Uzorci ploča iverica s prahom od zimskih guma imali su bolja svojstva od uzoraka s prahom od ljetnih guma. Uporaba otpadnih autoguma u proizvedenim ivericama rezultirala je smanjenjem ravnotežnog sadržaja vode i mehaničke čvrstoće uzoraka.*

KLJUČNE RIJEČI: *mehanička svojstva; ploča iverica, fizička svojstva; otpadna ljetna guma; otpadna zimska guma*

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1 INTRODUCTION
1. UVOD

Wood is one of the most commonly used natural raw materials for various applications ranging from power generation to construction panels. This variety in the usage of wood materials increases the need for raw materials. As a result, problems related to intensive natural resources and environmental issues increase gradually (Couto *et al.,* 2013).

Waste tires are generally burned or left uncontrolled at landfills, but nature is not coping with the consequences of its devastation (Svoboda *et al.,* 2018; Dwivedi *et al.,* 2020). The primary reason of this problem is a change in the physical and chemical structure of tires resulting from production goals such as extended service life, safety, noise reduction, and unforeseen changes due to unknown alterations performed by manufacturers in production recipes. Furthermore, the three-dimensional cross-linked structure generated by the irreversible vulcanization process makes scrap tires a durable and non-biodegradable material in the face of various environmental influences. As a result, the issue of the inability to manage waste tires has long been a source of concern for scientists, industry representatives, and governments (Ayrilmis *et al.,* 2009a; Hejna *et al.,* 2020). In order to solve this problem, different recycling methods and usage areas for waste tires should be developed (Saputra *et al.,* 2020; Formela, 2021).

Recycled rubber materials are already utilized in the rubber manufacturing industry following appropriate cleaning, repair, or refining operations (Xu *et al.,* 2020). In addition, waste tires may also be utilized in various sectors, including matting, waterproofing systems, membrane linings, artificial reefs, sports fields, and erosion control (Ramarad *et al.,* 2015; Svoboda *et al.,* 2018; Xu *et al.,* 2020). However, these utilization sectors were judged insufficient to limit the number of tires found in landfills and thrown on fields at random (Martínez-Barrera *et al.,* 2020).

In 2015, the Turkish Ministry of Environment and Urbanization reported that 315.000 metric-ton scrap tires were sold to the recycling sector. Furthermore, it has been reported that around 300,000 tons of trash tires are generated in Türkiye each year (Başboğa *et al.,* 2020). For this reason, it is urgently necessary to develop different applications for the evaluation of waste tires (Ayrilmis *et al.,* 2009a).

In addition to conventional recycling processes, one of the innovative approaches to solving this problem is to create new composites using various polymers or polymer combinations from waste tires as filling/reinforcing components (Dwivedi *et al.,* 2020; Hejna *et al.,* 2020).

In contrast to isotropic materials such as concrete and steel, wood materials have greater mechanical behavioral complexity because they are an anisotropic material (Pereira *et al.,* 2019). Wood-based boards are more isotropic composite materials than solid wood, produced by bonding different shaped pieces of wood under pressure and at high temperatures (Mesquita *et al.,* 2019). These high temperatures used in the production of wood-based composite boards cause a decrease in the hygroscopicity of the materials (López *et al.,* 2018). Improving the dimensional stability of wood-based panels such as particleboard and the development of water-resistant wood composites have long been the target of wood technology experts and the industry (Mesquita *et al.,* 2019).

In this regard, waste tire rubbers are a promising alternative raw material in terms of usability in wood-based composites with many advantages such as high durability against chemicals and fungi, high energy absorption, high elasticity, etc. (Ayrilmis *et al.,* 2009a; Ayrilmis *et al.,* 2009b; Terzi *et al.,* 2009; Ashori *et al.,* 2015; Ghofrani *et al.,* 2016). It was reported that the addition of rubber to wood composites positively affected shock absorbing, energy absorbing, water repellent, anti-rot, and electrical insulation properties (Vilela *et al.,* 2017; Xu *et al.,* 2020) and negatively affected mechanical properties (Hejna *et al.,* 2020).

The aim of the study is to investigate the production possibilities of particleboard by using waste tire rubbers with different properties as fillers and find a solution to a serious environmental pollution problem caused by waste tires. In addition, it is aimed to reduce the need for wood raw material in the particleboard industry. For this purpose, waste summer and winter tires were ground, rubber powders were obtained by separating rubber materials from other materials, and particleboards with different properties were produced using this sawdust in different percentages. Experimental samples were cut from the produced particleboards and related experiments were carried out.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

In the study, continental™ brand summer and winter tires, which have completed their useful life, were used. Used tires were removed from steel wires and ground into a fine powder (size of powders were from 0.11 mm to 2 mm as shown in Table 1) at the recycling factory. This powdered material was then mixed with wood chips and used to make particleboard. The core and surface layer chips (size distributions shown in Table 1) used in production, urea-formaldehyde glue with

Table 1 Size distribution of wood chips and waste tire powders
Tablica 1. Distribucija veličine drvnog iverja i praha od otpadnih autoguma

Sieve size *Veličina sita*	**Surface layer chips, %** *Iverje površinskog sloja, %*	**Sieve size** *Veličina sita*	**Core layer chips, %** *Iverje središnjeg sloja, %*	**Sieve size** *Veličina sita*	**Summer tire powders, %** *Prah od ljetnih guma, %*	**Winter tire powders, %** *Prah od zimskih guma, %*
4 mm	0.00	6.3 mm	0.00	2 mm	0.00	0.00
2 mm	1.00	4 mm	5.00	0.84 mm	5.10	4.80
1 mm	5.00	2 mm	40.00	0.59 mm	8.80	9.80
0.8 mm	7.00	1 mm	35.00	0.42 mm	23.70	25.20
0.6 mm	10.00	0.8 mm	7.00	0.25 mm	28.10	27.60
0.4 mm	30.00	0.5 mm	5.00	0.18 mm	13.50	11.40
0.315 mm	20.00	0.315 mm	3.00	0.15 mm	9.60	13.50
0.2 mm	15.00	0.2 mm	2.00	0.13 mm	6.80	4.50
0.1mm	10.00	0.1mm	2.00	0.11 mm	3.80	2.75
< 0.1 mm	2.00	< 0.1 mm	1.00	< 0.11mm	0.60	0.45

65 % solids content, and ammonium chloride (NH_4Cl) hardeners prepared with water as a 20 % solution, were all supplied by the Kastamonu Entegre™ company. After the chips were taken from the dry chip silo with a humidity of 3-5 %, they were placed in nylon bags to prevent moisture absorption.

2.2.1 Production of test samples
2.2.1. Proizvodnja ispitnih uzoraka

The average density of the particleboards was targeted at 500 kg/m^3, and in line with this target, the amounts and ratios of chip-waste rubber-resin-hardener were calculated from the Equation 1 to 3 according to the volume of the press mold measuring 500 mm × 500 mm × 18 mm, and the production was carried out on these calculated values.

d target × *V mold* = *m oven dry chips* + *m solid content adhesive* + *m solid content hardener* (1)

m solid content adhesive = 0.10 *m oven dry chips* x *solid content of adhesive* (2)

m solid content hardener = 0.01 *m oven dry chips* x *solid content of solution* (3)

Surface and core layer chips or chips- waste tire mixtures were added to a drum gluing mixer separately. The urea-formaldehyde resin was applied in 10 %, and the ammonium chloride solution was applied in 1% based on the weight of oven-dried chips. The mixture (resin and hardener) was applied separately to the surface layers and core layer of the chip-waste tire mixture by spraying with the help of compressed air in a 100 lt sample volume drum gluing mixer (no brand, Kastamonu Türkiye) with two points injection, and 5 rpm rotating speed. Gluing machine and chips blend are given in Figure 1. In addition, the use of chip-waste rubber in test board production also varies, and the chip-waste tire mixture ratios used in test board production are given in Table 2.

The glued chips were laid out by hand as surface layer + core layer + surface layer, paying attention to the equal distribution in the press mold. Next, the pre-cold pressing process was applied to the prepared board draft by applying a pressure of around 1 kN. Then, the prepared board draft was applied to hot pressing for 10 minutes, including the heating time of the loading press platens, at a temperature of 190 ºC

Table 2 Tire and wood mixture ratios in board production
Tablica 2. Omjeri praha guma i drvnog iverja u proizvodnji ploča

Groups *Grupe*	**Surface/core/surface layer ratio, %** *Površinski sloj /središnji sloj / površinski sloj, %*	**Wood chip ratio, %** *Udio drvnog iverja, %*	**Summer tire (*S*) waste ratio, %** *Udio praha otpadnih ljetnih autoguma (S), %*	**Winter tire (*W*) waste ratio, %** *Udio praha otpadnih zimskih autoguma (W), %*
Control *Kontrolni uzorak*	20-60-20	100	0	0
S 10		90	10	0
S 20		80	20	0
S 30		70	30	0
S 40		60	40	0
W 10		90	0	10
W 20		80	0	20
W 30		70	0	30
W 40		60	0	40

Figure 1 A) Gluing machine, B) Surface layer blend, C) Core layer blend
Slika 1. A) Oprema za obljepljivanje iverja, B) smjesa za površinski sloj, C) smjesa za središnji sloj

Figure 2 A) Hot press and mold, B) W 10 group samples, C) S 40 group samples
Slika 2. A) Vruća preša i kalup, B) skupina uzoraka W 10, C) skupina uzoraka S 40

and a pressure of 20 kg/cm^2. Cemilusta brand SSP 125 model (İstanbul, Türkiye) hot press was used in the production. Three boards were produced for each group. Hot press and test samples are given in Figure 2.

After pressing, the particleboards were kept in a Protech brand climate chamber (Antalya, Türkiye) adjusted to (65±5) % humidity and (20±2) ºC until they reached a constant weight and reached air-dry humidity after cooling.

2.2 Methods
2.2. Metode

According to EN 322 (1999) and EN 323 (1999), the oven-dry density, air-dry density, and equilibrium moisture content at (65±5) % RH and (20±2) ºC were determined. The EN 317 (1999) standard was used to measure the thickness swelling (TS, 2 hours and 24 hours) and water absorption (*WA*, 2 hours and 24 hours) characteristics. Bending strength and modulus of elasticity properties were determined by using Shimadzu (Made in Japan) brand AG/IC 50KN model universal test machine according to EN 310 (1999), and the EN 319 (1999) standard was used for determining internal bond strength. Mitutoyo brand digital micrometers with 0.01 mm graduations were used to measure the thickness and length of all test samples. Nucleon brand NKD-400 model oven (Ankara, Türkiye) was used to dry the samples. Mass of specimens was measured by using Radwag brand WTB 200 model laboratory scales. All experiments were performed in the Wood Mechanics and Technology Laboratories, Forest Faculty of Kastamonu University. To perform descriptive statistics, SPSS Version 22.0 software was used.

A stereo microscope and scanning electron microscope (SEM) were used to examine the structure of the panels. The stereo microscope was an Olympus SZ61 (Tokyo, JAPAN), equipped with a digital microscope camera and a magnification range from 6.7x to 45x. A Quanta FEG 250 (FEI, USA) was used for SEM analysis, and after that the particleboards were coated with a thin layer of gold to improve surface resolution of the boards using a Cressington Sputter Coater 108 Auto Au-Pd Coating Machine, at Kastamonu University Central Research Laboratory, under 40 mA for 30 seconds.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Physical properties
3.1. Fizička svojstva

The determined physical properties and standard deviations of the produced boards are shown in Table 3. As a result of the experiments, the average dry density (d_{Dry}) of the produced particleboards was 520 kg/m^3. The average air-dry density ($d_{Air\ Dry}$) was 540 kg/m^3. It was concluded that the targeted density objective was mostly met. When the data was examined, it was observed that the highest equilibrium moisture was 9.1 % in the control samples. Depending on the amount of waste tire rubber contained in the boards, there was a decrease in the air-dry equilibrium moisture of the samples. The

Table 3 Physical properties of boards with a standard deviation
Tablica 3. Fizička svojstva ploča (u zagradi su standardne devijacije)

Groups *Skupine*	d_{Dry} **kg/m³**	$d_{Air\ Dry}$ **kg/m³**	**Equilibrium moisture, %** *Ravnotežni sadržaj vode, %*	***TS* 2 hours, %** *TS nakon 2 sata %*	***TS* 24 hours, %** *TS nakon 24 sata %*	***WA* 2 hours, %** *WA nakon 2 sata %*	***WA* 24 hours, %** *WA nakon 24 sata %*
Control	528.32 (20.07)	554.13 (20.81)	9.09 (0.21)	17.23 (0.99)	18.46 (1.33)	107.42 (5.69)	110.66 (6.07)
S 10	522.75 (12.44)	543.86 (9.98)	8.12 (1.29)	15.48 (1.61)	16.84 (1.66)	101.80 (4.98)	108.18 (5.79)
S 20	514.24 (38.03)	536.17 (40.10)	8.29 (0.28)	13.09 (1.16)	14.16 (1.34)	90.62 (5.43)	95.44 (5.42)
S 30	506.05 (24.58)	521.90 (24.41)	6.74 (0.08)	9.02 (0.65)	9.48 (0.53)	88.96 (4.75)	93.50 (4.09)
S 40	542.19 (13.21)	574.12 (23.88)	6.12 (0.13)	7.68 (1.09)	8.84 (0.82)	72.26 (8.27)	83.48 (6.71)
W 10	503.39 (6.93)	528.17 (9.38)	8.89 (0.41)	13.71(1.44)	15.37 (1.30)	97.57 (4.40)	101.05 (3.88)
W 20	504.02 (15.91)	522.94 (15.79)	7.46 (0.10)	10.61 (1.19)	12.09 (1.32)	83.80 (5.36)	92.06 (3.52)
W 30	529.55 (17.54)	544.94 (19.36)	6.88 (0.52)	10.15 (1.29)	11.59 (1.59)	74.42 (4.33)	84.21 (3.74)
W 40	525.38 (15.76)	539.25 (15.83)	5.99 (0.23)	6.88 (0.73)	7.45 (1.02)	89.94 (7.37)	94.29 (6.31)

Table 4 Results of variance analysis of thickness swelling
Tablica 4. Analiza varijance rezultata debljinskog bubrenja

Experiment *Eksperiment*	**Source** / *Izvor*	**Type III Sum of squares** *Zbroj kvadrata, tip III*	***df***	**Mean square** *Srednji kvadrat*	***F***	***Sig.***
2 hours *TS*, % *nakon 2 sata TS, %*	Waste type (A) /*vrsta otpada (A)*	22.795	1	22.795	15.390	.000
	Ratios (B) /*udjeli (B)*	704.459	3	234.820	158.540	.000
	Waste type * Ratios (A*B) *vrsta otpada * udjeli (A*B)*	44.209	3	14.736	9.949	.000
	Error / *pogreška*	146.632	99	1.481		
	Total / *ukupno*	15740.310	108			
24 hours TS, % *nakon 24 sata TS, %*	Waste type (A) / *vrsta otpada (A)*	11.948	1	11.948	6.920	.010
	Ratios (B) / *udjeli (B)*	843.293	3	281.098	162.810	.000
	Waste type * Ratios (A*B) *vrsta otpada * udjeli (A*B)*	65.133	3	21.711	12.575	.000
	Error / *pogreška*	170.928	99	1.727		
	Total / *ukupno*	18951.260	108			

lowest equilibrium moisture was in the winter tire group, with 40 % of waste tires. No correlation was observed between fully dry densities and moisture.

It was observed that the percentage of thickness swelling of the samples after soaking in water for 2 hours was at most 17.23 % in the control group, and for 24 hours, it was at the highest of 18.46 % in the control group. Therefore, to understand whether there was a statistically significant difference between the 2-hour and 24-hour thickness swelling results of the groups in Table 2, the results of the variance analysis are shown in Table 4, and the homogeneity results of the groups according to the Duncan analysis are shown in Table 5.

When the results of the analysis of variance were examined, it was seen that the tire type and tire amount have a combined effect on the changes in the thickness swelling values after 2 hours and 24 hours in water. As a result of the DUNCAN analysis, it was observed that there was a significant difference in the 95 % confidence interval between the control group, the summer group, and the winter groups in the samples kept in water for 2 hours. According to the analysis, swelling to the lowest thickness was observed in the groups using waste winter tires, while swelling to the maximum thickness was observed in the control groups.

When analyzing Table 4, it was observed that there was a significant difference in the 95 % confidence level between the groups as the amount of waste tire increased. However, after the 24-hour thickness swelling test, there was no significant difference in the

Table 5 Duncan's results of thickness swelling value
Tablica 5. Rezultati Duncanova testa debljinskog bubrenja

	Waste type / *Vrsta otpada*			**Mixture ratio** / *Omjer smjese*				
	Control *kontrolni uzorak*	Summer *ljetne*	Winter *zimske*	0 %	10 %	20 %	30 %	40 %
2 hour *TS*, % / *nakon 2 sata TS*, %	A	B	C	A	B	C	D	E
24 hour *TS*, % / *nakon 24 sata TS*, %	A	B	B	A	B	C	D	E

Table 6 Results of WA variance analysis
Tablica 6. Analiza varijance rezultata upijanja vode

Experiment *Eksperiment*	**Source** / *Izvor*	**Type III Sum of squares** *Zbroj kvadrata, tip III*	***df***	**Mean square** *Srednji kvadrat*	***F***	**Sig.**
2 hours *TS*, % *nakon 2 sata TS*, %	Waste type (A) / *vrsta otpada (A)*	93.622	1	93.622	2.586	.111*
	Ratios (B) / *udjeli (B)*	5357.367	3	1785.789	49.323	.000
	Waste type * Ratios (A*B) *vrsta otpada * udjeli (A*B)*	3436.747	3	1145.582	31.641	.000
	Error / *pogreška*	3584.393	99	36.206		
	Total / *ukupno*	884659.506	108			
24 hours *TS*, % *nakon 24 sata TS*, %	Waste type (A) / *vrsta otpada (A)*	121.518	1	121.518	4.127	.045
	Ratios (B) / *udjeli (B)*	3969.748	3	1323.249	44.938	.000
	Waste type * Ratios (A*B) *vrsta otpada * udjeli (A*B)*	1470.487	3	490.162	16.646	.000
	Error / *pogreška*	2915.190	99	29.446		
	Total / *ukupno*	1004140.459	108			

*Not significant $p < 0.05$ / *nije značajno za* $p < 0,05$

Table 7 Duncan's results of WA value
Tablica 7. Rezultati Duncanova testa upijanja vode

	Waste type / *Vrsta otpada*			**Mixture ratio** / *Omjer smjese*				
	Control *kontrolni uzorak*	Summer *ljetne*	Winter *zimske*	0 %	10 %	20 %	30 %	40 %
2 hours *TS*, % / *2 sata TS*, %	-	-	-	A	B	C	D	D
24 hours *TS*, % / *24 sata TS*, %	A	B	B	A	B	C	D	D

95 percent confidence interval between the summer and winter groups. The 95 percent confidence range decreased significantly as the amount of waste tire increased in both values.

The results of groups of 2-hour and 24-hour water absorption in Table 2 were analyzed with a variance test. The results of the variance test are shown in Table 6, and the homogeneity results of the groups according to the Duncan analysis are shown in Table 7. At the 95 percent confidence level, there was no statistically significant difference between tire types and control groups for 2-hour water absorption values, according to the analysis of variance. However, at the 95 percent confidence level, both the tire ratio and the intersection of the tire type and tire ratio have a statistically significant influence on the 2-hour water absorption values. The tire type, ratio, and interaction are statistically successful in the 95 percent confidence interval for the 24-hour test findings.

When the statistical distribution of the groups was investigated, it was discovered that as the number of waste tires increased, the weight gain percentages dropped. There was no statistically significant difference in the 95 percent confidence interval between the groups with 30 % and 40 % waste tire additives.

Thickness swelling in the samples showed a decrease between 8.77 % and 59.64 % compared to the control group. Weight gains decreased between 2.24 % and 24.56 %. When the studies in the literature were examined, the strength of the samples to water intake increased due to the increase in the percentage of rubber powders in the particleboard sample. The swelling values of particleboards to their thickness were between 14 % and 53 % lower than those of the control group (Ayrilmis *et al.,* 2009b). In addition, the average thickness swelling values of OSBs with rubber powder were determined to be between 11.4 % and 32.2 % lower than the values obtained from the control sample. It was stated that this decrease was between 16.4 % and 42.1 % with the use of polyisocyanate glue in another study (Ayrilmis *et al.,* 2009a). In addition, the study results showed significant improvements in the water absorption strength and thickness swelling properties of the samples depending on the increase in the waste tire powder ratio in the model (Ashori *et al.,* 2015; Abasi *et al.,* 2018).

3.2 Bending strength (*MOR*) and modulus of elasticity of bending (*MOE*)
3.2. Čvrstoća na savijanja (*MOR*) i modul elastičnosti (*MOE*)

Table 8 shows the results of the evaluated mechanical properties of the particleboard groups, while Tables 9 and 10 provide the results of variance analysis and Duncan's evaluation of the research.

Control samples showed the highest mechanical performance, according to Table 8. The second-best performance was 10 % added winter tire group, and generally, the mechanical performance of particleboards decreased with increasing tire content. A sig-

Table 8 Mechanical properties of boards with a standard deviation
Tablica 8. Mehanička svojstva ploča (u zagradi su standardne devijacije)

Groups	**Bending strength (*MOR*), N/mm²** *Čvrstoća na savijanje, (MOR),* N/mm²	**Modulus of elasticity (*MOE*), N/mm²** *Modul elastičnosti, (MOE),* N/mm²	**Internal bonding (*IB*), N/mm²** *Čvrstoća na raslojavanje, (IB),* N/mm²
Control *Kontrolni uzorak*	5.14 (0.69)	1100.33 (119.33)	0.33 (0.02)
S 10	3.63 (0.34)	807.11 (52.34)	0.26 (0.04)
S 20	2.77 (0.24)	629.25 (55.91)	0.22 (0.03)
S 30	2.10 (0.27)	460.15 (59.71)	0.20 (0.03)
S 40	1.60 (0.25)	310.82 (37.94)	0.15 (0.01)
W 10	4.88 (0.38)	973.75 (64.28)	0.32 (0.05)
W 20	4.41 (0.29)	902.77 (70.85)	0.31 (0.02)
W 30	2.72 (0.19)	661.84 (64.66)	0.22 (0.02)
W 40	2.46 (0.23)	454.36 (30.72)	0.23 (0.03)

Table 9 Results of mechanical properties variance analysis
Tablica 9. Analiza varijance rezultata mehaničkih svojstava

Experiment *Eksperiment*	**Source** / *Izvor*	**Type III Sum of squares** *Zbroj kvadrata, tip III*	***df***	**Mean square** *Srednji kvadrat*	***F***	**Sig.**
MOR	Waste type (A) / *vrsta otpada (A)*	21.470	1	21.470	156.180	.000
	Ratios (B) / *udjeli (B)*	57.347	3	19.116	139.052	.000
	Waste type * Ratios (A*B) *vrsta otpada * udjeli (A*B)*	2.688	3	.896	6.517	.001
	Error / *pogreška*	9.898	72	.137		
	Total / *ukupno*	1009.343	81			
MOE	Waste type (A) / *vrsta otpada (A)*	693958.038	1	693958.038	140.958	.000
	Ratios (B) / *udjeli (B)*	2712510.255	3	904170.085	183.657	.000
	Waste type * Ratios (A*B) *vrsta otpada * udjeli (A*B)*	43449.679	3	14483.226	2.942	.039
	Error / *pogreška*	354466.807	72	4923.150		
	Total / *ukupno*	45121340.646	81			
IB	Waste type (A) / *vrsta otpada (A)*	.060	1	.060	34.587	.000
	Ratios (B) / *udjeli (B)*	.143	3	.048	27.466	.000
	Waste type * Ratios (A*B) *vrsta otpada * udjeli (A*B)*	.016	3	.005	3.137	.031
	Error / *pogreška*	.125	72	.002		
	Total / *ukupno*	5.847	81	5.847		

nificant decrease in bending strength and modulus of elasticity was determined when the waste tire content exceeded 20 %. Generally, the winter tire groups have higher mechanical performance than the summer tire groups, according to Table 8. The waste tire type, waste tire content, and interactions significantly affect bending strength, modulus of elasticity, and internal bonding. The mechanical performance of the particleboards was observed with significant differences ($\alpha = 0.05$) according to the DUNCAN test (Table 10) and it was proved that mechanical performance decreased with increasing tire content.

As a result, it was observed that there was a decrease between 5.06 % and 68.87 % in the bending strength of the samples compared to the control samples and a reduction between 11.50 % and 71.75 % in the modulus of elasticity. It was reported that the bending strength and modulus of elasticity of particleboards

Table 10 Duncan's results of mechanical properties
Tablica 10. Rezultati Duncanova testa mehaničkih svojstava

	Waste type / *Vrsta otpada*			**Mixture ratio** / *Omjer smjese*				
	Control / *kontrolni uzorak*	Summer / *ljetne*	Winter / *zimske*	0 %	10 %	20 %	30 %	40 %
MOR	A	C	B	A	B	C	D	E
MOE	A	C	B	A	B	C	D	E
IB	A	C	B	A	B	C	D	D

and plywood decreased as the percentage of rubber powder in the sample increased. In the literature, the average bending strength values of the samples were found to be between 13 and 58 percent lower than the values of the control samples (Ayrilmis *et al.*, 2009b; Abasi *et al.,* 2018). A similar decreasing trend was shown in the bending strength and modulus of elasticity of the samples.

It was reported that winter tire rubbers contain pores in their structure. These pores absorb the water film formed on the road surface due to sun or contact pressure that causes snow melting. In addition, it was stated that winter tires have different hysteresis properties from summer tires due to the different polymer structure and additives content (Dörrie *et al.,* 2010; Ludvigsen, 2017). Therefore, it was thought that the bending strength and modulus of elasticity of the samples produced from winter tire wastes are higher than those of the summer tire group due to the formation of a stronger bond between glue-chip and waste tire. On the other hand, the Poisson's ratio of rubber is close to 0.5; this indicates that the rubber material is almost incompressible in bulk. Therefore, changing the ratios between wood chips and rubber powders negatively affects the bending strength of the particleboard (Ayrilmis *et al*., 2009b).

A decrease between 4.51 % and 54.66 % was observed in the internal bonding values compared to the control samples. It was stated that there was a significant decrease due to the increase in the percentage of tire content in the board, the average internal bonding values of OSB panels being between 17.6 % and 48.5 % lower than the values of the control samples (Ayrilmis *et al.,* 2009a). The reason for this situation was that the strength of the chip was not sufficient due to the concentration of the tensile force applied to the sample in the rubber chip, which has low strength properties. This situation causes a significant decrease in the internal bonding values of the samples (Ayrilmis *et al.,* 2009b).

The incorporation of waste tire particles into the polymer matrix often results in the deterioration of the mechanical performance of the materials because of insufficient interfacial interaction between the matrix and the filler (Ramarad *et al.,* 2015; Xu *et al.,* 2020). In addition, it was stated that the increase in porosity in the structure causes a decrease in the strength of the boards (Nazerian *et al.,* 2020). The porosity in the board structure and microstructure can be seen in Figure 3 and Figure 4. In Figure 3, waste tire powders can be seen with the naked eye within the board structure. It can also be seen that the core layer porosity is significantly higher than that of the control group. Similarly, it was observed that the rubber powders showed an uneven distribution among the wood chips in the surface layer. The SEM photographs in Figure 4 show that there are incompatibility and local micro gaps between the wood and rubber structures in the microstructure compared to the control group. On the other hand, it was reported that unmodified waste tire powders are mostly non-polar and contain few functional groups in the surface layer necessary for bonding (Hejna *et al.,* 2020). For this reason, it was stated that it would be very beneficial to create various functional groups on the waste tire powder surfaces that could increase the interfacial interactions between the continuous and dispersed phases (Hejna *et al.,* 2020). For this purpose, it was recommended to add a suitable coupling agent to the waste tire powders and thus modify or improve these wood particles. To enhance mechani-

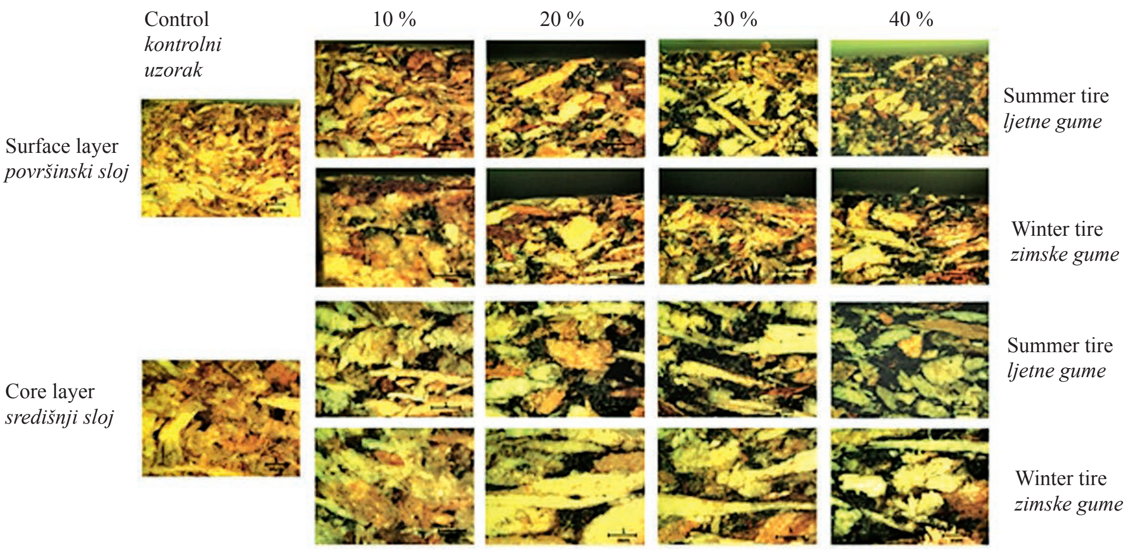

Figure 3 Stereo microscope examination
Slika 3. Pregled stereomikroskopom

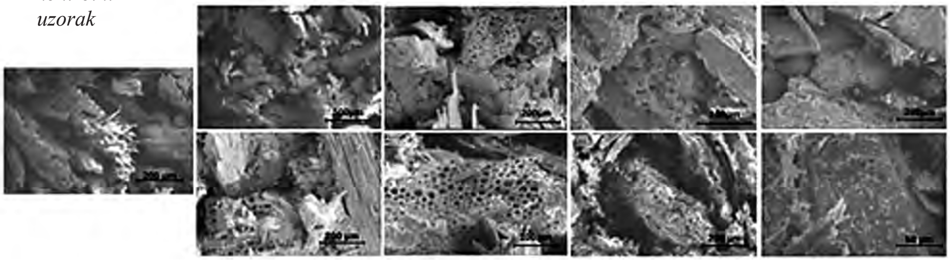

Figure 4 Scanning electron microscope (SEM) examination
Slika 4. Pregled pretražnim elektronskim mikroskopom (SEM)

cal strength, it would be recommended to use chemicals such as maleic anhydride or glycidyl methacrylate as binders in future studies. (Wang *et al.,* 2003). On the other hand, an excessive increase in press temperature can reduce the strength of fiber components. Therefore, it would be recommended to increase the press time as an alternative to higher press temperature to guarantee a complete cure of the resin (Nazerian *et al.,* 2020).

4 CONCLUSIONS
4. ZAKLJUČAK

The following conclusions have been made from the results of the present study:
- The groups containing waste winter tire powder achieved better performance than summer waste tire groups.
- The use of waste rubber in sample production resulted in a decrease in the equilibrium moisture content of the samples.
- The hydrophobic nature of the tire rubber enhanced the water absorption strength of particleboards.
- Mechanical performance of groups was affected by tire content, and it was mainly determined that the values decreased dramatically between 20 % to 30 % waste tire ratio groups.

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Physical and Chemical Properties of Highland Bamboo (*Yushania alpina*) Culms Grown in Ethiopia

Fizička i kemijska svojstva gorskog bambusa (*Yushania alpina*) uzgojenoga u Etiopiji

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ABSTRACT • *Bamboo is the fastest growing plant currently known on earth, a property that enables it to be the best alternative as a future source of wood fiber. This study investigated the effect of site and culm height on the physical and chemical properties of Yushania alpina culms grown in Ethiopia. Matured Yushania alpina 3 to 5-year-old samples were harvested from Hagere-Selam and Rebu-Gebeya sites. The culms were subdivided into three equal lengths (bottom, middle, and top), and the variations in physical and chemical properties between the two sites and the culm heights of Yushania alpina were investigated. The results showed that the average values of MC, basic density, tangential and longitudinal shrinkage of Yushania alpina culms for Hagere-Selam and Rebu-Gebeya sites were (91.78 and 80.32 %), (0.65 and 0.63 g/cm³), (6.63 and 5.84 %) and (0.63 and 0.56 %), respectively. The average values of cellulose, lignin, extractive and ash contents in the culms for Hagere-Selam and Rebu-Gebeya sites were (52.84 and 50.71 %), (26.55, and 26.04 %), (8.41 and 8.02 %) and (1.95 and 2.17 %), respectively. The results revealed that the site affected the MC, basic density, cellulose, lignin, extractive, and ash contents of Yushania alpina culms but not the tangential and longitudinal shrinkage. The culm height of Yushania alpina affected MC, basic density, tangential shrinkage, longitudinal shrinkage, cellulose, lignin, extractive, and ash contents. In the case of both sites, the highest percentages of MC, tangential and longitudinal shrinkage, and ash content were observed at the base and lowest at the top of the culms. On the contrary, both sites observed the highest magnitude of basic density, cellulose and extractive at the top and lowest at the base of the culms. The variations in physical and chemical properties at different sites and culm heights influence the utilization of Yushania alpina culms for industries and end products.*

KEYWORDS: *bamboo (Yushania alpina); basic density; cellulose; lignin; moisture content; shrinkage*

SAŽETAK • *Bambus je trenutačno najbrže rastuća biljka u svijetu, što je čini najboljom alternativom za budući izvor drvnih vlakana. Ovom je studijom istraživan utjecaj staništa i visine bambusa Yushania alpina, uzgojenoga u Etiopiji, na fizička i kemijska svojstva njegove stabljike. Uzorci Yushania alpina stari tri do pet godina skupljeni su sa staništa Hagere-Selam i Rebu-Gebeya. Stabljike su podijeljene na tri jednake duljine (baza, sredina i vrh) na kojima su istraživane varijacije fizičkih i kemijskih svojstava stabljike s obzirom na dva staništa i visinu bambusa. Rezultati su pokazali da su prosječne vrijednosti sadržaja vode, gustoće te tangentnoga i uzdužnog utezanja*

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stabljike Yushania alpina sa staništa Hagere-Selam i Rebu-Gebeya bile 91,78 i 80,32 %; 0,65 i 0,63 g/cm³; 6,63 i 5,84 % te 0,63 i 0,56 %, a prosječne vrijednosti sadržaja celuloze, lignina, ekstraktiva i pepela sa staništa Hagere-Selam i Rebu-Gebeya bile su 52,84 i 50,71 %; 26,55 i 26,04 %; 8,41 i 8,02 % te 1,95 i 2,17 %. Rezultati su pokazali da je stanište utjecalo na sadržaj vode, gustoću te sadržaj celuloze, lignina, ekstraktiva i pepela u stabljikama Yushania alpina, ali ne i na tangentno i uzdužno utezanje. Visina stabljike Yushania alpina utjecala je na sadržaj vode, gustoću, tangentno i uzdužno utezanje te na sadržaj celuloze, lignina, ekstraktiva i pepela. Za oba staništa utvrđen je najveći sadržaj vode, tangentno i uzdužno utezanje te sadržaj pepela u bazi stabljike, a najmanji u vrhu stabljike. Nadalje, u vrhu stabljike s oba staništa primijećena je najveća gustoća, sadržaj celuloze i ekstraktiva, a najmanje su vrijednosti izmjerene u bazi stabljike. Varijacije fizičkih i mehaničkih svojstava s obzirom na stanište i visinu stabljike utječu na upotrebu stabljike Yushania alpina u industriji i za završne proizvode.

KLJUČNE RIJEČI: *bambus (Yushania alpina); gustoća; celuloza; lignin; sadržaj vode; utezanje*

1 INTRODUCTION
1. UVOD

Bamboo is the fastest growing plant currently known on earth, a property that enables it to be the best alternative as a future source of wood fiber (Liese and Köhl, 2015). Unlike timber, bamboo culms need short rotation (3-5 years) to mature before they can be harvested and utilized (Liese and Köhl, 2015). Bamboo is a perennial plant that belongs to the subfamily Bambusoideae of the family Poaceae (Gramineae), and it contains more than 1,500 species. It provides more than 1,500 applications, from traditional utilization in rural areas to industrial production, construction, and other versatile uses (Liese, 1987; Zhaohua, 2011; Hinde and Kaba, 2018). Bamboo has a higher strength-to-weight ratio than wood, enabling easy harvesting, transporting, and manufacturing of products (Wahab *et al.*, 2009; Anokye *et al.*, 2014). The above characteristics of bamboo have encouraged and intensified bamboo research in recent years.

The utilization of bamboo for various applications is governed by its properties, like any wood material. Moisture content, density, and shrinkage are physical properties that influence the dimensional stability, toughness, strength, working properties, and durability of bamboo and bamboo products (Liese and Köhl, 2015). The basic chemical properties found in bamboo are cellulose, hemicellulose, and lignin, which influence its utilization for different applications (Liese, 1985). The properties of bamboo culms are mainly affected by culm position, age, topography, and climate (Liese and Köhl, 2015; Tolessa *et al.*, 2019). The physical and chemical properties of bamboo vary between species, sites, age, and different parts of culm positions (Santhoshkumar and Bhat, 2015; Liese and Köhl, 2015; Tolessa *et al.*, 2019). Information on the physical and chemical properties of bamboo is necessary for assessing its suitability for various end products (Kamruzzaman *et al.*, 2008; Tolessa *et al.*, 2019). Such information will also enable increasing the utilization of bamboo species as substitutes for solid wood in wood-based industries.

Recently, *Yushania alpina* (*Y. alpina*) culms and splints have been used to construct traditional houses, rudimentary furniture, handcrafts, mats, fencing, beehive, and household utensils (baskets, winnowing trays) (Desalegn and Tadesse, 2014; Desalegn, 2015). The highland bamboo (*Y. alpina*) is indigenous to Ethiopia and available in the highlands of Kenya, Sudan, Zambia, Zaire, Burundi, Rwanda, Cameroon, and Tanzania (Embaye *et al.*, 2003). This bamboo species is the most cultivated and widespread throughout the country. It is preferred mainly because of its suitability and ease of processing and converting into different products.

In Ethiopia, the multiple uses of bamboo in industrial applications are not getting the most economic advantage, and its utilization is limited to domestic services (Mulatu and Kindu, 2010; Zenebe *et al.*, 2014). This is due to insufficient basic information on its properties. Previously, few studies have been done on physical and chemical properties (Muche and Degu, 2019; Tsegaye *et al.*, 2020; Dessalegn *et al.*, 2021). There is still limited information about the variation of physical and chemical properties between sites, within culms, and along the culm heights of *Y. alpina* grown in Ethiopia. Therefore, this study investigated the effect of site and culm heights on the physical and chemical properties of *Y. alpina* grown at Hagere-Selam (Sidama Region) and Rebu-Gebeya (Amhara Region).

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

A total of twenty matured *Yushania alpina* culm samples (3-5 years old) were harvested from potential sites of Hagere-Selam (Sidama Region) and Rebu-Gebeya (Amhara Region). First, the bamboo culms were harvested, and their branches were removed from the top parts of the culms leaving the entire length to be about 9 m. After that, the culms were subdivided into three equal lengths, labeled bottom, middle, and top portions, with lengths of 3 m (Liese and Köhl, 2015).

Table 1 Climate and altitude of Hagere-Selam and Rebu-Gebeya where *Y. alpina* culm samples were harvested
Tablica 1. Klima i nadmorska visina Hagere-Selama i Rebu-Gebeye, na kojima su prikupljeni uzorci stabljika *Y. alpina*

Parameters / *Parametri*	**Sites** / *Staništa*	
	Hagere-Selam	**Rebu-Gebeya**
Altitude m.a.s.l, m / *nadmorska visina,* m	1500-2850	2300-4100
Annual average precipitation range, mm/year *raspon prosječnih godišnjih padalina*, mm/god.	1000-1600	900-1500
Annual average temperature range, °C *raspon prosječne godišnje temperature*, °C	11-22	0-15

Table 2 Description of *Y. alpina* culms grown at Hagere-Selam and Rebu-Gebeya
Tablica 2. Svojstva stabljika *Y. alpina* sa staništa Hagere-Selam i Rebu-Gebeya

Parameters / *Parametri*	**Sites** / *Staništa*	
	Hagere-Selam	**Rebu-Gebeya**
Height / *visina,* m	13	12
Diameter / *promjer,* mm	70-90	50-80
Culm thickness / *debljina stabljike*, mm	7.2	6

Hagere-Selam site lies on geographical coordinates of 6°29'0" North, 38°31'0" East; whereas Rebu-Gebeya coordinates are 10°33'0" North, 37°46'0" East.

2.1 Determination of physical properties
2.1. Određivanje fizičkih svojstava

2.1.1 Determination of moisture content
2.1.1. Određivanje sadržaja vode

Three centimeters long specimens representing two sites (Hagere-Selam and Rebu-Gebeya) and three culm heights (base, middle, and top) were cut from fresh *Y. alpina* to determine its initial moisture content. Green weight of each specimen was measured using an analytical balance with a 0.01g accuracy (IS 6874, 2008). The specimens were then oven-dried at a temperature of (103±2) °C until attaining constant weight. The moisture content was calculated using Eq 1.

$$Moisture\ content\ (\%) = \frac{W_g - W_{od}}{W_{od}} \cdot 100 \qquad (1)$$

Where W_g is the green weight of specimens and W_{od} is the oven-dry weight of the specimen

2.1.2 Determination of basic density
2.1.2. Određivanje nominalne gustoće uzoraka

Three centimeters long specimens, (representing 2 sites and three culm heights), were cut from fresh *Y. alpina* culms for the determination of basic density. The green weights of all specimens were measured using an analytical balance with an accuracy of 0.01 g. The water displacement method was used to determine the volume of each specimen. The specimens were then oven-dried at a temperature of (103±2) °C. The repeated measurement of weight was recorded until the constant weight was reached. Basic density was determined based on ISO 22157-2:2004 and IS 6874 (2008). Basic density was calculated using Eq 2.

$$Basic\ density = \frac{Ovendry\ weight\,[\mathrm{g}]}{Green\ volume\,[\mathrm{cm}^3]} \qquad (2)$$

2.1.3 Determination of shrinkage
2.1.3. Određivanje utezanja

Specimens representing 2 sites and three culm heights were prepared from round-shaped *Y. alpina* culms, 3-cm in length, to determine tangential and longitudinal shrinkage. For each specimen, the green weight and dimensions of wall thickness at four points and lengths at four points in green conditions were measured using an analytical balance and digital caliper with an accuracy of 0.01g and 0.01 mm, respectively (Figure 1). The specimens were oven-dried at a temperature of (103±2) °C. The repeated measurements for weight were recorded until constant weight was reached. Shrinkage was determined based on ISO 22157-2:2004 and IS 6874 (2008). The shrinkage was calculated using Eq 3.

$$Shrinkage\,(\%) = \frac{D_i - D_f}{D_i} \cdot 100 \qquad (3)$$

Where D_i is the initial dimension of the specimens before oven-drying (mm) and D_f is the final dimension of the specimens after oven-drying (mm)

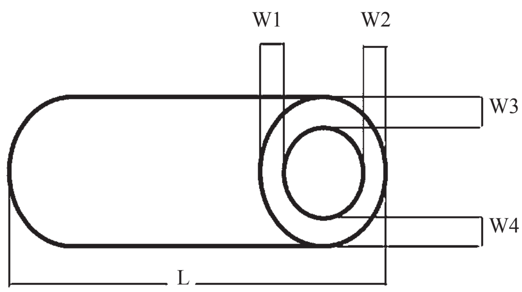


Figure 1 Position and direction measurement for wall thickness and longitudinal shrinkage
Slika 1. Mjerna mjesta i smjer mjerenja debljine stijenke i uzdužnog utezanja

Table 3 Standard methods used for chemical analysis of bamboo culms
Tablica 3. Standardne metode primijenjene za kemijsku analizu stabljika bambusa

Chemical composition *Kemijski sastav*	**Standard** *Standard*
Cellulose content *sadržaj celuloze*	Kurschner-Hoffer method
Klason lignin content *sadržaj Klasonova lignina*	ASTMD 1106-56
Alcohol-toluene solubility *topljivo u smjesi alkohola i toluena*	ASTMD 1107-56
Ash content / *sadržaj pepela*	ASTMD 1102-84

2.2 Determination of chemical composition
2.2. Određivanje kemijskog sastava

The harvested bamboo culms were dried and converted into small-size strips suitable for further milling processes. Thereafter, they were placed in a hammer mill and Willey mill to reduce it to the appropriate size. Then the milled powder bamboo samples were filtered using a 40 mesh size (425 µm) and 60 mesh sieve (250 µm). The particles were stored in an airtight container labeled with the appropriate code for chemical analysis.

The chemical composition including extractive (alcohol-toluene solubility), ash, and lignin content were determined using the standard procedures of the American Society for Testing Materials (ASTM) (Table 3). Cellulose content was determined according to alkali extraction and Kurchner-Hoffer method (Brown, 1975). Toluene was used instead of benzene and reported as alcohol-toluene extractive (Tolessa *et al.*, 2017). The amounts were expressed on a percentage basis of the starting oven-dry mass. The lignin content test was performed with extractive-free bamboo derived from the alcohol-toluene extractive test.

2.3 Statistical analysis
2.3. Statistička analiza

The data were analyzed statistically to assess significant differences between the two sites (Hagere-Selam and Rebu-Gebeya) and along the three culm heights (base, middle, and top) using descriptive statistics and analysis of variance (ANOVA) by R software, version 4.1.3. The least significant difference (LSD) was used for mean comparison at $p<0.05$.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Physical properties
3.1. Fizička svojstva

3.1.1 Moisture content
3.1.1. Sadržaj vode

Analysis of variance for the initial moisture content of *Y. alpina* culms harvested from Hagere-Selam and Rebu-Gebeya sites is presented in Table 4. The results revealed that *Y. alpina* culms harvested from Hagere-Selam had a higher value of initial MC than those harvested from Rebu-Gebeya. These differences may be associated with the age and season of felling of bamboo culms (Liese, 1985). For this study, the culms 3 to 5 years old were harvested during the dry season but in different months.

The results revealed that the values of initial MC for the two sites decreased from the base to the top position of the culms (Table 4). Similar trends to this finding were seen in *Bambusa balcooa*, *Bambusa tulda*, *Bambusa salarkhanii*, and *Melocanna baccifera*, grown in Bangladesh (Kamruzzaman *et al*. 2008). Other researchers also found similar variations in their studies (Wahab *et al*., 2009; Anokye *et al*., 2016; Zakikhani *et al*., 2017). According to Wahab *et al*. (2009), the variation of *MC* along the culm height was due to differences in anatomical structure and chemical composition between locations along the bamboo culms. On the other hand, the decreasing trend of initial *MC* might be due to a smaller proportion of vascular bundles at the bottom when compared to the top position of culms (Anokye *et al*., 2014).

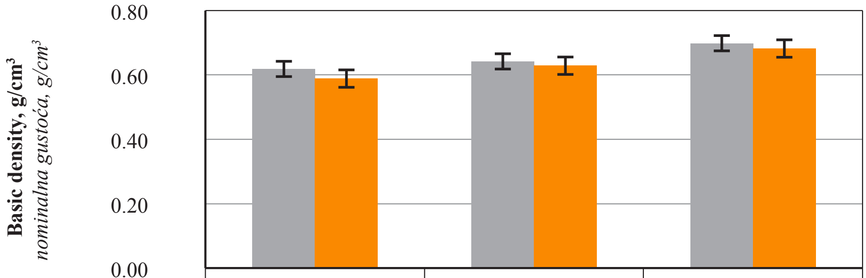
3.1.2 Basic density
3.1.2. Nominalna gustoća

Density is among the main factors that affect the utilization of bamboo culm as raw material. The over-

Table 4 Analysis of variance for basic density and shrinkage at different sites and culm heights of *Yushania alpina*
Tablica 4. Analiza varijance nominalne gustoće i utezanja uzoraka *Yushania alpina* s različitih staništa i različite visine stabljike

Mean-squares and statistical significances / *Srednji kvadrati i statistička značajnost*					
Basic density, g/cm³ and shrinkage of oven-dry, % / *Nominalna gustoća,* g/cm³ *i utezanje nakon sušenja, %*					
Source of variation *Izvor varijacije*	***DF***	**Initial *MC*** *Početni sadržaj vode*	**Basic density** *Nominalna gustoća*	**Tangential** *Tangentno utezanje*	**Longitudinal** *Uzdužno utezanje*
Site (S) / *stanište (S)*	1	3272***	0.013**	0.298^{ns}	0.009^{ns}
Culm high (P) / *visina stabljike (P)*	2	7904***	0.030***	17.626***	1.940***
S:P	3	31**	0.000^{ns}	0.014^{ns}	0.000**

^{ns} Not significant at $p>0.1$, * significant at $p<0.05$, ** significant at $p<0.01$, *** significant at $p<0.001$, DF-degree of freedom / ^{ns} *nije značajno za p>0,1, * značajno za p<0,05, ** značajno za p<0,01, *** značajno za p<0,001, DF – stupanj slobode*



	Bottom	Middle	Top
Hagere-Selam	0.62	0.64	0.70
Rebu-Gebeya	0.59	0.63	0.68

Figure 2 Basic density variation in *Y. alpina* culms at different sites and culm heights
Slika 2. Varijacije nominalne gustoće stabljike *Y. alpina* za različita staništa i visine stabljike

all mean values of basic density for the Hagere-Selam and Rebu-Gebeya sites were 0.65 and 0.63 g/cm^3, respectively. This shows that the culms harvested from the Hagere-Selam site were significantly denser than those from the Rebu-Gebeya site (Table 4). Generally, the density of bamboo ranges from about 0.4 to 0.9 g/cm^3 depending on the anatomical structure reflected in the quantity and distribution of fibers around the vascular bundles (Zakikhani *et al*., 2017). The density of this finding was in the range of generally recognized values of bamboo density. The result shows that the culm height had significant effects on the basic density at $p<0.001$ level, whereas the site had a significant effect on basic density at $p<0.01$ level (Table 4). However, the table shows that the interaction effect between the site and culm height did not significantly affect the basic density at $p>0.05$ level (Table 4).

The results revealed that the basic density significantly increased along with the height of *Y. alpina* culms for both sites (Figure 2). Many researchrs reported similar trends to this finding. They found an increase in basic density with increasing height of bamboo culms from the base to the top (Wahab *et al*., 2009; Santhoshkumar and Bhat, 2015; Vetter *et al.,* 2015). The increase of basic density from the base towards the top position of culms was also reported for thirteen bamboo species grown in Malaysia (Siam *et al*., 2019). This variation is associated with anatomical structure variations at different bamboo culms heights (Liese, 1985). According to Santhoshkumar and Bhat (2015), the increase in the magnitude of density from the bottom to the top position of the bamboo culm was due to the increase in the proportion of fibrous tissue and increased frequency of the occurrence of vascular bundles.

3.1.3 Shrinkage
3.1.3. Utezanje

Shrinkage is another main factor that affects the utilization of bamboo culm as a raw material in different wood industries. Unlike wood, bamboo begins to shrink from the very beginning of drying. The results revealed that the overall mean tangential or culm-wall shrinkage for the Hagere-Selam and Rebu-Gebeya sites was 6.63 % and 5.84 %, respectively (Figure 5). The overall mean longitudinal shrinkage for the Hagere-Selam and Rebu-Gebeya sites was 0.63 % and 0.56 %, respectively (Figure 5). Shrinkage of the *Y. alpina* culm grown at Hagere-Selam was higher in both tangential (culm-wall thickness) and longitudinal direction when compared to the Rebu-Gebeya site (Figure 5). This variation may be related to the initial MC and the culm-wall thicknesses of the bamboo culms (Siam *et al*., 2019).

The tangential and longitudinal shrinkage of *Y. alpina* culm at different heights for Hagere-Selam and Rebu-Gebeya sites is presented in Figure 3 and Figure 4, respectively.

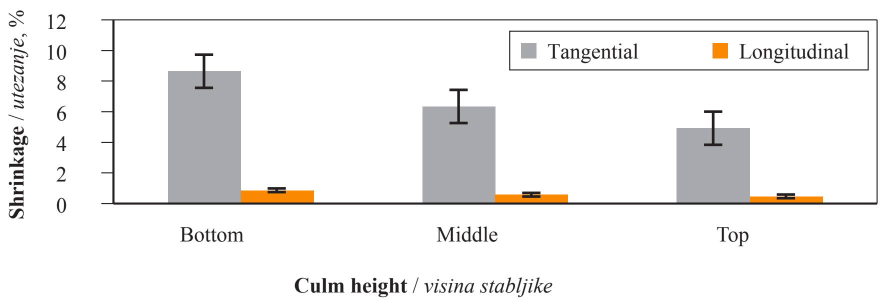


Figure 3 Tangential and longitudinal shrinkage at different culm height of *Y. alpina* grown at Hagere-Selam
Slika 3. Tangentno i uzdužno utezanje uzoraka za različite visine stabljike *Y. alpina* sa staništa Hagere-Selam

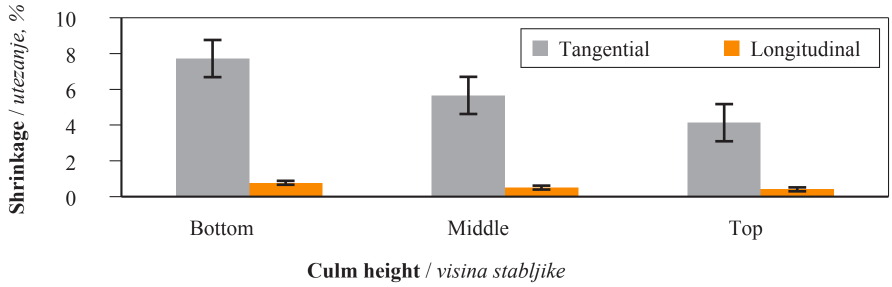


Figure 4 Tangential and longitudinal shrinkage at different culm height of *Y. alpina* grown at Rebu-Gebeya
Slika 4. Tangentno i uzdužno utezanje uzoraka za različite visine stabljike *Y. alpina* sa staništa Rebu-Gebeya

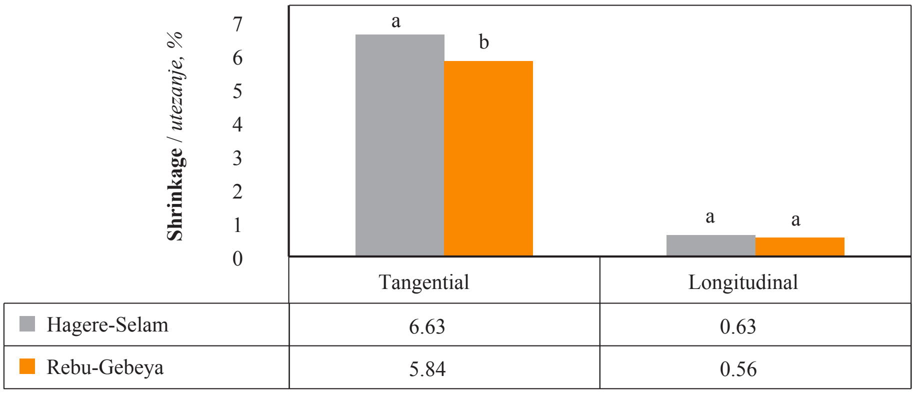


Figure 5 Tangential and longitudinal shrinkage in *Y. alpina* culms at different sites
Slika 5. Tangentno i uzdužno utezanje uzoraka *Y. alpina* s različitih staništa

The statistical analysis of variance revealed that site and culm height had a highly significant effect on tangential shrinkage at $p<0.001$ (Table 4). The same table shows that the culm height had a significant effect on longitudinal shrinkage at $p<0.001$, whereas the site had a significant effect on the longitudinal shrinkage at $p<0.05$. However, the interaction between the site and the culm height position had an insignificant effect on the tangential and longitudinal shrinkage at $p>0.05$ (Table 4).

The results showed that the tangential and longitudinal shrinkage of the culm decreased from the base to the top positions of *Y. alpina* for both sites (Figure 3, Figure 4). A similar trend was observed in *Bambusa balcooa*, *Bambusa tulda, Bambusa salarkhanii,* and *Melocanna baccifera* species grown in the Philippines (Kamruzzaman *et al*., 2008). These variations are associated with higher parenchyma cell content and fewer vascular bundles at the base positions. On the other hand, lower parenchyma cell content and a higher number of vascular bundles are found at the top positions of bamboo culms (Wahab *et al*., 2009).

3.2 Chemical composition
3.2. Kemijski sastav

3.2.1 Cellulose content
3.2.1. Sadržaj celuloze

Cellulose is the main constituent of lignocellulosic wood material, and it is located predominantly in the secondary cell wall. The analysis of variance shows that the site and culm height had a significant ($p<0.001$) effect on the cellulose content of *Y. alpina* bamboo (Table 5). The same table shows that the interaction effect between site and culm height did not show a significant ($p>0.05$) effect on cellulose content (Table 5). The overall mean value of cellulose content in *Y. alpina* culms grown at Hagere-Selam was 52.84 %, which is statistically higher than the bamboo culms collected from the Rebu-Gebeya site, which was 50.71 %. The results obtained from this study were higher than those found in the previous study on the same bamboo species (46.76 %) (Tsegaye *et al*., 2020). The cellulose content of this finding was in the range of softwood (40 – 52 %) and hardwoods (38 – 56 %). Normally, the cellulose content of bamboo ranges from 40 – 50 % (Fengel and Wegener, 1984). The results obtained in this study were in the range of the values mentioned above for woods and other bamboo species. Bamboo species with cellulose content in this range are suitable for pulp and papermaking, bioenergy, and biobased composite production (Hammett *et al*., 2001; Li *et al*., 2007). It can also be used for applications similar to those of softwood and hardwood.

In the case of both sites, statistically, the highest cellulose contents were observed at the top; followed by the middle and lowest at the bottom position (Figure 8, Figure 9). The percentage cellulose content of *Y. alpina* culms for both sites showed an increasing trend from the base to the top (Figure 8, Figure 9). The same trend to this finding was reported for the same bamboo species at age of three (Tolessa *et al*., 2019). The increased cellulose content was associated with the increase in cell wall thickness (Tolessa *et al*., 2017).

3.2.2 Klason lignin content
3.2.2. Sadržaj Klasonova lignina

Lignin is a phenolic substance consisting of an irregular array of variously bonded hydroxyl- and methoxy-substituted phenylpropane units. Statistically, the overall mean values of lignin content of *Y. alpina* culms collected from the Hagere-Selam (26.55 %) differed insignificantly from the culms collected from the Rebu-Gebeya site (26.04 %) (Figure 7). The lignin content in different tropical bamboo species was reported in the range of 24.84 to 32.65 % (Razak *et al*., 2013). Fengel and Wegner (1984) investigated the ranges of lignin for softwoods (24 – 37 %) and hardwoods (17 – 30 %). The results obtained from this study were in the range of the above reports for bamboo, softwood, and hardwood. According to Zhang *et al*. (2022), the high lignin content of bamboo culms can provide excellent physical and mechanical properties. On the other hand, the high lignin content contributes to bamboo rigidity and makes it suitable for structural applications, such as construction and furniture-making.

The analysis of variance shows that the culm height had a significant effect on lignin content at $p<0.01$ (Table 5). The site showed a significant ($p<0.1$) effect on lignin content (Table 5). However, the interaction effect between the site and culm height did not show a significant ($p>0.05$) effect on the lignin content of the culms (Table 5). The results revealed that the highest percentage of lignin content was observed at the bottom; followed by middle and minimum at the top culm positions for both sites (Figure 8, Figure 9). A similar variation pattern to this finding was observed in the same bamboo species (Tolessa *et al*., 2019). A similar variation pattern to this study was reported for *Melocanna baccifera* (Hossain *et al*., 2022).

3.2.3 Extractive content
3.2.3. Sadržaj ekstraktiva

Non-structural chemical compositions found in wood and bamboo materials are known as extractive substances. On the other hand, extractives in bamboo are non-cell wall components with diverse chemical compositions such as resins, lipids, waxes, tannins, pentosan, hexosan, starch, and silica (Fengel and Wegener, 1984). According to the analysis of variance, site and culm height showed a significant ($P<0.001$) effect on the extractive content (Table 5). However, the interaction effect between the site and culm height did not show a significant effect on the extractive content of *Y. alpina* at $p>0.05$ (Table 5).

The overall mean value of extractive content in *Y. alpina* culms collected from Hagere-Selam was 8.41 %, which is insignificantly higher than the culms collected from the Rebu-Gebeya site, which was 8.02 %. Extractives in bamboo can enhance the structural rigidity of its cell wall and effectively resist diseases and pests/decay (Zhang *et al*., 2022).

Statistically, in the case of both sites, the highest extractive contents were observed at the bottom position; followed by the middle and lowest at the top position (Figure 8, Figure 9). The same variation pattern to this finding was reported in *Y. alpina* culms (Tolessa *et al*., 2019).

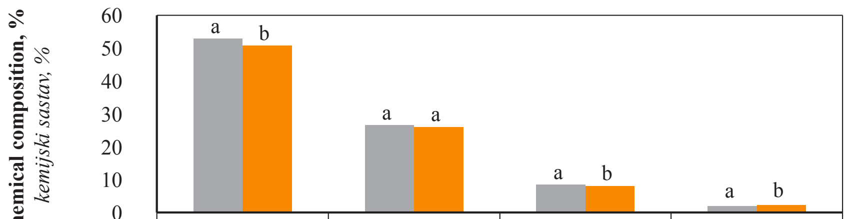
3.2.4 Ash content
3.2.4. Sadržaj pepela

Ash is a term generally used to refer to inorganic substances such as silicates, sulfates, carbonates, or metal ions. The analysis of variance shows that the main effects of site and culm height had a significant effect on ash content at $p<0.001$ (Table 5). However, the interaction effects between the site and culm height did not show a significant effect on ash content at $p>0.05$ (Table 5). The overall mean value of ash content in *Y. alpina* culms collected from Rebu-Gebeya was 2.17 %, which is significantly higher than the culms collected from the Hagere-Selam site, which was 1.95 %. Liese and Köhl (2015 stated that bamboo growth sites affect the amount of ash in bamboo. This difference might be due to the topography, soil, and climate of the area where the bamboo culms are grown. Values lower than those determined in this study were reported for highland bamboo, namely 1.87 % (Tolessa

Table 5 Analysis of variance for cellulose, lignin, extractive, and ash contents in *Y. alpina* culms at different sites and culm heights
Tablica 5. Analiza varijance sadržaja celuloze, lignina, ekstraktiva i pepela stabljike *Y. alpina* za različita staništa i visine stabljike

Mean-squares and statistical significances / *Srednji kvadrati i statistička značajnost*					
Chemical composition in *Yushania alpina* bamboo culms, % / *Kemijski sastav stabljike bambusa Yushania alpina, %*					
Source of variation *Izvor varijacije*	**DF**	**Cellulose** *Celuloza*	**Lignin**	**Extractive** *Ekstraktivi*	**Ash** *Pepeo*
Site (S) / *stanište (S)*	1	40.87***	2.38	1.37***	0.45**
Culm height (P) / *visina stabljike (P)*	2	149.72***	27.92***	1.89***	5.01***
S:P	2	74^{ns}	0.45^{ns}	0.039^{ns}	0.022^{ns}

^{ns} Not significant at $p>0.1$, * significant at $p<0.05$, ** significant at $p<0.01$, *** significant at $p<0.001$, '.' is significant at p<0.1, DF-degree of freedom / ^{ns} *nije značajno za p>0,1, * značajno za p<0,05, ** značajno za p<0,01, *** značajno za p<0,001, '.' značajno za p<0,1, DF – stupanj slobode*

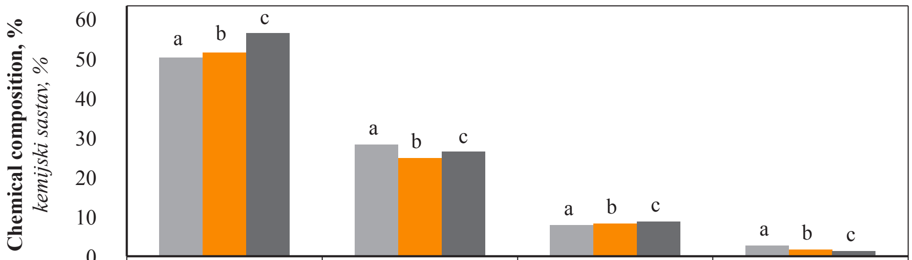


	Cellulose	Lignin	Extractive	Ash
Hagere-Selam	52.84	26.55	8.41	1.95
Rebu-Gebeya	50.71	26.04	8.02	2.17

Figure 6 Chemical composition variation in *Y. alpina* culms at different sites (within the same parameter having the same letters, there is no significant difference between the two sites at p<0.05)
Slika 6. Varijacije kemijskog sastava stabljika *Y. alpina* s različitih staništa (za iste parametre s istim slovima nema značajne razlike između dva staništa za p<0,05)

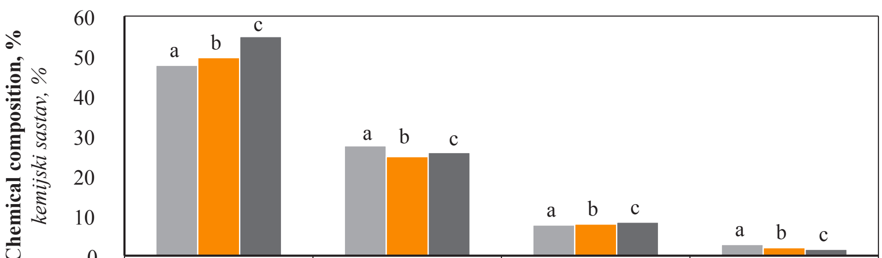
et al., 2019). However, higher than these values were reported for the same bamboo species, namely 3.77 % (Tsegaye *et al*., 2020).

Statistically, in the case of both sites, the highest ash contents were observed at the bottom position; followed by the middle and lowest at the top position (Figure 7, Figure 8). The same variation to this finding was reported in *Y. alpina* culms at the age of three (Tolessa *et al*., 2019). According to Tolessa *et al*. (2019), the ash content variation along the culm height varied with moisture content.



	Cellulose	Lignin	Extractive	Ash
Bottom	50.33	28.26	7.97	2.71
Middle	51.60	24.86	8.38	1.73
Top	56.60	26.53	8.87	1.40

Figure 7 Chemical composition variation in *Y. alpina* at different culm heights grown at Hagere-Selam (within the same parameter having the same letters, there is no significant difference between the two sites at p<0.05)
Slika 7. Varijacije kemijskog sastava *Y. alpina* pri različitim visinama stabljike za stanište Hagere-Selam (za iste parametre s istim slovima nema značajne razlike između dva staništa za p<0,05)



	Cellulose	Lignin	Extractive	Ash
Bottom	47.65	27.49	7.71	2.84
Middle	49.59	24.80	7.95	2.03
Top	54.90	25.83	8.39	1.64

Figure 8 Chemical composition variation in *Y. alpina* at different culm heights grown at Rebu-Gebeya (within the same parameter having the same letters, there is no significant difference between the two sites at p<0.05)
Slika 8. Varijacije kemijskog sastava *Y. alpina* pri različitim visinama stabljike za stanište Rebu-Gebeya (za iste parametre s istim slovima nema značajne razlike između dva staništa za p<0,05)

4 CONCLUSIONS
4. ZAKLJUČAK

This study investigated the variation of physical and chemical properties in Yushania alpina culm with site and culm height. The *Y. alpina* culms grown at the Hagere-Selam had higher values of green moisture content (*MC*) and basic density compared to the culms collected from the Rebu-Gebeya. The green *MC* of *Y. alpina* culms decreased from the base toward the top positions for both sites. Similarly, the tangential and longitudinal shrinkage from green to oven-dry conditions of *Y. alpina* culms showed a decreasing trend from the base to the top for both sites. In contrast, the opposite tendency was observed in the basic density, which decreased from the top to the base positions of the bamboo culms for the two sites. Density is the most important parameter affecting its practical utilization. Based on density, most of the other characteristics of bamboo culms can be predicted. A significant effect of site and culm height on chemical properties was observed. The amount of cellulose and lignin content found was in the range of wood and other bamboo species. Consequently, *Y. alpina* culm can be a potential source of pulp and paper, and bioenergy, and is suitable for structural applications, such as construction and furniture-making and biobased composite production. Further study should be carried out on other potential bamboo species abundantly available in Ethiopia, such as lowland bamboo (*Oxythenatera abyssinica*) and other introduced and exotic bamboo culms.

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Prediction of Optimum Veneer Drying Parameters with Artificial Neural Networks for Production of Plywood with High Mechanical Properties

Primjena umjetnih neuronskih mreža za predviđanje optimalnih parametara sušenja furnira za proizvodnju furnirske ploče visokih mehaničkih svojstava

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ABSTRACT • *Veneer drying is the manufacturing process in the plywood industry that most affects energy consumption and panel properties such as bonding and bending. Therefore, the veneer drying temperature and moisture content should be accurately adjusted. Moreover, the determination of veneer thermal conductivity is as important as these two parameters and the thermal conductivity values should also be specified when forming the drying programs. This study aimed to predict the optimum values of the veneer drying temperatures, moisture content and thermal conductivity, which gave the best mechanical properties, by artificial neural network (ANN) analysis. Poplar (Populus deltoidesI-77/51) and spruce (Picea orientalis L.) veneers and urea formaldehyde (UF) resin were used in the production of plywood. The thermal conductivity of veneer and the bonding, bending strength and elasticity modulus of the panels were tested by the relevant standards. The most accurate and reliable prediction models were obtained by analyzing the experimental data with ANN. The optimum veneer drying temperature, moisture content and thermal conductivity values that gave the best values for all three mechanical properties were 149 °C, 6.2 % and 0.02668 W/mK for poplar and 116 °C, 4.4 % and 0.02534 W/mK for spruce.*

KEYWORDS: *veneer drying temperature; moisture content; thermal conductivity; artificial neural network; plywood*

SAŽETAK • *Sušenje furnira proizvodni je proces u industriji furnirskih ploča koji najviše utječe na potrošnju energije i svojstva ploče kao što su čvrstoća lijepljenja i savijanja. Stoga je potrebno točno prilagoditi temperaturu sušenja i sadržaj vode u furniru. Nadalje, određivanje toplinske vodljivosti furnira jednako je važno kao temperatura sušenja i sadržaj vode, pa je pri izradi programa sušenja potrebno specificirati i vrijednosti toplinske vodljivosti. Cilj ovog istraživanja bio je primjenom umjetne neuronske mreže (ANN) predvidjeti vrijednosti optimalne temperature sušenja, sadržaja vode i toplinske vodljivosti furnira koje će rezultirati najboljim mehaničkim*

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svojstvima ploče. Za potrebe ispitivanja furnirske su ploče proizvedene od furnira topolovine (Populus deltoides-I-77/51) i furnira smrekovine (Picea orientalis L.) te su slijepljeni urea formaldehidnom smolom (UF). Prema odgovarajućim standardima ispitani su toplinska vodljivost i čvrstoća lijepljenja furnira te čvrstoća na savijanje i modul elastičnosti ploča. Najtočniji i najpouzdaniji modeli predviđanja dobiveni su analizom eksperimentalnih podataka uz pomoć umjetne neuronske mreže. Optimalne vrijednosti temperature sušenja, sadržaja vode i toplinske vodljivosti uz koje se postižu najbolja mehanička svojstva za furnirske ploče od topolovine iznosile su 149 °C, 6,2 % i 0,02668 W/mK, a za furnirske ploče od smrekovine 116 °C, 4,4 % i 0,02534 W/mK.

KLJUČNE RIJEČI: *temperatura sušenja furnira; sadržaj vode; toplinska vodljivost; umjetna neuronska mreža; furnirska ploča*

1 INTRODUCTION
1. UVOD

Plywood, one of the most traditional wood-based composite materials, is widely preferred in construction, interior design and furniture (Ferretti, 2021). The global plywood production market, which totaled $88.3 billion in 2017, is predicted to have a compound annual growth rate (CAGR) of 7.8 % between 2017 and 2022, and this market will reach $128.3 billion by 2022 (BCC Research, 2018). In the plywood industry, which has a very large market, it is extremely important to improve the production processes and integrate new technologies into the systems. Veneer drying process is the most important production process of veneer-based composite materials such as plywood (Guan *et al*., 2020). The efficiency of the veneer drying process is an issue that requires great attention both in terms of energy consumption and for improving the adhesion properties of the panels (Huang *et al*., 2011). The problems related to this process, in which the veneer is dried to the most suitable moisture content for adhesion, directly affect the adhesive properties such as curing and physical and mechanical properties of the panel (Bekhta *et al*., 2020). Moreover, the potential to cause an increase in both adhesive consumption and pressing and drying costs proves the economic importance of this process (Bekhta *et al*., 2014). It is a fact that the environment can be harmed in terms of global warming due to improper drying process, material properties and economic losses, as well as unnecessary energy consumption. Veneer drying in plywood production is one of the processes that consumes the most energy and 70 % of the total thermal energy consumption is due to this process (Han *et al*., 2015).

It is extremely important to set two parameters correctly in the veneer drying process. These parameters are the veneer moisture content and drying temperature. Aydin and Colakoglu (2005) stated that the veneer moisture content should not be above 7 % and that the veneers were dried up to 3 % moisture content. Furthermore, Bekhta *et al*. (2014) mentioned that, if the veneer moisture content is above (8±2) %, it can cause great disadvantages. According to FAO (1990), veneer drying temperatures between 90-160 °C were accepted as normal and it was stated that temperatures could be increased by about 175 °C for high temperature drying applications to shorten the drying time. High temperature drying can save up to 44 % of energy and reduce drying time by 25 % compared to normal drying (Theppaya and Prasertsan, 2004). In addition to these advantages, drying at high temperatures can adversely affect the surface, and thermal and chemical properties of veneers (Demirkir *et al*., 2016).

In the literature, there are few studies investigating the effects of the veneer moisture content (Aydin *et al*., 2006; Bekhta *et al*., 2020) and drying temperatures (Aydin and Colakoglu, 2005) on the technological properties of plywood panels. In addition to these studies in which experimental data were compared, there were some studies in which optimum results were achieved without further experimentation with the prediction models obtained successfully with artificial neural network (ANN) analysis. These optimization studies were mostly carried out on the veneer drying temperature, and optimum values were obtained for different wood species, giving the best mechanical properties (Demirkir *et al*., 2013; Ozsahin *et al*., 2019; Ozsahin and Aydin, 2014). In contrast, Demir and Aydin (2021) determined the effect of the veneer moisture content on the thermal properties of veneers with ANN prediction model.

In order to set the veneer drying programs correctly, the moisture content, drying temperature as well as the thermal conductivity of the veneer should be selected in the most appropriate way. It is known that the thermal conductivity is an important parameter used in developing drying models, determining the adhesive curing and heat transfer rate (Hassanin *et al*., 2018). Based on this, the lack of comprehensive optimization studies evaluating the veneer moisture content, drying temperatures and thermal conductivity parameters was seen in the literature. Therefore, this study aimed to predict the optimum values of the veneer moisture content, drying temperatures and thermal conductivity coefficients by ANN analysis for achieving the best mechanical strength properties.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials and plywood manufacture
2.1. Materijali i proizvodnja furnirskih ploča

In this experimental study, poplar (*Populus deltoidesI-77/51*) and spruce *(Picea orientalis L.*) veneers were used in the manufacture of plywood. While the poplar logs were peeled freshly, spruce logs were steamed for 12-16 hours before veneer production. After the peeling process, the veneers with 500 mm × 500 mm × 2 mm dimensions were obtained from the logs. The veneers were dried at 110, 130, 160, and 180 °C until 4 %, 6 % and 8 % moisture content. The veneers moisture content was continuously controlled with a magnetic moisture meter until it reached the specified moisture content levels. Three-layer-plywood panels, 6 mm thick, were manufactured by using urea formaldehyde (UF) with 55 % solid content. UF resin solution used in the plywood manufacturing was composed of 100 parts UF resin, 30 parts wheat flour and 10 parts NH_4Cl (with15 % concentration) as hardener, by weight. The glue was applied at a rate of 160 g/m^2 to every single veneer layer. The panel drafts belonging to the groups formed within the scope of the study were pressed by adjusting the press pressure to 8 kg/cm^2, the press temperature to 110 ºC and the press time to 6 minutes. Two panels produced from each group were kept in the climatization room at 20 °C and 65% relative humidity before the experiments.

The produced plywood panels bonding strength, bending strength and elasticity modulus were determined according to EN 314-1 (2004) and EN 310 (1993) standards, respectively. The samples manufactured with UF resin were tested after immersion in water at 20 °C for 24 h. Twenty-five and twelve specimens were used for the evaluation of bonding strength and bending strength tests, respectively. The thermal conductivity coefficients of the poplar and spruce veneers were measured in five replicates by the ASTM C 518 (2004) standard. The Lasercomp Fox-314 Heat Flow Meter was used for the determination of thermal conductivity. Its top and lower layers were set to 20°C and 40°C, respectively, for all specimens. The panels temperature during the measurement of the thermal conductivity was maintained to these constant temperatures.

2.2 ANN analysis
2.2. ANN analiza

In this study, Artificial Neural Network (ANN) analysis of the MATLAB Toolbox was used to determine the optimum veneer drying parameters. The experimentally obtained values of thermal conductivity, bonding, bending strength and elasticity modulus were used in the training phase and these values were predicted for the veneer drying parameters that were not used experimentally. First, the thermal conductivity model was determined from the prediction models, and then the prediction models for the mechanical properties were determined. The first prediction model used wood species, veneer drying temperature and veneer moisture content as the input variables, while other models additionally included thermal conductivity in input variables. The network structures of the models obtained from the training phase within the scope of this study are shown in Figure 1.

During the analysis, experimental data were divided into two groups as training and testing data. Training data, which contains approximately 67 % of the total data, was used in the development of prediction models, while the remaining data (approximately 33 %) was used to test the performance of the obtained models. The adjustments used in ANN trainings are given in Table 1.

At the end of the ANN analysis, some diagnostic tools were used to reveal the statistical relationship between the actual data and the predicted data in both training and testing data sets. These are the mean absolute percent error (*MAPE*), the root mean square error (*RMSE*) and coefficient of determination (R^2). The optimum veneer drying parameters were predicted by using ANN models determined using these tools.

Table 1 Adjustments used in ANN trainings
Tablica 1. Prilagodbe koje se primjenjuju u treninzima ANN-a

Type of ANN *Vrsta ANN-a*	Feed forward and backpropagation multilayer *višeslojno širenje naprijed i natrag*
Transfer function (hidden layers) *Funkcija prijenosa (skriveni slojevi)*	Hyperbolic tangent sigmoid function (tansig) *hiperbolična tangentna sigmoidna funkcija (tansig)*
Transfer function (output layers) *Funkcija prijenosa (izlazni slojevi)*	Linear transfer function (purelin) *linearna prijenosna funkcija (purelin)*
Training algorithm *Algoritam treninga*	Levenberg-Marquardt algorithm (trainlm) *Levenberg-Marquardtov algoritam (trainlm)*
Learning rule *Pravilo učenja*	Momentum gradient reduction backpropagation algorithm (traingdm) *algoritam povratnog širenja smanjenja gradijenta momenta (traingdm)*
Performance function *Funkcija izvedbe*	Mean square error (MSE) *srednja kvadratna pogreška (MSE)*

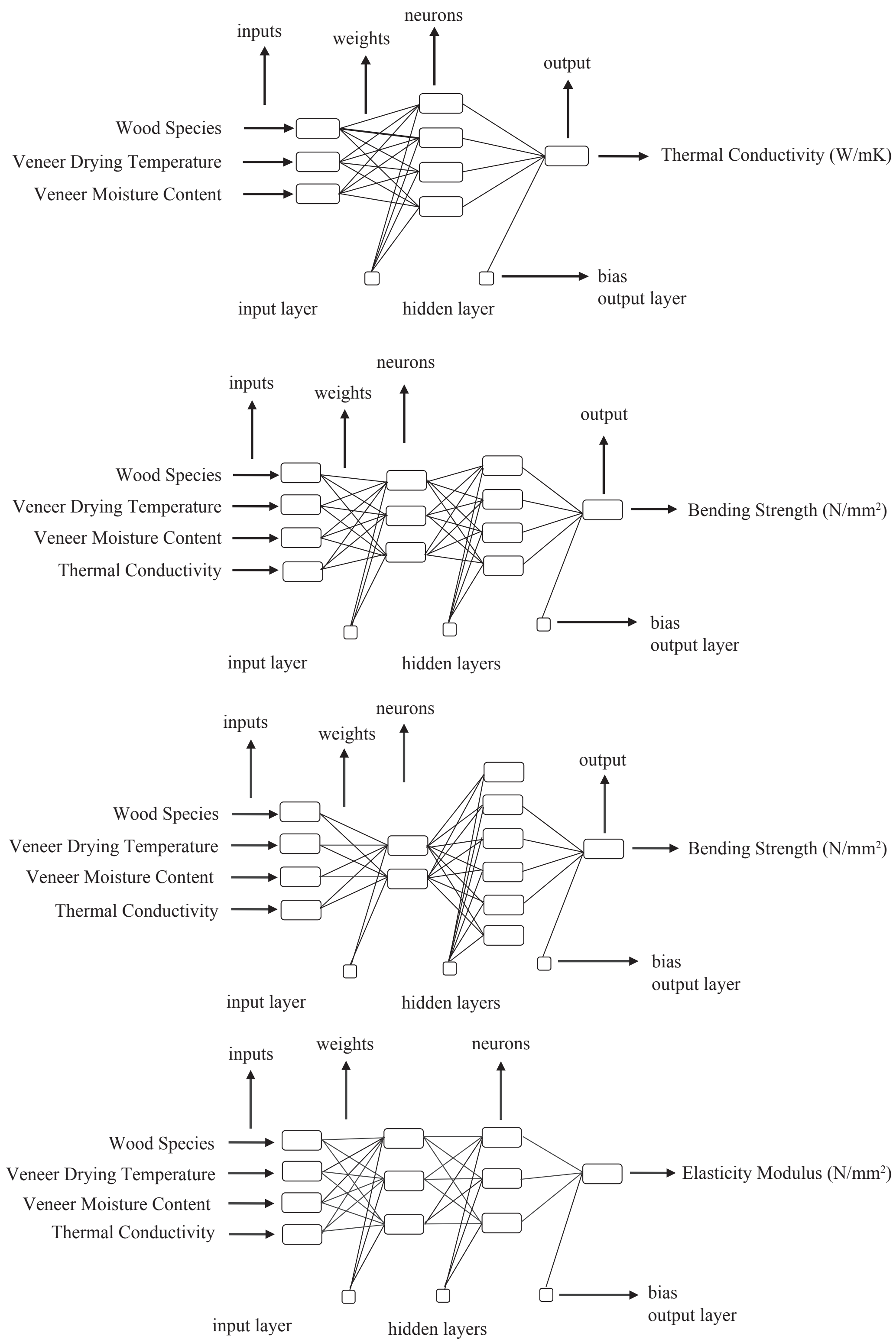

Figure 1 Structures of prediction models
Slika 1. Strukture modela predviđanja

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

The predicted data with the experimental (actual) data are given in Tables 2 and 3 according to training and testing data sets, as well as the performance values of the prediction models. In the experimental results, it was observed that the veneer thermal conductivity values increased due to the increase in the veneer moisture content and the highest thermal conductivity values were obtained at 180 °C. The bonding values of the panels decreased, while the veneer drying temperature and moisture content values increased in both wood species. Furthermore, it was observed that the bending strength and elasticity modulus values of the plywood produced from poplar veneers were almost not affected

Table 2 Training data set used for thermal conductivity and mechanical strength and prediction model results
Tablica 2. Primijenjeni skup podataka za trening za toplinsku vodljivost i mehaničku *čvrstoću* te rezultati modela predviđanja

Wood species *Vrsta drva*	Veneer drying temperature, °C *Temperatura sušenja furnira,* °C	Veneer moisture content, % *Sadržaj vode u furniru*	Thermal conductivity, W/mK *Toplinska vodljivost,* W/mK			Bonding strength, N/mm² *Čvrstoća lijepljenog spoja,* N/mm²			Bending strength, N/mm² *Čvrstoća na savijanje,* N/mm²			Elasticity modulus, N/mm² *Modul elastičnosti,* N/mm²		
			Actual *Stvarna*	**Predicted** *Predviđena*	**Error, %** *Pogreška,* %	**Actual** *Stvarna*	**Predicted** *Predviđena*	**Error, %** *Pogreška,* %	**Actual** *Stvarna*	**Predicted** *Predviđena*	**Error, %** *Pogreška,* %	**Actual** *Stvarna*	**Predicted** *Predviđena*	**Error, %** *Pogreška,* %
Poplar *topolovina*	110	6	0.03034 *0.00009*	0.03022	0.41060	1.49 *0.13*	1.49	0.00	74.38 *7.89*	74.33	0.06	4316.39 *422.98*	4288.54	0.65
	110	8	0.03467 *0.00011*	0.03458	0.25742	1.87 *0.21*	1.87	0.07	83.43 *4.12*	83.26	0.20	5086.50 *337.70*	5084.56	0.04
	130	4	0.02712 *0.00008*	0.02714	-0.07575	1.47 *0.16*	1.47	-0.03	82.99 *7.76*	83.23	-0.28	3968.30 *486.03*	3979.95	-0.29
	130	8	0.03153 *0.00007*	0.03151	0.07664	1.34 *0,1*	1.34	0.06	79.06 *6.01*	79.17	-0.14	4865.91 *360.78*	4863.32	0.05
	160	6	0.02727 *0.00012*	0.02734	-0.25999	1.76 *0.27*	1.76	0.05	88.13 *6.99*	87.56	0.65	5145.32 *398.63*	5136.56	0.17
	160	8	0.03309 *0.00009*	0.03305	0.12185	1.33 *0.16*	1.33	-0.21	72.13 *6.66*	72.21	-0.12	4297.63 *490.04*	4311.25	-0.32
	180	4	0.03019 *0.00011*	0.03015	0.11737	1.47 *0.28*	1.47	0.05	78.05 *6.72*	77.83	0.28	4635.93 *294.48*	4612.14	0.51
	180	6	0.03108 *0.00008*	0.03108	0.00080	1.24 *0.23*	1.24	-0.06	85.23 *5.72*	84.85	0.44	4844.19 *214.08*	4857.71	-0.28
Spruce *smrekovina*	110	4	0.02488 *0.00005*	0.02479	0.37682	1.39 *0.23*	1.39	-0.11	74.52 *4.77*	74.66	-0.19	4485.44 *278.20*	4473.82	0.26
	110	8	0.02877 *0.00008*	0.02861	0.57112	1.29 *0,2*	1.28	0.84	64.38 *7.24*	64.38	0.00	4533.12 *217.17*	4573.67	-0.89
	130	4	0.02540 *0.00007*	0.02533	0.27914	1.34 *0.17*	1.34	-0.01	66.82 *7.92*	66.82	-0.01	4389.64 *323.56*	4434.17	-1.01
	130	6	0.02653 *0.00012*	0.02656	-0.09443	1.26 *0.16*	1.27	-0.74	60.03 *2.97*	60.07	-0.07	3057.93 *142.31*	3095.54	-1.23
	160	4	0.02578 *0.00006*	0.02583	-0.18449	1.36 *0.21*	1.36	-0.11	59.67 *5.14*	59.52	0.25	4550.55 *317.18*	4549.59	0.02
	160	8	0.02667 *0.00010*	0.02653	0.53174	1.24 *0.25*	1.26	-1.51	54.20 *3.97*	54.57	-0.69	3673.39 *180.02*	3701.88	-0.78
	180	6	0.02809 *0.00006*	0.02808	0.03055	1.27 *0.1*	1.26	0.84	62.88 *5.75*	63.04	-0.25	4234.94 *180.92*	4230.18	0.11
	180	8	0.02996 *0.00007*	0.03000	-0.11814	1.26 *0.19*	1.26	0.36	60.93 *3.47*	60.72	0.35	4916.41 *383.34*	4926.92	-0.21

Standard deviation values are shown in italic. / *Vrijednosti standardne devijacije otisnute su kurzivom.*

Table 3 Testing data set used for thermal conductivity and mechanical strength and prediction model results
Tablica 3. Primijenjeni testni skup podataka za toplinsku vodljivost i mehaničku *čvrstoću* te rezultati modela predviđanja

Wood species *Vrsta drva*	**Veneer drying temperature, °C** *Temperatura sušenja furnira,* °C	**Veneer moisture content, %** *Sadržaj vode u furniru*	**Thermal conductivity, W/mK** *Toplinska vodljivost,* W/mK			**Bonding strength, N/mm²** *Čvrstoća lijepljenog spoja,* N/mm²			Bending strength, N/mm² *Čvrstoća na savijanje,* N/mm²			**Elasticity modulus, N/mm²** *Modul elastičnosti,* N/mm²		
			Actual *Stvarna*	**Predicted** *Predviđena*	**Error, %** *Pogreška,* %	**Actual** *Stvarna*	**Predicted** *Predviđena*	**Error, %** *Pogreška,* %	**Actual** *Stvarna*	Predicted *Predviđena*	**Error, %** *Pogreška,* %	**Actual** *Stvarna*	**Predicted** *Predviđena*	**Error, %** *Pogreška,* %
Poplar *topolovina*	110	4	0.02981 *0.00009*	0.02967	0.45838	1.91 *0.26*	1.93	-0.82	87.27 *3.02*	86.37	1.03	4757.80 *440.41*	4601.75	3.28
	130	6	0.02832 *0.00007*	0.02738	3.31151	1.68 *0.23*	1.66	0.93	84.28 *5.09*	83.83	0.53	4681.47 *458.20*	4712.40	-0.66
	160	4	0.02630 *0.00005*	0.02645	-0.58552	1.46 *0.26*	1.38	5.60	81.71 *3.41*	80.20	1.85	4738.93 *315.28*	4733.70	0.11
	180	8	0.03534 *0.00012*	0.03528	0.16519	1.31 *0.18*	1.31	0.08	77.48 *6.82*	77.15	0.42	4771.69 *437.27*	4218.57	11.59
Spruce *smrekovina*	110	6	0.02660 *0.00005*	0.02674	-0.52975	1.38 *0.1*	1.31	4.94	67.33 *7.74*	66.28	1.56	4752.53 *294.21*	4667.60	1.79
	130	8	0.02747 *0.00006*	0.02764	-0.62730	1.23 *0.2*	1.26	-2.62	58.28 *3.67*	58.44	-0.28	3301.51 *139.74*	3121.57	5.45
	160	6	0.02660 *0.00007*	0.02612	1.81271	1.27 *0.09*	1.26	0.61	56.13 *4.79*	55.34	1.41	3925.54 *395.84*	3949.79	-0.62
	180	4	0.02705 *0.00006*	0.02661	1.61564	1.34 *0.2*	1.33	0.72	67.45 *3.50*	66.76	1.03	4662.68 *330.25*	4689.71	-0.58

Standard deviation values are shown in italic. / *Vrijednosti standardne devijacije otisnute su kurzivom.*

Table 4 Performance values of the best prediction models
Tablica 4. Vrijednosti svojstava najboljih modela predviđanja

Prediction models *Modeli predviđanja*	***MSE***	R^2	Data sets	MAPE	RMSE
Thermal conductivity *toplinska vodljivost*	0.00022287	0.9931	Training / *treniranje*	0.22%	0.0001
			Testing / *testiranje*	1.14%	0.0004
Bonding strength *čvrstoća lijepljenog spoja*	0.00038275	0.9856	Training / *treniranje*	0.32%	0.01
			Testing / *testiranje*	2.04%	0.04
Bending strength *čvrstoća na savijanje*	0.00018906	0.9983	Training / *treniranje*	0.25%	0.23
			Testing / *testiranje*	1.01%	0.84
Elasticity modulus *modul elastičnosti*	0.00046219	0.9459	Training / *treniranje*	0.43%	22.44
			Testing / *testiranje*	3.01%	215.69

by the increase in the veneer drying temperature and moisture content.

The best training performances and MSE values were realized as 0.00022287 in the 35th iteration for the thermal conductivity, 0.00038275 in the 19th iteration for the bonding strength, 0.00018906 in the 25th iteration for the bending strength and 0.00046219 in the 28th iteration for the elasticity modulus. The performance values of the best prediction models obtained by ANN analysis are given in Table 4.

MAPE, *RMSE* and R^2 parameters are mostly used to evaluate the performance of prediction models obtained by ANN (Yadav and Nath, 2017; Kucukonder *et al*., 2016). Yadav and Nath (2017) determined that the prediction performance of models with *MAPE* values below 10 % was quite good. Similarly, some researchers determined that the prediction abilities of the models were successful if the *RMSE* values were quite low and the R^2 values were close to 1 (Taspınar and Bozkurt, 2014; Ozsahin, 2012). In this study, the *MAPE* values for the thermal conductivity, bonding, bending strength and elasticity modulus were 0.22 %, 0.32 %, 0.25 %, 0.43 % for training and 1.14 %, 2.04 %, 1.01 %, 3.01 % for testing, respectively. The *RMSE* values were 0.0001, 0.01, 0.23, 22.44 for training and 0.0004, 0.04, 0.84, 215.69 for testing, respectively. Furthermore, the R^2 values were calculated as 0.9931, 0.9856, 0.9983 and 0.9459, respectively. The calculated values of these diagnostic tools proved the reliability and precision of the prediction models obtained from ANN analysis.

Thanks to the reliable and predictable ANN models, the output values corresponding to the intermediate input variables that are not used in the experiments can be predicted with high precision. In this study, high-precision predictions were made with the help of models for the veneer drying temperatures and moisture content, which were not used in the experiments. The effects of input variables on output variables are shown in Figures 2 and 3 according to wood species.

According to the data obtained from the ANN prediction models in Figures 2 and 3, it was determined that the veneer drying temperature and the veneer moisture content had a significant effect on the thermal conductivity, bonding strength, bending strength and elasticity modulus and these effects differed according to the wood species. In particular, it was clearly observed that the thermal conductivity coefficients increased depending on the increase in the veneer moisture content. The reason for this linear relationship could be shown as the fact that the water molecules, which increased with the increase in moisture, were more conductive than the air. According to the ISO 10456 (2007) standard, the thermal conductivity value (0.060 W/mK) of the water molecule was determined to be higher than the thermal conductivity value (0.025 W/mK) of the air. Sonderegger and Niemz (2009) worked on the effect of moisture content on the thermal conductivity of beech plywood and observed that the thermal conductivity values increased with the increase in moisture content. A similar relationship between the moisture content and thermal conductivity was also found in some studies on solid wood and wood-based panels in the literature (Taoukil *et al*., 2013; Troppová *et al*., 2015). Contrary to the veneer moisture content, nonlinear relationships were observed between the veneer drying temperature and thermal conductivity. However, the veneer drying temperature value, which gave the highest thermal conductivity values among the groups, was 180 °C in both wood species. Sonderegger and Niemz (2009) mentioned that the thermal conductivity values of wood materials increased depending on the increase in temperature.

It was determined that the bonding strength values obtained from the experiments and the predicted values were above the 1 N/mm² limit value specified in the EN 314-2 (1993) standard. The bonding values of the panels generally decreased, while the values of the veneer drying temperature and moisture content increased in both wood species. Aydin and Colakoglu (2005) stated that the veneer drying process at high temperatures could negatively affect the wettability of the wood surface with the adhesive, thus reducing the bonding strength. Similarly, Bekhta *et al*. (2020) mentioned that prolonged drying at a very high temperature could render the veneer surface ineffective, resulting in poor wetting of the veneer and thus poor adhesion. Furthermore, Aydin *et al*. (2006) investigated the effects of

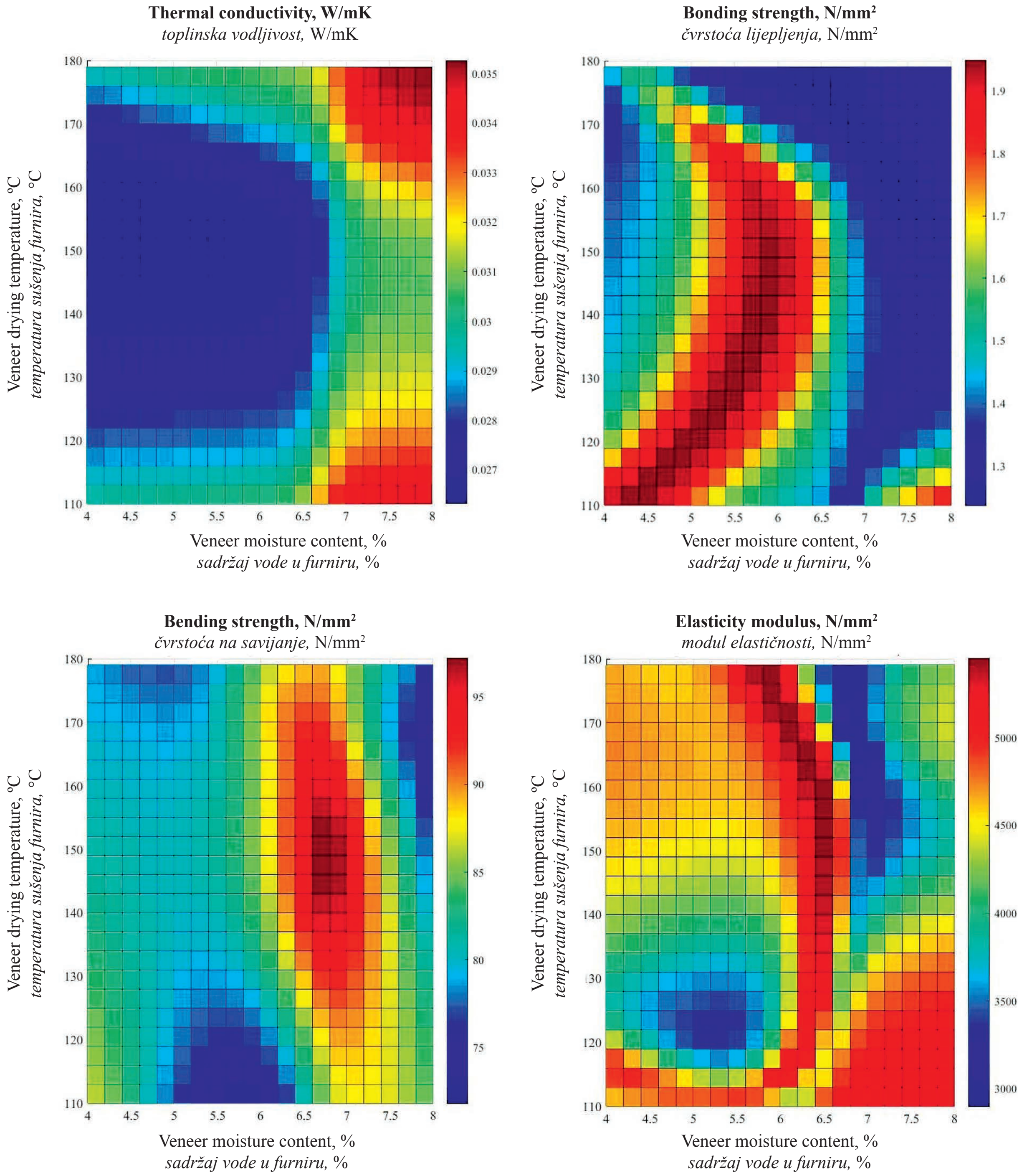

Figure 2 Effects of veneer drying temperature and veneer moisture content on thermal conductivity and mechanical strength values of poplar plywood panels
Slika 2. Učinci temperature sušenja i sadržaja vode u furniru na vrijednost toplinske vodljivosti i mehaničke čvrstoće furnirske ploče od topolovine

moisture content on the mechanical properties of plywood and found that the mechanical properties decreased with increasing veneer moisture content. However, the bending strength and elasticity modulus values of the plywood produced from poplar veneers were almost not affected by the increase in the veneer drying temperature and moisture content. In contrary, the highest bending strength and elasticity modulus values in the spruce plywood panels were obtained from the lowest values of veneer drying temperatures and moisture content.

By using the prediction models, optimum veneer drying temperature and moisture content values for wood species were determined and they are given in Table 5 according to output variables.

The veneer drying temperature values that gave the best mechanical properties of the plywood produced from poplar and spruce veneers are given in Ta-

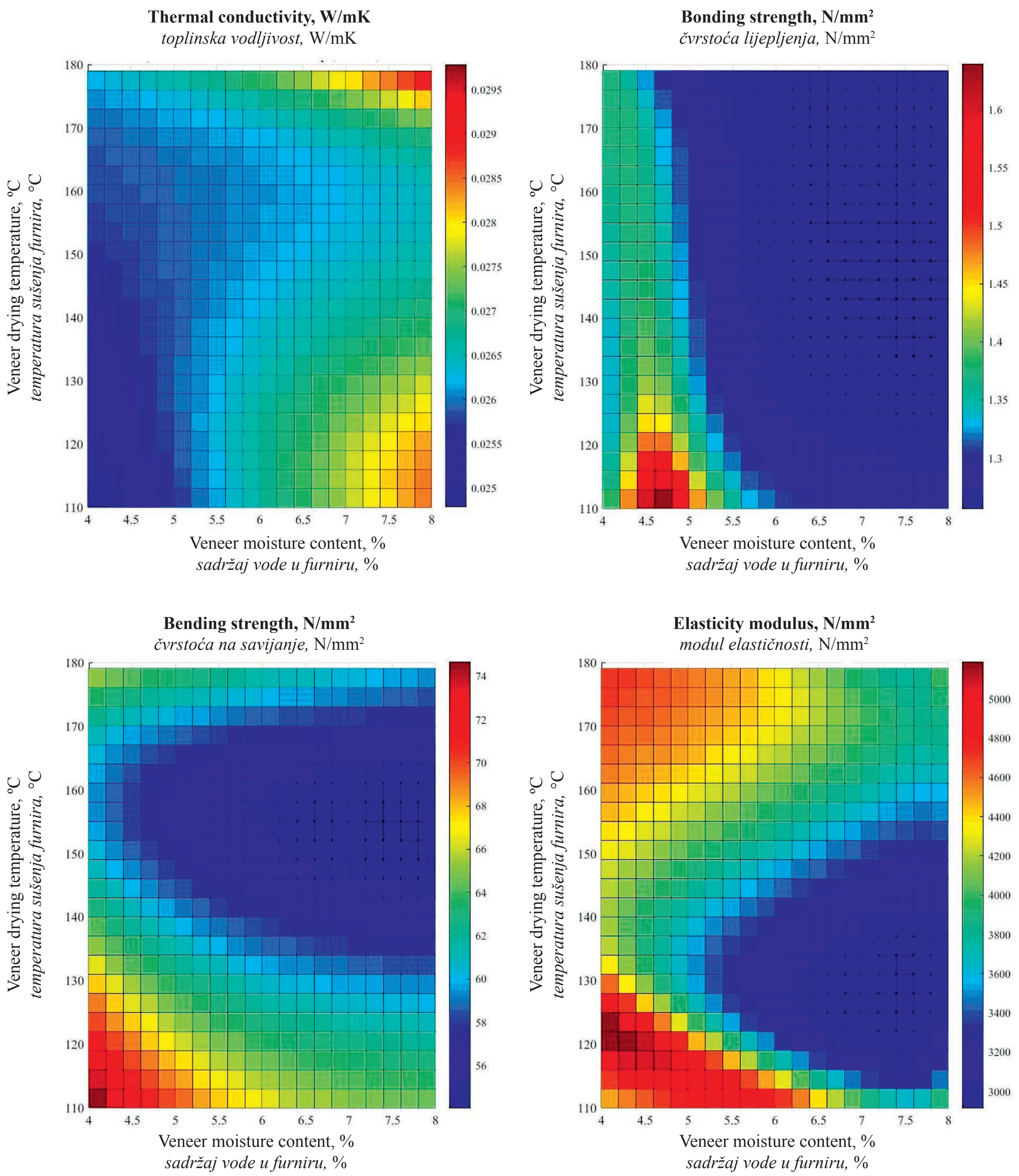

Figure 3 Effects of veneer drying temperature and veneer moisture content on thermal conductivity and mechanical strength values of spruce plywood panels
Slika 3. Učinci temperature sušenja i sadržaja vode u furniru na vrijednost toplinske vodljivosti i mehaničke čvrstoće furnirske ploče od smrekovine

ble 5. ANN optimization studies that could be used in veneer drying processes for the plywood industry were limited in the literature. Although the optimum values giving the best mechanical strength properties were determined for different wood species in a few studies on the veneer drying temperature, no optimization studies were found regarding the veneer drying moisture. Demirkir *et al*. (2013) worked on some manufacturing parameters such as the effect of veneer drying temperature on the bonding strength of the plywood produced from Scots pine, maritime pine and European black pine veneer and determined the veneer drying temperature that provides the best bonding strength with ANN. They found optimum values of the plywood produced with melamine urea formaldehyde (MUF) resin in the range of 110-125 ºC for the Scots pine veneers, 110-124 ºC for the maritime pine veneers and 110-112 ºC for the European black pine veneers (Demirkir *et al*.,

Table 5 Optimum veneer drying temperature, veneer moisture content and thermal conductivity results
Tablica 5. Rezultati optimalne temperature sušenja, sadržaja vode i toplinske vodljivosti furnira

Mechanical properties *Mehanička svojstva*	**Wood species** *Vrsta drva*	**Optimum values** / *Optimalna svojstva*			**Maximum strength values, N/mm²** *Najveće vrijednosti čvrstoće,* N/mm²
		Veneer drying temperature, °C *Temperatura sušenja furnira,* °C	**Veneer moisture content, %** *Sadržaj vode u furniru,* %	**Thermal conductivity, W/mK** *Toplinska vodljivost,* W/mK	
Bonding strength *čvrstoća lijepljenog spoja*	Poplar *topolovina*	128	5.4	0.02744	1.95
	Spruce *smrekovina*	110	4.6	0.02538	1.64
Bending strength *čvrstoća na savijanje*	Poplar topolovina	146	6.6	0.02791	97.23
	Spruce *smrekovina*	110	4	0.02479	74.67
Elasticity modulus *modul elastičnosti*	Poplar *topolovina*	164	6.2	0.02842	5454.98
	Spruce *smrekovina*	119	4	0.02505	5189.42

2013). In this study, the optimum results for spruce veneers, which was one of the coniferous wood species, were determined and the optimum veneer temperature value for bonding strength was determined as 110 °C. This result was found to be similar to the results found in the literature. Ozsahin *et al*. (2019) determined the optimum drying temperatures of alder and Scots pine veneers according to the results of mechanical properties and found that the highest values of bonding, bending strength and elasticity modulus were obtained from 190, 195 and 196°C in the alder veneers and from 165, 162 and 161°C in the Scots pine veneer, respectively. Moreover, Ozsahin and Aydin (2014) determined by the ANN analysis the optimum drying temperature values of 169 °C for UF and 125 °C for FF in beech veneers, 162 °C for UF and 151 °C for FF in spruce veneers. Similarly, the poplar veneers, which was one of the hardwood species, had higher optimum veneer temperature than spruce veneer, which was one of the coniferous wood species in this study. It can be seen in Table 5 that the optimum moisture content values of poplar veneers were higher than those of spruce veneers.

In addition to the veneer drying temperatures and the optimum veneer moisture content, the veneer thermal conductivity coefficients are given in Table 5. Figures 2 and 3 also show that the values of thermal conductivity coefficient increased with the increase of the moisture content of poplar and spruce veneers. Demir and Aydin (2021) obtained optimal results by modeling the experimental results of thermal conductivity with ANN according to the moisture content changes of beech, Scots pine and poplar veneer between 3 % and 15 % and determined that the veneers with high moisture content had high values of thermal conductivity.

According to the predicted results, the optimum values of veneer drying temperatures, moisture content and thermal conductivity of panels, which gave the best values for all three mechanical properties, were 149 °C, 6.2 % and 0.02668 W/mK for poplar and 116 °C, 4.4 % and 0.02534 W/mK for spruce.

4 CONCLUSIONS
4. ZAKLJUČAK

In this study, the optimum values of veneer drying temperature, moisture content and thermal conductivity, which gave the best mechanical properties of plywood panels, were determined by ANN analysis. While the experimental data were analyzed by ANN, the drying temperature and moisture content were used as input variables and the veneer thermal conductivity prediction model was first obtained. Then, the experimental thermal conductivity values were added in addition to the two input variables and the prediction models of mechanical properties were obtained. The accuracy and reliability of the ANN models were proven by the performance functions. In the testing phase, the *MAPE* values in the prediction models of the thermal conductivity, bonding, bending strength and elasticity modulus were 1.14 %, 2.04 %, 1.01 %, 3.01 %, whilst the *RMSE* values were 0.0004, 0.04, 0.84, 215.69, respectively. The R^2 values were 0.9931, 0.9856, 0.9983 and 0.9459.

The intermediate values that were not used in the experiments between 110 °C -180 °C veneer drying temperatures and 4 % - 8 % moisture content levels were predicted with high accuracy with the help of these models. Figures 2 and 3 show the changes in thermal conductivity and mechanical properties with these intermediate values. Furthermore, the optimum drying temperature, moisture content and thermal conductivity values that gave the best mechanical properties are presented in Table 5. The optimum veneer drying temperature, moisture content and thermal conductivity values that gave the highest bonding strength values were 128 °C, 5.4 % and 0.02744 W/mK for poplar, and 110 °C, 4.6 % and 0.02538 W/mK for spruce, respectively. The values for the bending strength were 146°C, 6.6% and 0.02791 W/mK for poplar, and 110 °C, 4 % and 0.02479 W/mK for spruce, respectively. These values for the elasticity modulus were 164 °C, 6.2 % and 0.02842 W/mK for poplar, and 119 °C, 4 % and 0.02505 W/mK for spruce, respectively. By using both the graphs and the table of optimum values, the mechanical properties of the plywood to be produced with poplar and spruce veneers can be predicted without further experimentation. It is thought that the findings of this study will be an important reference for veneer drying programs, which are extremely important for the plywood industry. If different wood species and adhesives are used, the precision of the prediction models in this study will be low and this is seen as the weakness of the study. Therefore, the optimization predictions can be carried out using different wood species of veneers and adhesives in future studies.

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Triboelectric and Hydrophobic Characterization of Functionalized Lignocellulosic Materials

Triboelektrična i hidrofobna svojstva funkcionaliziranih lignoceluloznih materijala

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ABSTRACT • *In the development of sustainable products, lignocellulosic materials with hydrophobic properties can be functionalized and used as reinforcement, especially in bio-composite materials, as well as in various applications such as packaging, water-repellent and self-renewing materials. This study is aimed to improve the surface properties and triboelectric properties of wood materials. Functionalized wood veneers were prepared by impregnating 3 different wood veneers (beech, mahogany and oak) with 5 different chemical solutions (cationic cellulose, cationic starch, polyethyleneimine, sodium alginate and carboxymethyl cellulose). Structural characterization of the functional wood materials obtained was investigated by Fourier-transform infrared spectroscopy (FT-IR) technique, wettability and surface properties were examined by contact angle measurements, and morphological properties were examined by scanning electron microscopy (SEM). The triboelectric properties of the devices prepared using functionalized wood materials were investigated. As a result, it was determined that the hydrophobic properties of wood materials were improved and showed triboelectric properties. It demonstrates that functionalized wood materials can be used to power low-power electronic devices.*

KEYWORDS: *lignocellulosic materials; triboelectric properties; surface properties*

SAŽETAK • *U razvoju održivih proizvoda lignocelulozni materijali hidrofobnih svojstava mogu se funkcionalizirati i upotrebljavati kao ojačivači u biokompozitnim materijalima ili mogu imati različitu primjenu u ambalaži te u vodoodbojnim i samoobnavljajućim materijalima. Cilj ovog istraživanja bio je pronaći način poboljšanja površinskih i triboelektričnih svojstava drvnih materijala. Za istraživanje je pripremljen funkcionalizirani furnir od tri različite vrste drva (bukovine, mahagonija i hrastovine), impregniran s pet različitih kemijskih otopina (kationskom celulozom, kationskim škrobom, polietileniminom, natrijevim alginatom i karboksimetilcelulozom). Karakterizacija strukture dobivenih funkcionaliziranih drvnih materijala provedena je Furierovom infracrvenom spektroskopijom (FT-IR), kvašenje i svojstva površine istražena su mjerenjem kontaktnog kuta, a morfološka svojstva ispitana su skenirajućim elektronskim mikroskopom (SEM). Nadalje, istražena su triboelektrična svojstva uređaja izrađenih od funkcionaliziranih drvnih materijala. Utvrđeno je da se funkcionalizirani drvni materijali mogu primijeniti za napajanje uređaja male snage.*

KLJUČNE RIJEČI: *lignocelulozni materijali; triboelektrična svojstva; svojstva površine*

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1 INTRODUCTION
1. UVOD

Wood is used in many applications due to its excellent mechanical properties, relative abundance and being a renewable resource. Cellulose, hemicellulose, and lignin are the main components of wood. There is a distinct color difference between carbohydrates and phenolics. Lignin, a phenolic compound, is much darker in color than cellulose and hemicellulose (Zhu *et al.,* 2016). Wooden surfaces in real use are susceptible to being affected by external factors that change their technological properties. The wood material is hydrophilic, and its hygroscopicity and dimensional stability change after the water binds to the wood surface, causing some defects in the wood material (Engelund *et al.,* 2013; Rautkari *et al.,* 2013; Keplinger *et al.,* 2015). Lignocellulosic materials with hydrophobic properties are of great interest to develop sustainable products that can be used in various applications such as packaging, water-repellent, and self-cleaning materials, oil and water separation, or as supplements in bio-composite materials. The hydroxyl functional groups present in cellulose provide the possibility to perform various chemical modifications to cellulosic substrates that can increase their hydrophobicity (Rodríguez-Fabià *et al.,* 2022).

Wood is a biodegradable material. It is environmentally friendly, renewable, abundant and inexpensive. Wood can be transformed into flexible wood-based micro or nanomaterials after being chemically processed. Its porous structure and unique properties (high mechanical properties, high surface area, high aspect ratio, and low density) pave the way for the use of wood and wood-derived materials in triboelectric nanogenerators (TENGs) (Chen *et al.,* 2018. TENGs display many advantageous features such as being light and miniaturized, not adversely affecting aesthetic and comfort properties, and having a wide range of materials. In recent years, many studies have been carried out on TENGs due to their low weight, small size, flexibility, many material options, useful design structures and superior performance at low frequencies (Chen *et al.,* 2018; Wang, 2022); Sun *et al.,* (2021) fabricated and characterized TENG devices using triboelectrically negative polydimethylsiloxane and triboelectrically positive zeolitic imidazolate framework-8. They reported that the devices they designed could be used to power smart buildings, household lamps, calculators, and electronic windows. In another study, Wang *et al.,* (2022) fabricated TENGs from lignin-based nanofibers produced using the electro-spinning technique. They emphasized that the effect of these lignin-based high performance tribopositive materials is important in sustainable energy.

The aim of this study is to improve the triboelectric behavior and hydrophobic properties of wood coatings. Functional wood veneers were obtained by impregnating 3 wood veneer types (beech, mahogany and oak) with 5 different chemicals (cationic cellulose, cationic starch, polyethyleneimine, sodium alginate and carboxymethyl cellulose). The surface properties and triboelectric properties of the obtained functional wood veneers were investigated, and their chemical structure, surface properties and morphology were also characterized.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Material
2.1. Materijal

Oak, Beech and Mahogany wood veneers (0.75 mm thickness) were supplied from Rasmus Farschs Vej (Denmark) company. Corn starch (unmodified regular corn starch containing approximately 73 % amylopectin and 27 % amylose), hydroxyethyl cellulose (average Mv ~90000), glycidyl trimethylammonium chloride (GTAC), sodium alginate, sodium hydroxide (NaOH), isopropyl alcohol, poly(ethyleneimine) solution, carboxymethylcellulose sodium salt were purchased from Sigma Aldrich. Ethanol was purchased from Merck. All reagents were used without further purification.

2.2 Preparation of solutions
2.2. Priprema otopina

Five different coating solutions were used to impregnate the wood. First, cationic starch (CS) and cationic cellulose (CC) were synthesized and characterized according to previous methods in the literature.

5 g of corn starch, 2.5 g of GTAC, 1.5 mL of 1 mol/L NaOH solution and 1.625 g of distilled water were added to a flask. The flask was incubated in a 60 °C water bath for 5 hours. At the end of this time interval, the reaction was stopped by adding 100 mL of ethanol to the flask and CS was precipitated. The precipitated CS was filtered in vacuum and washed with ethanol. CS was dried in an oven at 50 °C for 6 hours and milled. CS was obtained as a white powder (Şen *et al.,* 2017). CC was synthesized by the same method as CS. Isopropanol was used as the solvent instead of distilled water (Şen and Kahraman, 2018).

20 % aqueous solutions of 4 CS and CC, 10 % aqueous solutions of sodium alginate (SA) and carboxymethyl cellulose (CMC), 25 % aqueous solutions of polyethyleneimine (PEI) were prepared.

2.3 Applying solutions to samples
2.3. Nanošenje otopina na uzorke

Beech (B), Mahogany (M), and Oak (O) veneers were cut in 7 cm × 3.5 cm dimensions. 20 ml of 5 different solutions prepared for each coating type was poured on the tested wood. It was spread homogeneously with the in-situ growth technique. Wood coatings were dried in an oven at 50 °C for 24 hours. In total, 15 different samples (3 different kinds of wood coated by 5 different coating materials) were obtained. All samples were systematically coded. For example, the sample with beech veneer impregnated with cationic cellulose was coded as BCS, while the sample with sodium alginate impregnation on oak veneer was coded as OSA.

2.4 Device fabrication
2.4. Izrada uređaja

Triboelectric devices were prepared by using wooden materials impregnated with anionic and cationic solutions. All wood veneers were used in pairs with each other according to Sun *et al.* (2021) method to determine the best triboelectric performing device. 15 different wood coatings impregnated with solution were covered with 1 mm thick commercial copper foil to conduct electricity. The solution-impregnated wood veneers were joined with the copper foil-covered parts to the outside and device pairs were prepared. In this way, a total of 210 different device pairs were obtained.

2.5 Methods
2.5. Metode

Infrared (IR) spectra of coated wood materials were obtained on a Perkin Elmer UATR instrument. Each test material was directly analyzed in transmission mode in the range of 4000–400 cm^{-1} with 4 scans per sample at room temperature.

By applying a certain force to the prepared devices, the measurements of the electrical output formed in the material were taken. At the same time, a loading cell was mounted on the rigid frame of the motor to monitor the pressure applied to the samples. The electrical output was measured by a Digilent Analog Discovery 2, equipped with a WaveForms software.

The surface hydrophobicity of wood coatings was analyzed measuring the contact angle using a goniometer (Krüss Advance Drop Shape Analyzer, DSA100). The coated-wood material was cut into a thin strip before being analyzed. For the measurement of the contact angle, measurements were taken from 2 µl of distilled water (pH 7) or diiodomethane dropped on the tested wood surface. Measurements were taken within 10 seconds of instillation of drops.

The geometric mean method, which is based on the contact angle measurement of 2 different liquids deposited on a solid wood surface, was used to calculate the surface free energy of wood coatings using Eq. 1 described by Fernandez and Khayet (2015).

$$2\sqrt{\gamma_s^d \cdot \gamma_l^d} + 2\sqrt{\gamma_s^{nd} \cdot \gamma_l^{nd}} = \gamma_l(1+cosQ) \quad (1)$$

Where γ_s^d is the dispersive component of the solid; γ_l^d is the dispersive component of the liquid; γ_s^{nd} is the non-dispersive component of the solid; γ_l^{nd} is the non-dispersive component of the liquid; γ_l is the total surface free energy or surface tension of the liquid and Q is the contact angle measured between the liquid and the solid under study.

From the total surface tension and surface tension components estimated by the methods described above, the solubility parameter (δ) of wood surface coatings was calculated using the Eq.2 and Eq.3 (Fernandez and Khayet (2015)).

$$e_c = (\gamma_s / 0.75)^{3/2} \quad (2)$$

$$\delta = (e_c)^{1/2} \quad (3)$$

Where e_c (mJ/m^3) is the cohesive energy density, which is related to γ_s (mJ/m^2) as Eq. 3 (Victoria and Mohamed, 2015).

Morphology of the solution-impregnated wood coatings was determined by SEM using Phillips XL 30 ESEM-FEG microscope. Samples were cryo-fractured in liquid nitrogen, covered with a thin gold layer, and the fractured surface was examined under SEM analyses.

2.6 Statistical analyses
2.6. Statistička analiza

All measurements for the surface contact angle of water and diidomethane were repeated five times independently, and the results were expressed as means ± standard deviations. The data calculated from the surface contact angle of water and diidomethane for surface free energy and solubility parameter were analyzed using the Minitab statistical software. Statistical differences between groups were determined by the one-way ANOVA test followed by the Tukey Method ($p<0.05$).

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Structural characterization of functional wood materials
3.1. Strukturna obilježja funkcionalnih drvnih materijala

The FT-IR spectra of pure beech, mahogany and oak wood coatings and chemically impregnated coatings are shown in Figure 1. When the FT-IR spectrum of the pure beech veneer is examined, bands are observed at 1736 cm^{-1} of C–O stretch in cellulose and hemicellulose and at 1032 cm^{-1} of carbonyl groups (Geffert *et al.,* 2017). In the FT-IR spectrum of undoped mahogany, the band belonging to the unconjugated carbonyl group is seen at 1743 cm^{-1} (Jian, 2003; Wang *et al.,* 2017).

Similarly, a band of unconjugated carbonyl group is seen at 1732 cm^{-1} in the FT-IR spectrum of pure oak

(Kubovský *et al.,* 2020). In the FT-IR spectra of coatings containing cationic starch, belonging to the hydroxyl group at 3275 cm^{-1}, C-H stretching vibration at 2919 cm^{-1}, C-O stretching vibration at 1148, 1076 and 996 cm^{-1}, at 1473 cm^{-1} the band of C-N stretching vibration is seen (Pal *et al.,* 2005; Şen *et al.,* 2017). Similarly, when the FT-IR spectra of wood materials containing cationic hydroxyethyl cellulose are examined, the broad band of the stretch vibrations of the hydroxyl group are observed at 3378 cm^{-1}, the band of C-H stretching vibrations at 2867 cm^{-1}, the C-O in the anhydroglucose units of hydroxyethyl cellulose at 1049 cm^{-1}. A band of stretching vibrations and a band of C-N stretching vibrations at 1475 cm^{-1} were observed (Şen and Kahraman, 2018). In the FT-IR spectra of wood materials containing sodium alginate, bands of -COO- asymmetric and symmetric stretching vibrations at 1417 cm^{-1} and 1615 cm^{-1}, and bands of -OH and C-H stretching vibrations at 2922 cm^{-1} and 3428 cm^{-1} were detected (Zhang *et al.,* 2018). In the FT-IR spectra of polyethyleneimine-containing wood coating materials, the bands of C–H bond were observed at 2836 cm^{-1} and 2947 cm^{-1}, while the band of primary amines (N-H) was observed at 1585 cm^{-1} (Linden *et al.,* 2015). In the FT-IR spectra of the samples containing carboxymethyl cellulose, the band due to the stretching frequency of the hydroxyl group was observed at 3423 cm-1, the band due to the C-H stretching vibration at 2920 cm^{-1}, the band due to the presence of COO- at 1620 cm^{-1}, 1423 and 1328 cm^{-1}. The band due to $-CH_2$ and hydroxyl group bending vibration around the band due to $CH\text{-}O\text{-}CH_2$ stretching at 1054 cm^{-1} was observed (Haleem *et al.,* 2014). The presence of specific bands of impregnated chemicals in the FT-IR spectra of wood materials proves that the chemicals are successfully impregnated on wood materials.

3.2 Triboelectric performance of devices
3.2. Triboelektrična svojstva uređaja

The images of the highest measured stresses of Beech, Mahogany, and Oak veneers are shown in Figure 2. When the obtained results were examined, it was found that PEI impregnated beech veneer and CS impregnated Mahogany veneer had the highest (1.72V) triboelectric production. It was observed that the highest values obtained were in all PEI-impregnated wood coatings. On the other hand, it is seen that devices consisting mostly of CS-impregnated wood materials and PEI-impregnated wood materials have the highest stresses. As a result, it can be interpreted that PEI and CS have the highest tribopolarity. When the relationship between wood veneer types was examined, similar results were observed in beech and mahogany, while lower strains were measured in oak. It is thought that the reason for this is that the impregnation process becomes difficult due to the formation of tulle in the anatomical structure of the oak tree. Sun *et al.* (2021) prepared tribolelectric nanogenerators by applying zeolitic imidazolate framework-8, a metal-organic framework and poly(dimethylsiloxane) to spruce veneers. They reported measuring an open circuit voltage 80 times higher than that of natural wood (spruce). This

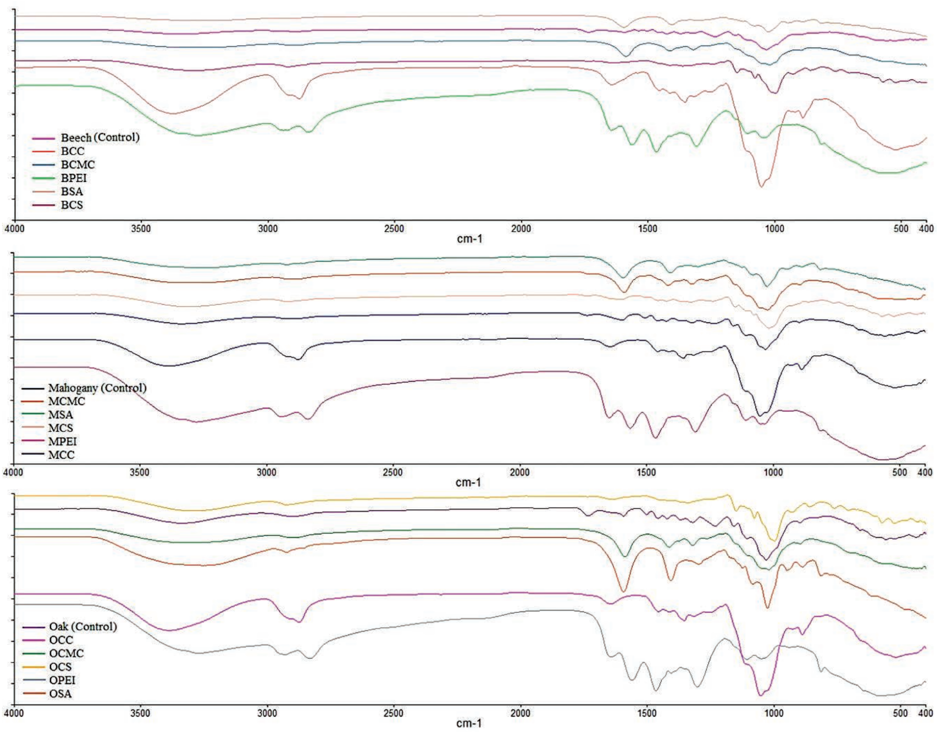

Figure 1 FT-IR spectra of all samples
Slika 1. FT-IR spektri svih uzoraka

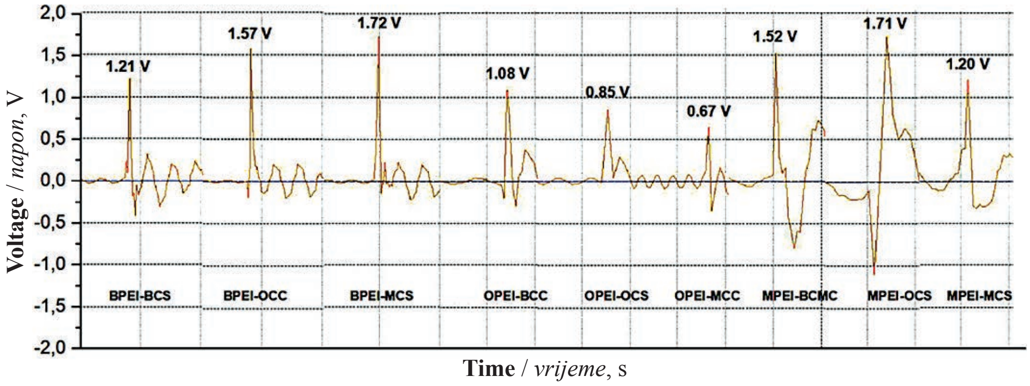

Figure 2 Triboelectric output of functionalized wood veneers
Slika 2. Triboelektrični izlaz funkcionaliziranih drvnih furnira

result is an indication that triboelectricity can be imparted to wood material by treating chemicals with various tribopolarity. It is seen that the results obtained in our study are compatible with the literature.

3.3 Contact angle and surface properties of functional wood materials
3.3. Kontaktni kut i površinska svojstva funkcionalnih drvnih materijala

Contact angle measurements were carried out to examine the wettability of pure wood veneers and the resulting functional wood veneers. The images of the contact angles of the samples with water (the closest value to the mean) are shown in Figure 3. According to the results, the highest contact angle value among all groups was measured as 105.9º in the functional material prepared by impregnating the oak veneer with cationic starch. On the other hand, the lowest contact angle value among all groups was measured as 25.7º in the functional material prepared by impregnating the beech veneer with carboxymethyl cellulose. In general, increases in contact angle values were observed with chemical impregnation of all wood coatings. Increasing the contact

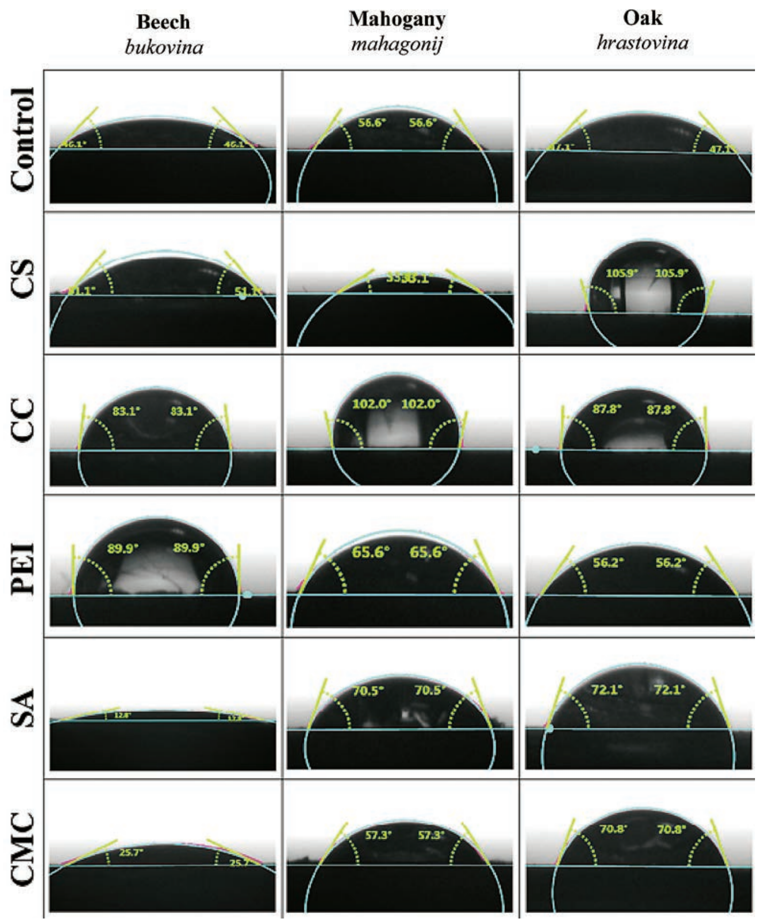

Figure 3 Contact angle images of functional wood materials with water (closest to mean)
Slika 3. Slike kontaktnog kuta vode na funkcionalnim drvnim materijalima (najbliža vrijednost srednjoj vrijednosti)

angle value gives the material a hydrophobic property and reduces its wettability (Huhtamäki *et al.,* 2018). While wooden materials are generally in contact with water, their mechanical properties decrease over time. By impregnating wood materials with chemicals, the mechanical properties of the coatings were kept stable.

Surface free energies and solubility parameters calculated from the contact angles of functional wood coatings with water and diiodomethane are shown in Table 1. The highest surface free energy in mahogany coating was observed in MCS sample as 62.25. The lowest surface free energy was calculated as 35.62 in the MPEI sample. While the highest surface free energy in beech veneer was 66.55 in BCMC sample, and the lowest surface free energy was observed in BPEI sample as 28.60. The highest surface free energy was calculated as 50.59 in the oak veneer, while the lowest surface free energy was calculated as 39.76 in the OCC sample. Similar results are valid for the calculated solubility parameter values. According to the results obtained, it is seen that the surface free energy and solubility parameter decrease with the increase of the contact angle and a more hydrophobic surface is obtained (Chieng *et al.,* 2019).

3.4 Morphology of functional wood materials
3.4. Morfologija funkcionalnih drvnih materijala

Morphological structures of pure and functionalized wood veneers were examined by scanning electron microscopy. Images of all samples at 500x magnification were used for characterization (Figure 4). When the SEM images were examined, it was determined that the chemicals impregnated on the wood coatings homogeneously filled the pores of the wood materials. Homogeneous distribution of chemicals impregnated on wood coatings is a factor that significantly affects the triboelectric and wettability properties of materials.

4 CONCLUSIONS
4. ZAKLJUČAK

In this study, triboelectric behavior and hydrophobic properties of functional wood coatings obtained by impregnating 3 different wood coatings with 5 different chemicals were examined. According to the structural characterization results, it was determined that the chemicals applied to all wood coating types were successfully impregnated. It was seen that the chemical groups showing the highest tribopolarity of the triboelectric measurements were PEI and CS, and at the same time, more tension was obtained in beech and mahogany veneer compared to oak veneer. This was measured by the BPEI-MCS device with the highest voltage of 1.72 V. The contact angle measurement results showed that the application of chemicals to wood coatings increases the wettability of the materials, that is, the hydrophobicity. The contact angle values of pure wood veneers increased from around 50 degrees to over 100 degrees af-

Table 1 Surface free energies and solubility parameters of functional wood coatings
Tablica 1. Slobodne površinske energije i parametri topljivosti funkcionalnih premaza za drvo

Sample / *Uzorak*		**Water** *Voda*	**Diiodomethane** *Dijodometan*	**Surface free energy** **ɤ, mJ/m²** *Slobodna površinska energija* ɤ, mJ/m²	**Solubility parameter** **δ, mJ$^{1/2}$/m$^{3/2}$** *Parametar topljivosti* δ, mJ$^{1/2}$/m$^{3/2}$
M	Control	$57.62^{\pm4.15}$	$43.46^{\pm2.05}$	$47.88^{\pm2.78b}$	$22.58^{\pm0.98b}$
	CC	$98.68^{\pm6.06}$	$40.92^{\pm2.51}$	$40.40^{\pm0.40c}$	$19.88^{\pm0.15c}$
	CMC	$57.10^{\pm2.52}$	$46.14^{\pm5.02}$	$47.43^{\pm2.71b}$	$22.42^{\pm0.96b}$
	CS	$33.32^{\pm5.72}$	$43.82^{\pm2.18}$	$62.10^{\pm3.02a}$	$27.44^{\pm1.00a}$
	PEI	$79.70^{\pm21.50}$	$51.02^{\pm2.05}$	$39.77^{\pm4.92c}$	$19.62^{\pm1.83c}$
	SA	$66.34^{\pm6.20}$	$41.96^{\pm6.39}$	$40.01^{\pm1.92c}$	$19.73^{\pm0.71c}$
B	Control	$44.98^{\pm2.73}$	$50.82^{\pm2.48}$	$54.13^{\pm2.12b}$	$24.76^{\pm0.73b}$
	CC	$82.00^{\pm6.70}$	$41.90^{\pm2.18}$	$39.57^{\pm2.03c}$	$19.57^{\pm0.75c}$
	CMC	$25.62^{\pm2.19}$	$40.98^{\pm2.59}$	$66.52^{\pm1.20a}$	$28.90^{\pm0.39a}$
	CS	$51.62^{\pm4.60}$	$40.30^{\pm3.45}$	$52.03^{\pm3.21b}$	$24.03^{\pm1.12b}$
	PEI	$92.36^{\pm3.27}$	$60.48^{\pm3.70}$	$28.64^{\pm2.22d}$	$15.35^{\pm0.89d}$
	SA	$14.16^{\pm3.04}$	$38.36^{\pm0.85}$	$71.04^{\pm0.85a}$	$30.36^{\pm0.27a}$
O	Control	$48.30^{\pm1.02}$	$62.90^{\pm3.13}$	$51.59^{\pm1.01a}$	$23.89^{\pm0.35a}$
	CC	$89.44^{\pm5.07}$	$39.72^{\pm1.21}$	$39.99^{\pm0.50b}$	$19.73^{\pm0.18b}$
	CMC	$71.24^{\pm3.03}$	$38.28^{\pm1.01}$	$43.39^{\pm1.30b}$	$20.98^{\pm0.47b}$
	CS	$107.20^{\pm8.64}$	$39.64^{\pm2.25}$	$43.97^{\pm1.77b}$	$21.18^{\pm0.64b}$
	PEI	$55.98^{\pm2.44}$	$42.46^{\pm4.84}$	$49.01^{\pm2.32a}$	$22.98^{\pm0.82a}$
	SA	$66.72^{\pm6.82}$	$46.62^{\pm2.71}$	$42.42^{\pm4.11b}$	$20.61^{\pm1.49b}$

All values are expressed as arithmetic mean of five different measurements, with ± standard deviation values. / *Sve su vrijednosti izražene kao aritmetička sredina pet različitih mjerenja s vrijednostima ± standardne devijacije.*
[a–d]Similar letters show no statistical difference within each group at $p<0.05$. / [a–d]*Ista slova ne pokazuju statističku razliku unutar svake skupine pri p<0,05.*

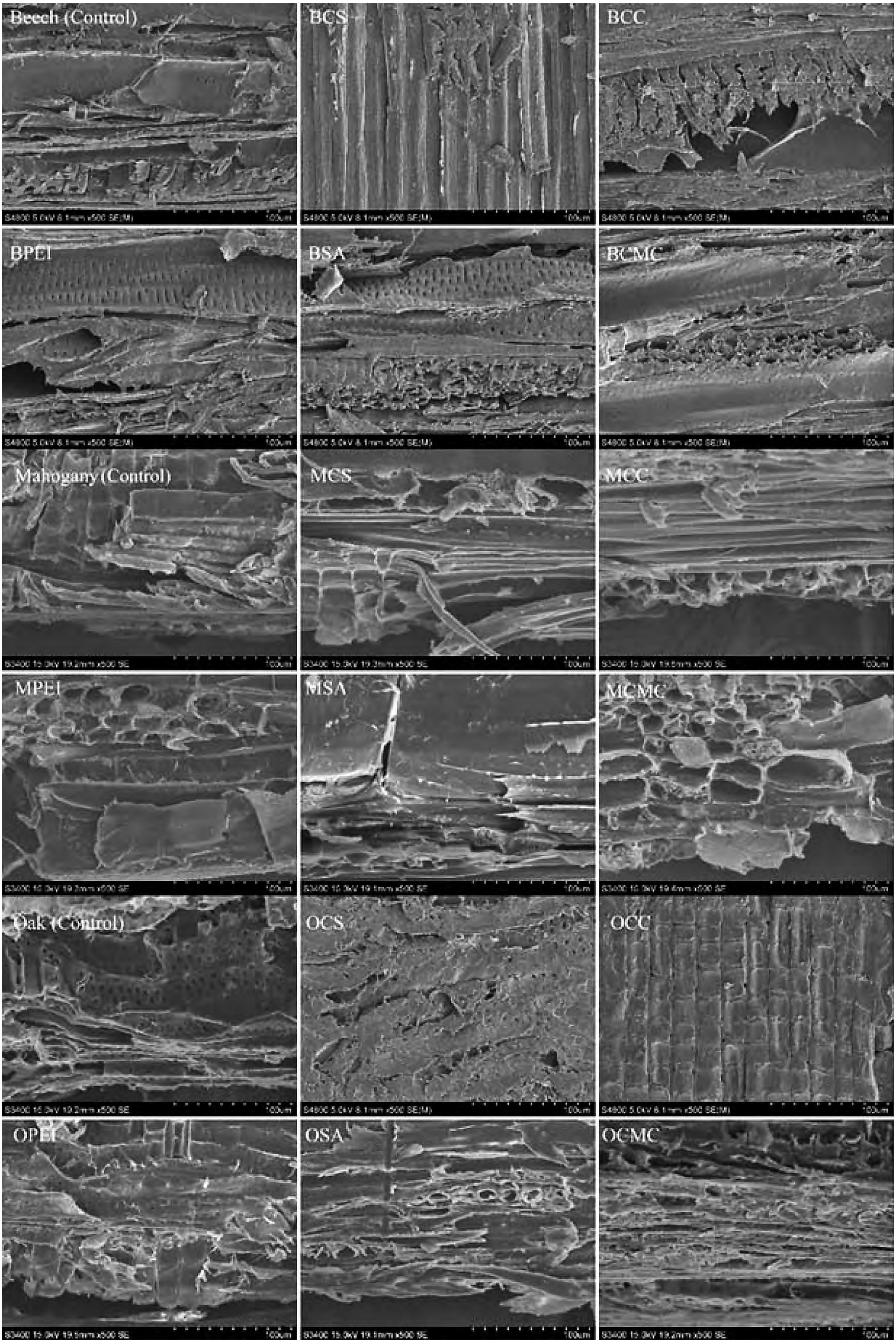

Figure 4 SEM images of functional wood materials
Slika 4. SEM slike funkcionalnih drvnih materijala

ter the impregnation process. On the other hand, it was determined that the surface energies and solubility parameters of the samples decreased depending on the contact angle values. In addition, SEM images proved that the chemicals successfully filled the pores of the wood material. Finally, biomaterials exhibit efficient tribopolarity behavior and may contribute to the applications of the next generation of sustainable smart materials, shedding light on future research studies.

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Selected Chemical and Physical Properties of Pine Wood Chips Inoculated with *Aspergillus* and *Penicillium* Mold Fungi

Odabrana kemijska i fizička svojstva drvne sječke od borovine inokulirane plijesnima *Aspergillus* i *Penicillium*

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ABSTRACT • *Mold fungi representing genera of Penicillium and Aspergillus commonly develop on the surface of freshly harvested wood chips during storage. As a result, they are often considered as low-quality material and intended for incineration. Thus, the aim of the present study was to investigate the effect of wood chips infestation with mold fungi representing genera of Aspergillus and Penicillium on their basic chemical and physical properties, such as: chemical structure (evaluated with FTIR spectroscopy), mass loss and hygroscopicity, after an incubation of 3, 6 and 9 weeks. Based on the visual assessment and ergosterol content analysis, it was found that inoculation of wood chips with molds led to the intense fungal development on their surface. However, as observed in FTIR spectra, the presence of molds caused no changes in wood chemical structure. Furthermore, no mass loss and no significant increase in the hygroscopicity of wood were observed. Therefore, pine wood chips overgrown by studied genera of fungi seem to be a valuable material for various applications.*

KEYWORDS: *mold fungi; wood chips; FTIR spectroscopy; mass loss; hygroscopicity*

SAŽETAK • *Plijesni rodova Aspergillus i Penicillium najčešće se razvijaju na površini svježe pripremljene drvne sječke tijekom skladištenja, zbog čega se takva sječka često smatra nekvalitetnim materijalom i spaljuje se. Stoga je cilj ovoga istraživanja bio istražiti utjecaj zaraženosti drvne sječke plijesnima rodova Aspergillus i Penicillium na njezina osnovna kemijska i fizička svojstva kao što su kemijska struktura (ispitano FTIR spektroskopijom), gubitak mase i higroskopnost nakon ikubacije od tri, šest i devet tjedana. Na temelju vizualne procjene i analize sadržaja ergosterola, utvrđeno je da je inokulacija drvne sječke plijesnima prouzročila intenzivan razvoj plijesni na površini*

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drvne sječke. Međutim, na FTIR spektrima je uočeno da plijesni nisu uzrokovale promjene u kemijskoj strukturi drva. Nadalje, nije uočen gubitak mase niti znatno povećanje higroskopnosti drva. Stoga se može zaključiti da je drvna sječka od borovine zaražena promatranim rodovima plijesni upotrebljiv materijal za različite primjene.

KLJUČNE RIJEČI: *plijesan; drvna sječka; FTIR spektroskopija; gubitak mase; higroskopnost*

1 INTRODUCTION
1. UVOD

Mold fungi are the example of microorganisms originating from different taxonomic groups, resulting in various physiological reactions. Due to this diversity, they have the ability to develop on many different types of surfaces such as e.g. wood, wood-based materials, gypsum boards, ceramics, etc. (Nielsen *et al.*, 2004). Factors influencing the development of molds include the availability of nutrients, humidity, air flow, sunlight, temperature and pH (Schmidt, 2006). The temperature ranging from 20 to 35 °C is considered to be the optimum for the development of mold fungi (Viitanen and Bjurman, 1995). Moreover, relative air humidity of approx. 80 % seems to be a minimum required for their growth (Viitanen *et al.*, 2011).

In terms of nutrients, mold fungi use starch, sugars and proteins causing the parenchyma cells to grow in the wood surface layer. According to Ahmed *et al.*, (2013), the depth of penetration is usually around 1 mm. They do not cause a degradation of structural constituents of wood and, therefore, they are significantly less destructive comparing to brown-rot and white-rot fungi (Broda, 2020; Woźniak, 2022). Although it is believed that they only reduce the aesthetic value of wood without reducing its mechanical properties, there are indications in literature that mold fungi can also cause some changes in the wood characteristics. Darwish *et al*. (2013) observed that the development of molds can reduce crystallinity of cellulose and cause a change in FTIR (Fourier transform infrared spectroscopy) spectra indicating changes in the chemical structure of lignin and carbohydrates. Furthermore, Salem *et al.* (2016) stated that the colonization of mold fungi can contribute to the increase in the content of copper, chlorine and aluminium and a decrease in the content of carbon. Hamed and Mansour (2018) observed that the advanced development of molds can lead to a degradation of wood ultrastructure.

Wood chips are the example of wooden materials susceptible to mold fungi, especially when they are freshly harvested (Idler *et al*., 2019). According to Mirski *et al*. (2019), the annual production of wood chips during the softwood processing in Polish sawmills can reach millions of cubic meters. They are usually considered as a by-product occurring during roundwood cutting process and they are intended for the storage after the processing is completed. Usually, the storage process takes place outdoors, which means that the wood can be exposed to increased humidity. Moreover, the elevated temperature inside the chip pile also favors the growth of microorganisms (Krigstin *et al.*, 2019). Studies performed by Alakoski *et al*. (2016) and Lieskovský *et al*. (2017) showed that mold fungi (including genera of *Aspergillus* and *Penicillium*) are commonly found in landfills. Although, as mentioned above, it is believed that molds do not deteriorate the basic properties of wood, a raw material visibly infested by them is considered to be of poor quality and is usually intended for incineration (Knoll *et al*., 1993). However, due to the progressive shortages of raw material (Taghiyari *et al*., 2020), the diversity of available energy biomass (Mat Aron *et al*., 2020) and the assumptions of sustainable economy (Kawamura and Wang, 2020), the alternative possibilities of using wood degraded by fungi are constantly sought. The example of potential applications for such material may be a preparation of termite attractants (Esenther *et al.*, 1961; Su, 2005), synthesis of adhesives (Heritage, 1954; Kawamura and Wang, 2020; Li and Geng, 2005) and manufacturing of wood-based composites (Ayrilmis *et al*., 2015; Ge *et al*., 2018; Nemli *et al*., 2018). However, in order to look for new ways of application of pine wood chips covered with mold fungi, their properties should be thoroughly investigated.

Therefore, taking into account that the knowledge about the effect of mold fungi development on the properties of wood chips is scarce and the issue is expected to gain even more importance due to the increased processing of hardwood in the years to come in Poland, hardwood usually being even less resistant to biotic factors (Gendek *et al*., 2018; Zajączkowski *et al*., 2013), it was decided to carry out the experiments aimed at determining the changes in the basic properties of wood chips, such as: chemical structure, hygroscopicity and mass loss caused by the inoculation. Moreover, due to the intensive search for the new applications for degraded wood, the present research can be a valuable reference and contribute to finding new ways of use of raw material overgrown by molds, other than for energy purposes.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

Pine (*Pinus sylvestris* L.) wood chips produced during roundwood processing were supplied by KPPD

Szczecinek S.A. (Kalisz Pomorski, Poland). In order to get rid of larger pieces, wood chips were sieved with flat sieves using a mesh with square perforations of 50 mm × 50 mm. The individual fragments of the bark remaining in the mixture after sorting were removed manually. Due to the fact that both wood moisture content and chip size can potentially influence the growth of microorganisms (Hukka and Viitanen, 1999; Idler *et al*., 2019), each batch of wood chips was characterized before being placed in the chamber and before the application of inoculum. Moisture content (*MC*) of wood was determined immediately before the inoculation. 20 pieces of wood chips were collected from each batch of material and their *MC* was investigated using a dry oven method according to PN-77/D-04100 (1978). In addition, 500 chips were collected from each batch to determine their length, width and thickness using a calliper with an accuracy of 0.01 mm. Moreover, in order to characterize their shape, the degree of slenderness (λ), flatness (Ψ) and width coefficient (m) were calculated according to the equations presented in the study of Dukarska *et al*. (2021). Bulk density of wood chips was determined by investigating the mass of wood chips loosely poured in a vessel with a volume of 5000 cm^3.

For the production of inoculum, the species of molds representing genera of *Aspergillus* and *Penicillium,* identified with the use of genetic methods in previous studies involving the analysis of the biodegradation of wood chips during short-term storage, were used. A specific list of species can be found in a recently published article (Mirski *et al*., 2022). It is a favorable solution due to the fact that the composition of the inoculum reflects a diversity of mold fungi actually occurring in landfills. The spore suspension was prepared according to the method previously applied by Góral *et al*. (2015) and Buśko *et al*. (2014). Isolates were incubated on plates filled with agar medium for 4 weeks. The plates were washed with saline to prepare spore suspensions. The concentration of spore suspension was determined using the haematocrit at approx. 5×10^5 spores/ml. The proportion of *Aspergillus* spores to *Penicillium* spores was 1:1. The material was sprayed with the suspension in the amount of 100 ml/1 kg of wood chips. The inoculated material was stored for 3, 6 or 9 weeks in the chamber. For the incubation, the temperature of 25°C and the relative humidity (RH) of 95 % were applied. The assumed RH was obtained by keeping the material above supersaturated solution of potassium sulphate. Wood chips incubated for 3, 6 and 9 weeks were labeled as Z-3, Z-6, Z-9, respectively. Moreover, the reference variant which was not subjected to the inoculation was labeled as Z-0.

In order to assess the progress in the development of fungi on wood chips during the assumed incubation periods, the method previously used by Kwaśniewska-Sip *et al*. (2018) was applied. 50 pieces of wood were collected from each variant after the end of incubation and evaluated visually with the help of loupe with the magnification of 10×. Wood chips were classified with the five-class assessment system used by Imken *et al*. (2020): class 0 = no fungal growth, class 1 = 1-25 % of the surface infested, class 2 = 26-50 % of the surface infested, class 3 = 51-75 % of the surface infested, class 4 = 76-100 % of the surface infested.

One of the commonly used methods of chemical analysis allowing for the determination of fungal development is the determination of ergosterol (ERG) content. For this purpose, the method previously used to analyze its content in wood dust (Pędzik *et al*., 2021; Stuper-Szablewska *et al*., 2017; Szwajkowska-Michałek *et al*., 2020) and bark (Szwajkowska-Michalek *et al*., 2019) was applied. A detailed description of the determination using UPLC can be found in the abovementioned papers.

Commonly used Fourier transform infrared spectroscopy was used to determine the effect of mold growth on the chemical structure of wood chips. The advantage of this technique is that it requires little time and material to perform the analysis (Chen *et al*., 2010; Traoré *et al*., 2016). It is used for example to assess the structure of wood components (Siuda *et al*., 2019), the effect of various degrading factors (Pandey and Pitman, 2003) and protective measures (Woźniak *et al*., 2020). (5 ± 0.01) g of wood chips were ground in laboratory mill and sieved to collect the fraction of 0.125 mm. The wood powder was mixed with KBr at a 1/200 mg ratio. The analysis was performed with the use of Bruker FTIR IFS 66/s spectrometer (Bruker, Ettlingen, Germany) with the Fourier transform range (500–4000 cm^{-1}), registering 32 scans at the resolution of 4 cm^{-1}.

Before the inoculation, 20 wood chips from each variant were dried to a constant weight at (102±1) °C. Then their weight was determined with an accuracy of 0.001 g. Chips prepared in this way were seasoned for 24 hours with the rest of the mixture to equalize the MC. Then, they were placed in the chamber together with the remaining material. After the incubation process, their weight was determined again with an accuracy of 0.001 g after drying to a constant weight at (102±1) °C. The mass loss (*ML*) was calculated according to Eq. 1.

$$ML = \frac{m_1 - m_2}{m_1} \cdot 100\ \% \tag{1}$$

Where: m_1 – mass of wood chip before inoculation (g), m_2 – mass of wood chip after incubation (g).

The methodology applied by Siuda (2019) to assess the hygroscopicity of oak wood modified with trialkoxysilanes was used to determine the effect of fungal inoculation on wood chips hygroscopicity. For this purpose, (10±0.05) g of wood chips from each variant were collected and ground in a laboratory mill to obtain the fraction of 0.2 mm. 10 pellets were formed from the obtained material with the use of a hand press. The diameter of each pellet made from (1±0.01) g of wood powder was 12 mm. Produced pellets were dried in a laboratory oven to a constant weight at (102±1) °C, and then placed in sealed containers over a supersaturated ammonium phosphate solution. The use of chosen salt ensured the conditions of RH at the level of 90-95 %. The weight gain of pellets was measured after 15 min; 30 min; 1 h; 2 h; 4 h; 8 h; 24 h; 48 h; 72 h and 120 h.

The obtained results were analyzed with the use of Statistica 13.3 software. The empirical distributions of the features were presented as histograms and their interpretation was carried out using the Shapiro-Wilk test. This test is commonly used to check the normality of the distribution of random variables. In order to investigate the influence of independent variables on the dependent variable, a multivariate analysis of variance (ANOVA) was used. During the analysis, the following tests were used: Fisher test, *t*-Student test, HSD Tukey test and Kruskal-Wallis test depending on the type of data, at the significance level of $\alpha = 0.05$. The use of the above tests allowed for the assessment of the significance of differences by identification of homogeneous groups.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

Based on the Shapiro-Wilk test results, it was found that only in the case of MC of chips labeled as Z-9 there was a normal distribution at the assumed significance level. For each variant, the chip length values were mostly concentrated around the average value (approx. 60 % of the results were in the range between 30 and 35 mm). A similar tendency was also observed in the case of width and thickness where approx. 80 % of the observations were in the range of 12-14 mm and approx. 70 % in the range of 4.0 – 4.5 mm, respectively.

Sieving the chips before the inoculation may have resulted in less variable dimensions due to the reduction in the occurrence of chips larger than 50 mm. When analyzing the MC distributions, it was found that, depending on the type of raw material, 40-50 % of the chips in the mixture were characterized by a *MC* of 20.0-20.5 %. Despite the fact that no normal distribution was found, it was decided that, as in the case of Mirski (2013) research, the power of statistical tests was sufficient enough to perform and evaluate the results of analysis of variance. The results of the analysis of the significance of differences between the variants are presented in Table 1.

Based on the analysis of homogeneous groups determined in the HSD Tukey test, it was found that regardless of the variant, there were no statistically significant differences in the dimensions, shape and *MC* of the materials. Therefore, when interpreting the results of further analysis, the influence of the size of chips and their initial MC on the development of microorganisms can be omitted.

After removing the wood chips from the chamber, different infestation levels were identified depending on the incubation time. The Shapiro-Wilk test showed that no normal distribution was found for infestation classes for either inoculated variant. Despite the fact that no normal distribution was found at the assumed significance level, the histograms showed that the vast majority of observations represented the mean value. Moreover, as can be seen from the data presented in Figure 1, the longer the assumed incubation time, the higher infestation class of the chips. Homogeneous groups determined in the Kruskal-Wallis test indicate that the differences between the variants were statistically significant. The greatest increase in the fungal growth was observed between the third and sixth week of the experiment, when the infested area increased from 1-25 % of the chip surface to 51-75 %. Further extension in the incubation time to 9 weeks caused a complete infestation of the wood surface. The progressive development of mold fungi on the surface of wood chips resulted from the optimal conditions for their growth (temperature of 25 °C and RH of 95 %) provided during the incubation (Sedlbauer, 2001; Viitanen

Table 1 Dimensions, moisture content and shape of the chips intended for inoculation
Tablica 1. Dimenzije, sadržaj vode i oblik sječke pripremljene za inokulaciju

Parameter *Parametar*	**Unit** / *Jedinica*	**Variant label** / *Oznaka varijante*		
		Z-3	Z-6	Z-9
l	mm	30.32 ± 3.21 *a*	30.38 ± 3.24 *a*	30.49 ± 3.35 *a*
b		12.84 ± 0.89 *a*	12.92 ± 1.02 *a*	13.05 ± 1.15 *a*
a		4.11 ± 0.29 *a*	4.14 ± 0.31 *a*	4.19 ± 0.37 *a*
MC	%	19.98 ± 0.99 *a*	20.00 ± 0.84 *a*	20.36 ± 0.59 *a*
Ψ	-	3.13 ± 0.19 a	3.14 ± 0.18 *a*	3.11 ± 0.31 *a*
λ		7.37 ± 0.57 *a*	7.35 ± 0.64 *a*	7.30 ± 0.76 *a*
m		2.36 ± 0.16 *a*	2.35 ± 0.21 *a*	2.34 ± 0.23 *a*
Bulk density	kg/m³	193.67 ± 1.53 *a*	194.00 ± 1.73 *a*	193.33 ± 2.08 *a*

Mean value ± standard deviation; letters a, b mark homogeneous groups in HSD Tukey test / *srednja vrijednost ± standardna devijacija; slovima a, b označene su homogene skupine u HSD Tukey testu*

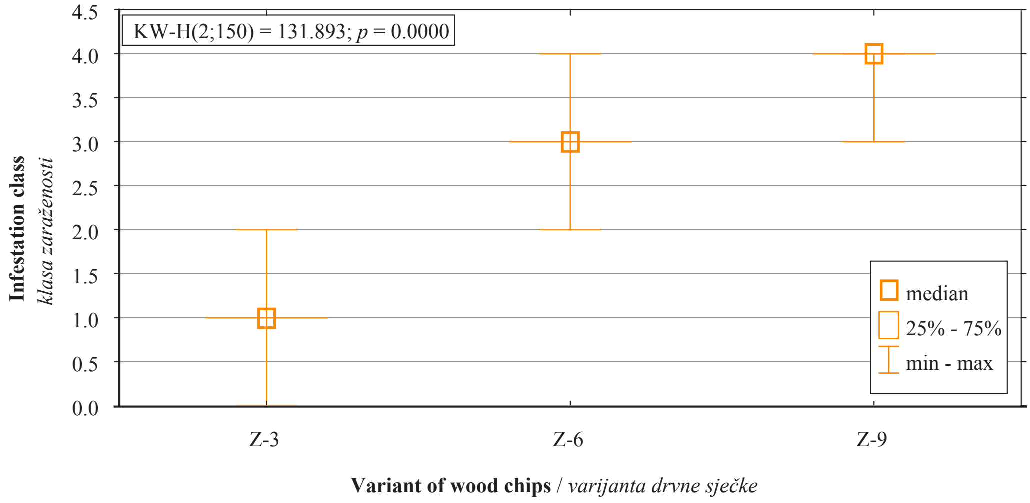

Figure 1 Infestation class of wood chips depending on incubation time
Slika 1. Klasa zaraženosti drvne sječke s obzirom na vrijeme inkubacije

and Bjurman, 1995). The effect of the incubation time on the infestation class tested in laboratory conditions may differ from the experiment carried out in a natural environment. In the case of wood stored in climatic chambers, constant conditions (RH, temperature) were ensured. On the other hand, in a natural environment characterized by variable weather conditions, which often differ significantly from the optimum, the so-called stress factors may occur. They result mainly from too low air RH or inadequate temperature (Zak and Wildman, 2004). Moreover, according to Johansson *et al*. (2013), the conditions of variable RH and ambient temperature during the incubation of wood slow down the growth of molds. This effect is particularly noticeable when the changes are rapid (Viitanen and Bjurman, 1995).

The results of ergosterol content determinations in inoculated wood chips differing in incubation times are presented in Figure 2. Homogeneous groups determined in the HSD Tukey test indicate that the content of ERG differed in a statistically significant way depending on the length of the incubation period and it increased over time. Between the third and sixth week of incubation, the ERG concentration increased by 14.50 mg/kg. Furthermore, in the case of the nine-week incubation, the ERG content increased by 57.33 mg/kg and 71.83 mg/kg when compared to the concentration determined in the sixth and third weeks, respectively. Therefore, the results of the analysis of ERG content correspond to the assessment of the infestation classes, which stays in agreement with the results presented by Pasanen *et al*. (1999). Thus, the results confirmed that, as the incubation time of wood chips is extended under favorable conditions, the area overgrown by molds is also extended.

The spectra of both sound and inoculated wood chips are presented in Figure 3. A wide peak corresponding to stretching vibrations of OH bonds of hydroxyl groups was observed in the range of 3700-3050 cm^{-1} (Ghahri *et al*., 2018). Moreover, in the range of

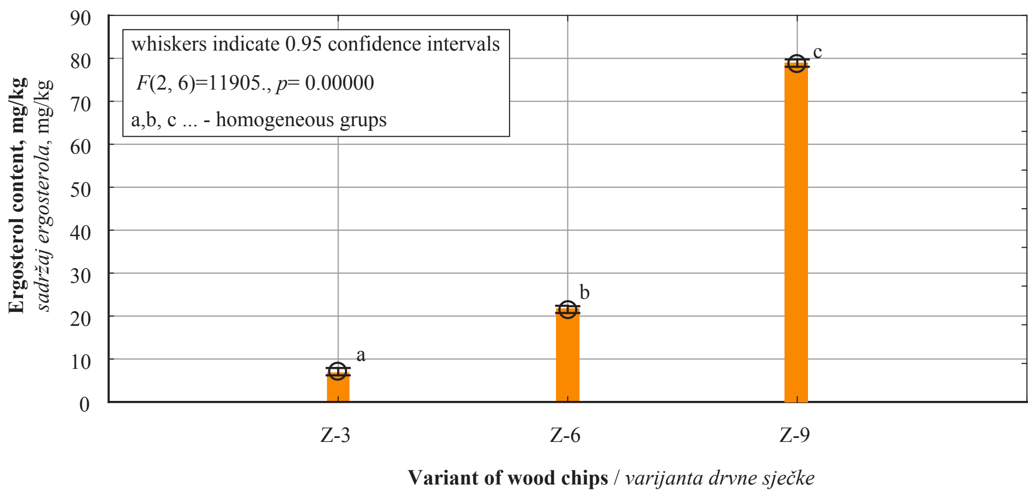

Figure 2 Ergosterol content in wood chips depending on incubation time
Slika 2. Sadržaj ergosterola u drvnoj sječki ovisno o vremenu inkubacije

2980-2830 cm^{-1}, stretching vibrations of C-H were identified and it was overlapped by the C-O stretching vibrations generated by fatty acids (Gupta *et al*., 2011). At about 1737 cm^{-1}, a peak from a vibration of C=O bond of hemicelluloses was recorded (Li *et al.*, 2011). In the fingerprint region (1800-700 cm^{-1}), several more band characteristics of wood were noted. The peak occurring at 1510 cm^{-1} corresponded to the vibrations of the aromatic ring of lignin. The peaks at 1372 cm^{-1} and 1161 cm^{-1} originated from deformation vibrations of C-H bonds and vibrations of C-O-C bonds, respectively. These bands were derived from the polysaccharides such as cellulose and hemicelluloses (Pandey and Pitman, 2003). At 1234 cm^{-1} there were also signals from carbon-oxygen stretching bonds occurring in lignin. Furthermore, the peak observed at 898 cm^{-1} was assigned to C-H deformation bond. The comparison between reference wood sample and those inoculated showed no significant changes in the course of FTIR spectra, regardless of the incubation period. This observation indicates that inoculation of wood chips with mold fungi did not have a significant effect on their chemical structure. Jelle and Hovde (2012) investigated the possibility of using attenuated total reflection infrared spectroscopy (ATR-FTIR) to detect mold fungi on the surface of various materials, including wood. Moreover, the authors found that the application of this method has a great application potential and that it may be helpful in determining the presence of mold on the surface of wood and gypsum boards. However, as they emphasized, this method requires further research consisting of determinations of possible limitations, reliability in the case of various species of both fungi and wood and possible directions of application.

The average values of the wood chips mass before inoculation and after the incubation process were compared. Based on the results of t-Student test, it was found that there were no statistically significant differences between the average mass before inoculation and average mass after incubation (p-value > 0.05). The incubation time had no effect on the obtained results. The percentages of mass loss are shown in Figure 4. Based on the presented average percentage values, it cannot be concluded that the growth of mold fungi caused a mass loss of wood chips. The ML of approx. 0.05 % most likely resulted from an error in the measurement method. Moreover, based on the Fisher test, it was found that there were no statistically significant differences between the variants (p-value > 0.05). Thus, the incubation time had no effect on the percentage mass loss. Mass loss in wood degraded by decaying fungi results from the decomposition of cell wall, which is caused by the decomposition of its structural components – carbohydrate substances such as cellulose or hemicelluloses and lignin. However, FTIR spectra did not indicate any significant changes in the content of these substances. Idler *et al.* (2019) confirmed that during the storage of fragmented wood, mold fungi are not a direct cause of mass loss due to the non-structural substances used for development. However, according to the authors, they may contribute indirectly to the activity of decaying microorganisms through a synergistic effect.

Figure 5 shows changes in moisture content during the exposure of homogenized wood to increased RH (90-95 %).

The course of changes in *MC* was very similar regardless of the wood chips variant. After 120 hours of exposure to high RH, wood reached *MC* of approx. 28 %, which is close to the fiber saturation point (FSP). According to Krzysik (1978), FSP for pine wood is 29 %. A very similar level of *MC* after 120 h was observed for homogenized, unmodified oak wood (Siuda 2019). In order to confirm that the time of incubation had no effect on the changes in *MC*, ANOVA was carried out. Based on the analysis of homogenous groups deter-

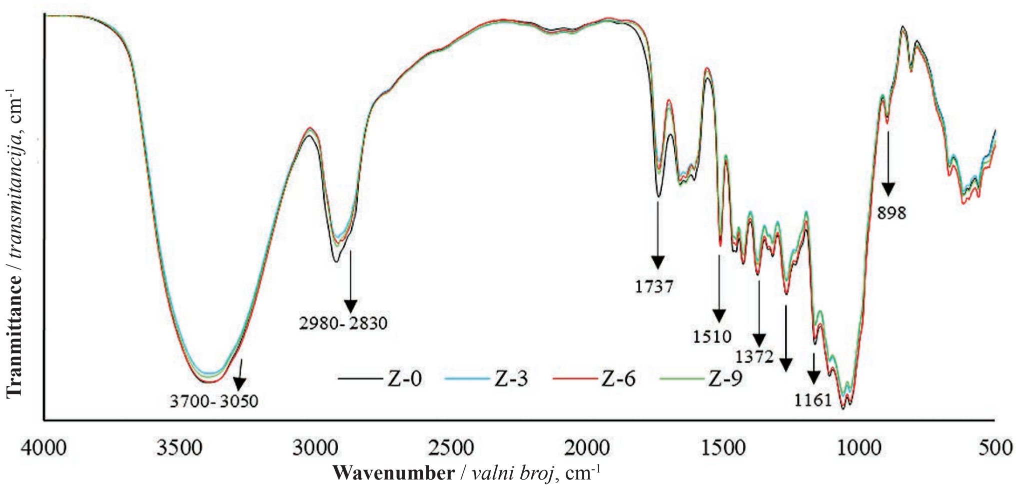

Figure 3 Course of FTIR spectra of wood chips depending on variant
Slika 3. FTIR spektri drvne sječke ovisno o njezinoj varijanti

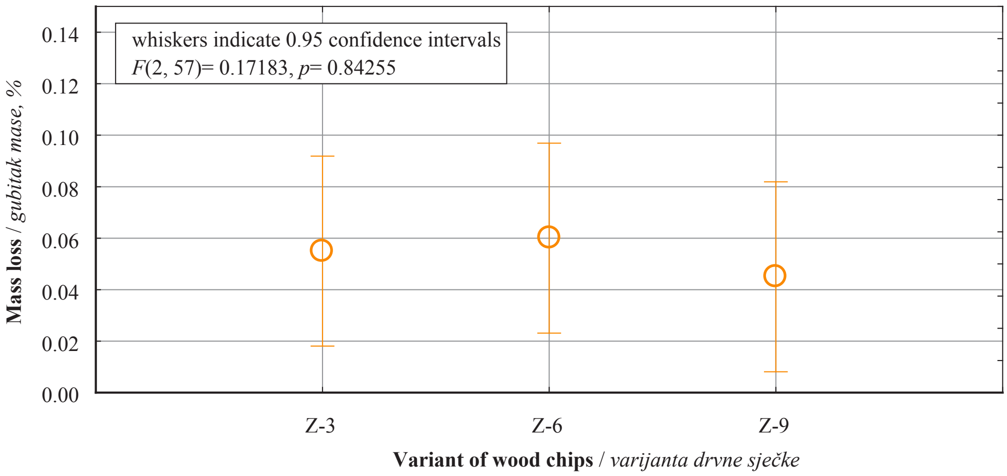

Figure 4 Percentage mass loss depending on the variant of wood chips
Slika 4. Postotak gubitka mase ovisno o varijanti drvne sječke

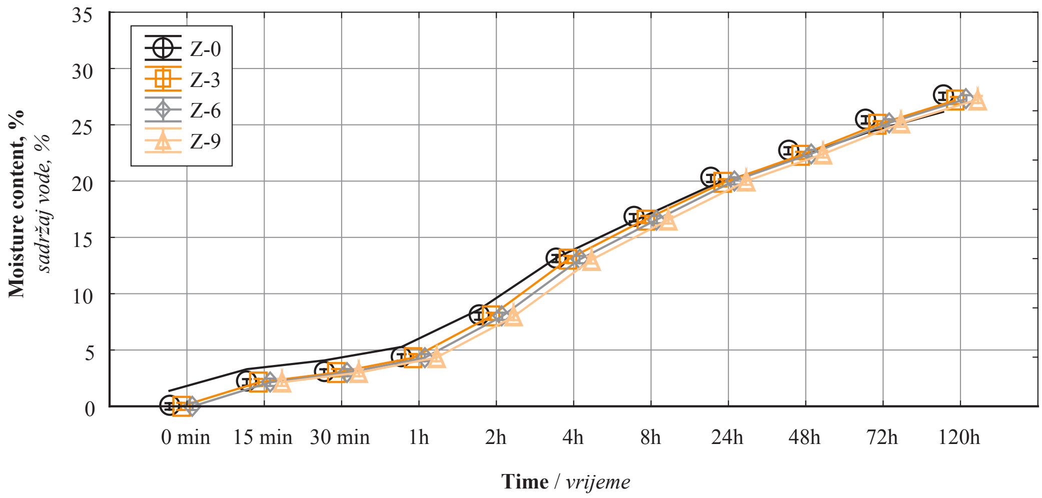

Figure 5 Changes in wood moisture content depending on wood chips variant
Slika 5. Promjene sadržaja vode u drvu ovisno o varijanti drvne sječke

mined in the HSD Tukey test, it was found that there were no statistically significant differences in *MC* between the variants for all measurement times. Thus, the incubation time and the development of molds had no effect on the hygroscopicity of wood. Therefore, the effect described by Rowell (2005) and Clausen (2010), who found that wood covered with mold fungi may be characterized by increased water absorption, was not observed. However, their development is not accompanied by deep penetration into the anatomical structures of wood. Taking into account that, in the case of advanced development of mold fungi, the hyphae may move through the pits degrading their membrane, the increase in absorbency may only occur in the surface layer of the infested material (Nilsson, 2009).

4 CONCLUSIONS
4. ZAKLJUČAK

The present research investigated the effect of wood chips inoculation with *Aspergillus* and *Penicillium* fungi on their selected properties. The results of visual assessment and ergosterol content have shown differentiation in infestation due to the length of the incubation period. The results of FTIR analysis, mass loss and hygroscopicity determinations show no changes caused by a surface infestation with molds. Therefore, pine wood chips, which are commonly overgrown by molds, especially during storage when they are freshly harvested, can be considered as a valuable material that can potentially find alternative applications, other than for energy purposes. In the future, research concerning the use of this type of material will be conducted in various ways.

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chips exposed to mold growth in a chipboard manufacturing process", and the paper contains the same data.

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Optimization of Production Parameters of Densified Laminated Veneer Lumber Produced by Using Urea-Formaldehyde Resin

Optimizacija parametara proizvodnje ugušćene lamelirane furnirske građe proizvedene upotrebom urea-formaldehidne smole

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ABSTRACT • *This research aims to optimize densified laminated veneer lumber production parameters of compression ratio, press temperature, press time, and adhesive spread rate to maximize their mechanical properties. In the manufacturing process of densified laminated veneer lumber, I-77/51 American poplar clone (Populus deltoides) veneers and urea formaldehyde adhesive are used. The results showed that the compression rate and press time had the most significant impact on the mechanical properties of densified laminated veneer lumber. The optimal production conditions were determined as follows: 38 % compression, press temperature of 170 °C, press time of (10±3) minutes, and spread rate of 150 g/m^2. Modulus of rupture, modulus of elasticity, tensile shear strength, and tensile strength perpendicular to panels surface of densified laminated veneer lumbers produced under these conditions increased by 49 %, 8 %, 71 %, and 23 %, respectively, compared to the control group of laminated veneer lumber. So, it can be said that the production parameters of densified laminated veneer lumbers can be optimized safely and effectively using Taguchi method-based grey relational analysis.*

KEYWORDS: *densification; laminated veneer lumber; urea formaldehyde; processing parameters; Taguchi method based grey relational analysis*

SAŽETAK • *Cilj ovog istraživanja bilo je optimiziranje parametara proizvodnje ugušćene lamelirane furnirske građe: stupnja ugušćenja, temperature prešanja, vremena prešanja i količine nanosa ljepila kako bi se povećala mehanička svojstva lamelirane furnirske građe. U procesu proizvodnje lamelirane furnirske građe upotrijebljeni su furniri drva klona američke topole I-77/51 (Populus deltoides) i urea-formaldehidno ljepilo. Rezultati su pokazali da su stupanj ugušćenja i vrijeme prešanja imali najveći utjecaj na mehanička svojstva ugušćene lamelirane furnirske građe. Utvrđeni su ovi optimalni uvjeti: stupanj ugušćenja 38 %, temperatura prešanja 170 °C, vrijeme prešanja 10±3 min i količina nanosa ljepila 150 g/m^2. Modul loma, modul elastičnosti, smična čvrstoća i vlačna*

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čvrstoća okomito na površinu ploče ugušćene lamelirane furnirske građe proizvedene uz navedene uvjete povećali su se za 49 %, 8 %, 71 % odnosno za 23 % u usporedbi s kontrolnom skupinom lamelirane furnirske građe. Dakle, može se reći da se parametri proizvodnje ugušćene lamelirane furnirske građe mogu sigurno i učinkovito optimizirati uz pomoć sive relacijske analize utemeljene na Taguchijevoj metodi.

KLJUČNE RIJEČI: *ugušćivanje; lamelirana furnirska građa; urea-formaldehid; parametri procesa; siva relacijska analiza utemeljena na Taguchijevoj metodi*

1 INTRODUCTION
1. UVOD

The mechanical properties of wood, such as strength, hardness, and abrasion, can be improved by densification due to the compression perpendicular to grain under high temperature and pressure (Haller and Wehsener, 2004; Kollmann *et al*., 1975; Seborg *et al*., 1956; Ülker and Burdurlu, 2016). Thus, it is possible to use the wood of fast-growing species with low density and low mechanical properties as structural material instead of high-value wood (Kutnar and Sernek, 2007). Wood surface roughness is also reduced by densification, which provides lower press time, press pressure, press temperature, and less glue, especially in the production of laminated materials such as plywood and laminated veneer lumber (LVL) (Bekhta and Marutzky, 2007; Bekhta *et al*., 2012; Bekhta and Salca, 2018; Bekhta *et al*., 2018; Fang *et al*., 2012; Ugovšek *et al*., 2013; Ülker and Burdurlu, 2016).

In the literature, there are many studies on the production of laminated materials such as plywood and LVL from pre-densified veneer (Bekhta and Marutzky, 2007; Bekhta *et al*., 2012; Bekhta and Salca, 2018; Bekhta *et al*., 2018; Ugovšek *et al*., 2013; Ülker and Burdurlu, 2016). However, the densification of veneer as a separate step before lamination increases production time and cost.

Both densification and lamination require high press temperature and pressure. So, the production of laminated material by combining the densification and lamination in the same step may provide significant cost advantages. However, the vapor pressure, caused by the water in the adhesive, and wood moisture create the risk of blistering and blowing. A higher temperature (more than 100 °C) and pressure, which are needed for softening of wood, increase these risks even more (Wang and Dai, 2005). In contrast, spring-back and dimensional stability improve with increasing condensation temperature and time (Fang *et. al*., 2012; Kúdela *et al*., 2018). Therefore, it is necessary to apply the press pressure, press temperature, press time, and adhesive spread rate (SR) more carefully, if densification and lamination are used in the same step.

The Taguchi method is a powerful analysis method that reduces the number of experiments using orthogonal arrays to optimize production parameters. Several studies have used the Taguchi method to optimize the parameters such as press pressure, temperature, time, and adhesive amount in the production of composite materials. (Alade *et al*., 2022a; Alade *et al*., 2022b; Buddi *et al*., 2018; Hamzaçebi, 2016; Shafie and Zarea-Hosseinabadi, 2019).

Taguchi method allows for determining the effect of production parameters and optimizing them on a single product feature. However, it is often insufficient to evaluate the quality of a product by adhering to a single feature. In such cases, multi-response optimization techniques are used. In several studies, the Taguchi method and grey relational analysis (GRA) were successfully used together to determine the optimal production conditions for multiple quality features (Gupta *et al*., 2019; Kavimani *et al*., 2022; Khan *et al*., 2021; Kopparthi *et al*., 2021; Onyekwere *et al*., 2021; Velmurugan and Babu, 2020).

In this study, the effects of compression ratio, press temperature, press time, and adhesive spread rate on the mechanical properties of densified laminated veneer lumber (dLVL) were investigated. In this context, the parameter levels were determined using Taguchi-based GRA to maximize the modulus of rupture (*MOR*), modulus of elasticity (*MOE*), tensile shear strength (*TSS*), and tensile strength perpendicular to panel surface (*TSPS*) of dLVL.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

The veneers to produce dLVLs were peeled from I-77/51 American poplar clone (*Populus deltoides*) logged at an altitude of 250 m in Düzce, Turkey. American poplar wood was chosen as wood of low value with lower mechanical properties (Candan *et al*., 2013) and with great potential of improvement by densification. The veneers were peeled in thicknesses of 1.8, 2.1, and 2.4 mm to produce dLVLs and 1.2 mm to produce LVLs for the control group. They were cut in dimensions 55 cm × 30 cm, conditioned at (20±3) °C and (65±1) % relative humidity until they reached a constant weight. Their equilibrium moisture content reached approximately 13 % before the production of panels. A commercial urea-formaldehyde (UF) adhesive (Poliure 2265, Polisan Kimya San. A.Ş., Kocaeli, Turkey) was used in the production of dLVLs. 10 % by weight hard-

Table 1 Production parameters of dLVLs and control group (non-densified) LVL
Tablica 1. Parametri proizvodnje dLVL-a i LVL-a kontrolne skupine (neugušćeno)

Parameters / *Parametri*		Level 1	Level 2	Level 3	Control
Compression rate / *Stupanj ugušćenja,* %		38	47	54	7*
Press temperature / *Temperatura prešanja,* °C		130	150	170	110
Press time, min *Vrijeme prešanja,* min	under 0.8 N/mm² / *pod tlakom 0,8* N/mm²	10	10	10	10
	under increased pressure / *pod povećanim tlakom*	3	5	7	-
Spread rate of adhesive, g/m² / *Količina nanosa ljepila,* g/m²		120	150	180	150

*Compression occurred at press pressure of 0.8 N/mm². / *Ugušćenje je postignuto primjenom tlaka prešanja od 0,8* N/mm².

ener (10 % NH_4Cl/water solution) and 20 % wheat flour by weight were added to prepare UF adhesive solution.

Equilibrium moisture content and air-dry density (*d*) were measured according to standards TS EN 322 (1999) and TS EN 323 (1999), respectively. The spring-back ratios (*SB*) of the produced test panels were calculated according to Eq. 1, respectively:

$$SB = \frac{t_{\mathrm{dLVL}} - t_{\mathrm{ms}}}{t_{\mathrm{ms}}} \cdot 100 \qquad (1)$$

Where t_{dLVL} is the thickness of dLVL panels in air-dry condition, t_{ms} is the thickness of panels compressed state, i.e., of a mechanical stop.

MOR and *MOE* were determined according to TS EN 310 (1999), while *TSS* was determined according to TS EN 314-1 (1998) standard. For each experimental group, five samples were tested. The *TSPS* was determined according to the principles specified in TS EN 319 (1999) standard, and three samples for each experimental group were tested. The universal test machine with 50 kN capacity (UTEST 7012) was used to determine these mechanical properties of dLVLs.

2.1 Experimental design
2.1. Postavke eksperimenta

Parameter design is the first step of the Taguchi method. Here, the parameters that affect product quality and their levels are determined. As a result of the literature review and preliminary experiments, four production parameters, compression rate (*CR*), press temperature (*PT*), press time (*Pt*), and spread rate of adhesive (*SR*), and three levels of each suitable to L9 Taguchi orthogonal array were determined (Table 2). Parameter levels are determined by the low-normal-high principles. In addition, to compare the dLVLs, control group (non-densified) LVL panels were produced in conditions shown in Table 1.

2.2 Production of dLVLs
2.2. Proizvodnja ugušćene lamelirane drvne građe

The UF adhesive was applied to one side of the veneers of various thicknesses (1.8, 2.1 and 2.4 mm) to create nine layers of dLVL drafts with 16.2, 18.9, and 21.6 mm thicknesses, respectively. In the same way, 10.8 mm thick control group LVL drafts were created from 1.2 mm thick veneers. There are four main stages in the dLVL pressing process, as shown in Figure 1. Initially, the prepared drafts were cold-pressed under 0.5 N/mm² pressure for 1 minute. Subsequently, the panels were placed in the laboratory hot press with a capacity of 180 tons (Cemil Usta SSP 180 T) and pressed under a pressure of 0.8 N/mm² for 10 minutes to reach the desired temperature of all veneer sheets. In the compression stage, the press pressure was increased

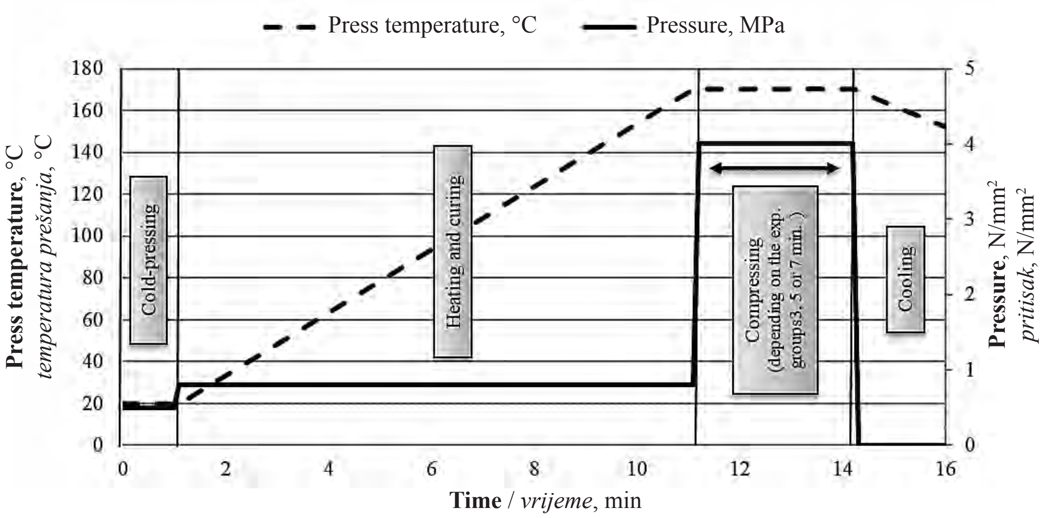

Figure 1 Schedule of dLVL pressing process
Slika 1. Raspored procesa prešanja ugušćene lamelirane drvne građe

to 4 N/mm^2 and the panels were brought to 10 mm. To ensure that all panels produced are of the same thickness, 10mm thick mechanical stops made of stainless steel were used. Thus, dLVLs were compressed at the rates of 38 %, 47 %, and 54 %. The compression step was not applied in the production of the control group LVL. The production parameters of the different experimental groups are presented in Table 2.

2.3 Single and multi-response optimization
2.3. Optimizacija s jednim i više odgovora

In this study, single responses are optimized by the Taguchi method, in which the functions called signal-to-noise ratio (*S/N*) are used and expressed in decibels (dB). The *S/N* for the *SB* was calculated according to the "smaller is better" principle using Eq. 2. In contrast, density, modulus of rupture (*MOR*), modulus of elasticity (*MOE*), tensile shear strength (*TSS*), and tensile strength perpendicular to panels surface (*TSPS*) were calculated according to the "larger is better" principle using Eq. 3, where $S/N_i(k)$: *S/N* of the i*th* experimental group for k*th* mechanical property, $y_i(k)$: test result of j*th* repetition for k*th* mechanical property, n: number of repetitions.

$$\left(S/N\right)_i(k) = -10\log_{10}\left[\frac{1}{n}\sum_{j=1}^{n} y_j(k)^2\right] \tag{2}$$

$$\left(S/N\right)_i(k) = -10\log_{10}\left[\frac{1}{n}\sum_{j=1}^{n}\frac{1}{y_j(k)^2}\right] \tag{3}$$

In both cases, the highest *S/N* calculated for each experimental group is considered as the best result. In addition, the contribution rates (*Cr*) and significance levels of the production parameters on mechanical properties were determined using analysis of variance (ANOVA) at a 95 % confidence interval. Taguchi-based experimental design and statistical analysis were performed using Minitab 18 software.

GRA was used to obtain optimal process parameters to maximize *MOR*, *MOE*, *TSS,* and *TSPS* at the same time. GRA was applied according to the following steps:

First, the results of *MOR*, *MOE*, *TSS*, and *TSPS* were normalized ($P_i(k)$) between the ranges from 0 to 1 to reduce the variability. The data are calculated according to Eq. 4.

$$P_i(k) = \frac{y_i(k) - \min y(k)}{\max y(k) - \min y(k)} \tag{4}$$

Then, the grey relational coefficient (ε_i) was calculated according to Eq. 5, where $\Delta_0(k)$ is the absolute difference between $P_i(k)$ and $P_i(k)$, $\Delta_{min}(k)$ and $\Delta_{max}(k)$ are the minimum and maximum value of absolute difference between $P_i(k)_{max}$ and $P_i(k)$. ξ is the distinguishing coefficient and commonly assumed to be 0.5 (Kavimani *et al*., 2022; Kopparthi *et al*., 2021; Onyekwere *et al*., 2021; Velmurugan and Babu, 2020).

$$\varepsilon_i(k) = \frac{\Delta_{min}(k) + \xi\Delta_{max}(k)}{\Delta_0(k) + \xi\Delta_{max}(k)} \tag{5}$$

In the fourth step, the grey relational degree (*GRD*) was calculated by summing the weighted grey relational coefficients (Eq. 6) and ranked as the largest best. In this study, equal weights *r(k)* are given for *MOR*, *MOE*, *TSS*, and *TSPS*.

$$GRD = \sum_{k=1}^{n}\frac{1}{r(k)}\varepsilon_i(k) \tag{6}$$

The grey relational degree (GRD) is a representation of the results of *MOR*, *MOE*, *TSS*, and *TSPS* as a single value. By using these values, Taguchi analysis was performed again to determine optimum process conditions. The dLVL for the confirmation test was produced in these optimum conditions. Finally, the mechanical properties of this dLVL and the control LVL produced under the standard conditions applied in industry were compared.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

The air-dry density of the I-77/51 American poplar clone wood measured as 0.49 g/cm^3. The air-dry density, *DR*, *SB, MOR*, *MOE*, *TSS*, and *TSPS* of dLVLs and control group LVL are given in Table 2.

The densities of the dLVLs increased by 31 % to 43 % compared to American poplar wood and 17 % to 28 % compared to the control group LVL. When the response table for the *S/N* (Table 3a) is examined, it is seen that the most effective production parameter on density is the *CR* (45 %), as expected. However, it was observed that the increase in *CR* did not cause a significant increase in the densities of dLVLs (Figure 2a). This can be attributed to the increase in the *SB* of dLVLs, as the *CR* increases, which has the greatest effect on *SB* (Table 3b and Figure 2b). The higher *CR* increases internal stresses and causes an increase in the *SB* (Blomberg *et al*., 2006; Kutnar *et al*., 2009; Laine *et al*., 2016; Unsal *et al*., 2011). Additionally, the water in the adhesive evaporates due to the high temperature applied in pressing of dLVLs. As a result of the increased press pressure required to increase the density, this water vapor may be trapped in dLVL, which causes increased steam pressure. Therefore, the dLVLs may have tended to return to their original thickness and increased the *SB* when the press was opened. On the other hand, as the *SR* increased, an increase was observed in the density of dLVLs, because the used adhesive filled the cell lumens (Figure 2a).

Table 2 Experimental results of produced dLVLs and control group LVL
Tablica 2. Eksperimentalni rezultati proizvedene ugušćene lamelirane drvne građe i kontrolne skupine lamelirane drvne građe

Exp. Group	*CR*, %	*PT*, °C	*Pt*, min	*SR*, g/m²	*d*, g/cm³	*SB*, %	*MOR*	*MOE*	*TSS*	*TSPS*
							N/mm²			
1	38	130	10+3	130	0.68	9	122.56	14122	4.15	1.25
2	38	150	10+5	180	0.68	14	103.52	11476	6.77	1.47
3	38	170	10+7	150	0.70	9	104.11	11926	5.39	1.22
4	47	130	10+5	150	0.69	43	88.06	10429	5.25	1.51
5	47	150	10+7	130	0.69	38	97.52	10425	4.68	1.02
6	47	170	10+3	180	0.67	37	119.66	10821	5.23	1.51
7	54	130	10+7	180	0.67	45	78.97	7722	5.63	1.21
8	54	150	10+3	150	0.68	53	83.87	9391	5.33	1.27
9	54	170	10+5	130	0.64	43	71.16	6362	8.07	0.85
Control *Kontrolni uzorak*	7	110	10	150	0.55	1	82.77	10222	5.50	1.15

Table 3 Response tables for *S/N* of *d* (a), *SB* (b), *MOR* (c), *MOE* (d), *TSS* (e), and *TSPS* (f)
Tablica 3. Tablice odgovora za *S/N* od d (a), *SB* (b), *MOR* (c), *MOE* (d), *TSS* (e) i *TSPS* (f)

a)

Level	*CR*	*PT*	*Pt*	*SR*
1	-3.24	-3.31	-3.38	-3.46
2	-3.30	-3.33	-3.45	-3.43
3	-3.55	-3.45	-3.26	-3.20
Delta	0.32	0.14	0.19	0.29
Rank	1	4	3	2
Cr	45%	8%	14%	33%

Signal to noise: larger is better
Omjer signal/šum: veći je bolji

b)

Level	*CR*	*PT*	*Pt*	*SR*
1	19.79	11.87	11.82	12.36
2	8.17	10.26	10.47	10.77
3	6.53	12.36	12.20	11.35
Delta	13.26	2.11	1.72	1.59
Rank	1	2	3	4
Cr	96%	2%	1%	1%

Signal to noise: larger is better
Omjer signal/šum: veći je bolji

c)

Level	*CR*	*PT*	*Pt*	*SR*
1	41.07	39.80	40.86	39.75
2	39.99	39.44	38.71	39.90
3	37.80	39.61	39.28	39.20
Delta	3.27	0.36	2.15	0.70
Rank	1	4	2	3
Cr	66%	1%	28%	5%

Signal to noise: larger is better
Omjer signal/šum: veći je bolji

d)

Level	*CR*	*PT*	*Pt*	*SR*
1	82.14	80.57	81.25	79.96
2	80.34	80.22	79.16	79.85
3	77.70	79.39	79.77	80.38
Delta	4.45	1.17	2.10	0.53
Rank	1	3	2	4
Cr	79%	4%	16%	1%

Signal to noise: larger is better
Omjer signal/šum: veći je bolji

e)

Level	*CR*	*PT*	*Pt*	*SR*
1	14.02	13.81	13.68	14.36
2	13.99	14.55	15.87	14.98
3	15.55	15.21	14.01	14.23
Delta	1.56	1.40	2.20	0.75
Rank	2	3	1	4
Cr	24%	21%	51%	4%

Signal to noise: larger is better
Omjer signal/šum: veći je bolji

f)

Level	*CR*	*PT*	*Pt*	*SR*
1	2.13	2.83	2.41	0.11
2	2.33	1.67	1.68	3.24
3	1.31	1.28	1.68	2.42
Delta	1.02	1.55	0.72	3.13
Rank	3	2	4	1
Cr	25%	6%	14%	55%

Signal to noise: larger is better
Omjer signal/šum: veći je bolji

As seen in the response tables for the *S/N*, the most effective production parameters on *MOR* and *MOE* are *CR* and press time, respectively. While the contribution rates of *CR* and press time on *MOR* were 66 % and 28 %, those on *MOE* were 79 % and 16 %, respectively (Table 3c and 3d). The *MOR* and *MOE* values unexpectedly decreased due to the increase in *CR* (Figure 2c and 2d), although an increase in mechanical properties was expected (Kutnar and Sernek, 2007; Pelit *et al*., 2018; Yu *et al*., 2017). As a result, it is predicted that the highest *MOR* and *MOE* values will be obtained in dLVLs that are compressed at 38 % and pressed for (10±3) minutes.

As seen in Table 3e, the most effective production parameter on *TSS* was press time (51 %), followed by *CR* (24 %), press temperature (21 %), and *SR* (4 %). As known, it is possible to use lower press temperature, pressure, shorter pressing time, and less adhesive,

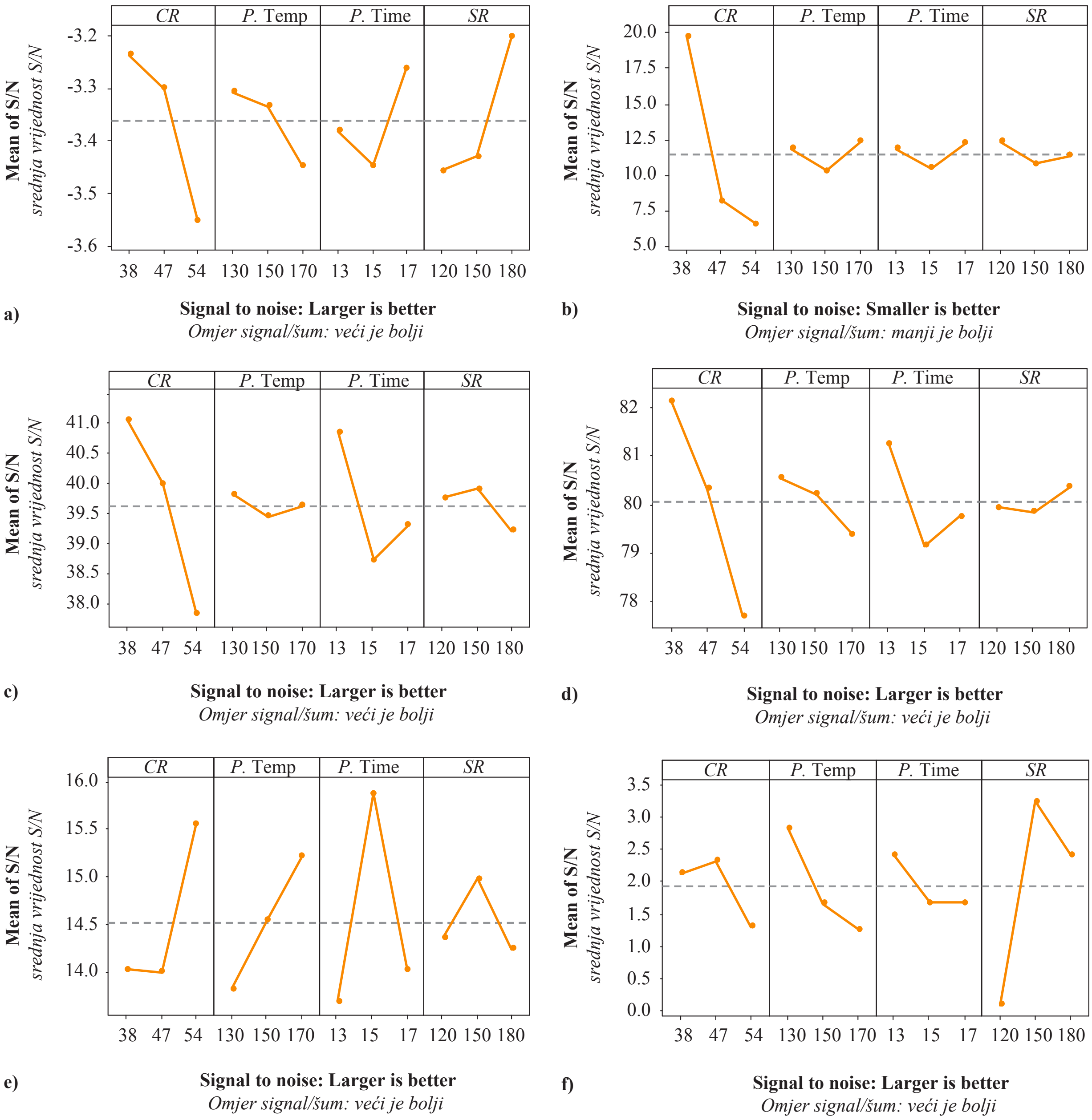

Figure 2 Main effect plots for *S/N* of d (a), *SB* (b), *MOR* (c), *MOE* (d), *TSS* (e), and *TSPS* (f)
Slika 2. Dijagrami glavnog učinka za *S/N* od d (a), *SB* (b), *MOR* (c), *MOE* (d), *TSS* (e) i *TSPS* (f)

as well as to increase mechanical properties by using densified veneer in the production of laminated materials (Bekhta and Marutzky, 2007; Bekhta *et al*., 2012; Bekhta and Salca, 2018). When the main effect plot for the *S/N* was examined, it was found that the highest *TSS* was obtained by dLVL densified by the highest compression rate of 54 % and at a temperature of 170 °C for (10±5) minutes (Figure 2e).

When the response table for the *S/N* (Table 3f) is examined, it can be seen that the most effective production parameters on the *TSPS* were the SR (55 %) and compression ratio (25 %), which made a statistically significant difference in *TSPS*. During the peeling process, cracks on the loose side of the veneer in the direction parallel to the fibers (lathe check) are inevitable (Huang, 2010). Furthermore, depending on the conditions, plastic deformation occurs during the densification, the cell lumens collapse, and the cell fractures develop (Bao *et al*., 2017; Blomberg and Persson, 2004). For these reasons, a decrease in *TSPS* can be expected. However, the adhesive used may have penetrated between these cracks and collapsed cell lumens, allowing them to repair. So, the highest *TSPS* was obtained at high SRs of 150 and 180 g/m^2, *CR* of 47 %, press temperature of 130 °C, and press time of (10±3) minutes (Figure 2f).

3.1 Multi-response optimization
3.1. Višestruka optimizacija mehaničkih svojstava

In this study, GRA, as a multi-response optimization method, was used to determine the optimum production conditions for *MOR*, *MOE*, *TSS*, and *TSPS* si-

Table 4 Calculated grey relational degree
Tablica 4. Izračunani sivi relacijski stupanj

Exp. Gr.	GRD	Orders
1	0.722	1
2	0.662	3
3	0.544	5
4	0.587	4
5	0.446	8
6	0.711	2
7	0.429	9
8	0.459	7
9	0.500	6

Table 5 Response tables for *S/N* of GRD
Tablica 5. Tablice odgovora za *S/N* GRD-a

Level	*CR*	*PT*	*Pt*	*SR*
1	-3.91	-4.93	-4.19	-5.29
2	-4.87	-5.78	-4.74	-4.63
3	-6.71	-4.76	-6.55	-5.56
Delta	2.80	1.02	2.37	0.93
Rank	1	3	2	4
Cr	48%	7%	38%	7%

*Signal to noise: larger is better / *Omjer signala i šuma: veći je bolji*

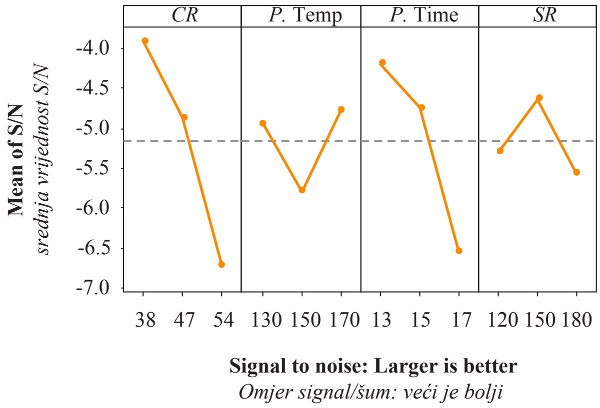

Figure 3 Main effect plots for *S/N* of GRD
Slika 3. Dijagrami glavnog učinka za *S/N* GRD-a

multaneously. The grey relational degrees (GRD) and the orders are shown in Table 4.

MOR, *MOE*, *TSS*, and *TSPS* were converted into a single response (GRD), and the effect of the production parameters on multiple performance was ordered by applying Taguchi method again. According to these results, the *CR* (48 %) and press time (38 %) had the highest effect on multiple performance of dLVLs (Table 5). The mechanical properties decreased with increasing compression ratio and pressing time (Figure 3).

3.2 Confirmation tests
3.2. Potvrdni testovi

At first, dLVL panels for confirmation were produced using optimum production conditions of 38 % compression, 170 °C press temperature, 10+3 minutes press time, and 150 g/m^2 *SR*, and their *MOR*, *MOE*, *TSS*, and *TSPS* were determined. It was found that the *MOR*, *MOE*, *TSS*, and *TSPS* of dLVLs produced under optimum conditions were 123.41, 11059, 9.40, and 1.42 N/mm^2, which is 49, 8, 71, and 23 % higher than control samples manufactured under standard conditions used in industry, respectively. The mechanical properties of dLVLs produced under optimum conditions were also compared with those of laminated materials produced with veneers of higher density, as published in literature (Table 6). As it can be seen, the dLVL may substitute LVL produced from high-value wood species and Taguchi-based GRA allows dLVL production parameters to be optimized safely and efficiently.

4 CONCLUSIONS
4. ZAKLJUČAK

The objective of this study is to determine the most suitable production conditions of compression ratio, press temperature, press time, and adhesive spread rate to maximize the mechanical properties of dLVL. The effects of the production parameters on the mechanical properties of *MOR*, *MOE*, *TSS,* and *TSPS* were also determined by using Taguchi and grey rela-

Table 6 Comparison of confirmation test results with control group and literature
Tablica 6. Usporedba rezultata potvrdnog testa s kontrolnom skupinom i literaturom

Comparison groups / *Grupe za usporedbu*		*MOR*	*MOE*	*TSS*	*TSPS*
		N/mm^2			
dLVL produced in optimum production conditions *dLVL proizveden u optimalnim proizvodnim uvjetima*		123.41	11059	9.40	1.42
Non-densified LVL (control group) / *neugušćeni LVL (kontrolna skupina)*		82.77	10222	5.50	1.15
Literature review *pregled literature*	Poplar LVL / *LVL od topolovine*	68.14[a]	6690[b]	4.24[c]	
	Beech LVL / *LVL od bukovine*	118.30[a] 95.41[b]	19512[a] 8773[b]	10.90[c]	-
	Beech plywood / *furnirska ploča od bukovine*	-	-	-	1.86[d]
	Eucalyptus LVL / *LVL od drva eukaliptusa*	94.90[a] 89.30[b]	9411[a] 9131[b]	-	-
	Oak LVL / *LVL od hrastovine*	-	-	8.94[c]	-

[a]Aydın *et al*., 2014; [b]Bal and Bektaş, 2012; [c]Uysal, 2006; [d]Réh *et al*., 2019

tionship analysis methods to optimize single- and multi-responses.

As *CR* increased, the density of dLVLs unexpectedly decreased due to the increase in *SB*. In addition, it caused an increase in density due to the fact that the adhesive used filled the cell spaces.

It was found that *CR* had the most significant impact on *MOR* and *MOE*, and contrary to expectations, dLVLs with 38 % compression showed the highest values. The most effective production parameters on *TSS* and *TSPS* were found to be press time and *SR*, respectively.

Multi-response optimization using grey relational analysis showed that the *CR* (48 %) and press time (38 %) had the most significant impact on the mechanical properties of dLVL. It has also been determined that the highest results will be obtained when the lowest *CR* of 38 % and the shortest pressing time of (10±3) minutes are applied. This combination may also reduce production costs.

Confirmation tests showed that the mechanical properties of dLVLs produced in optimum production conditions improved by 49 % (*MOR*), 8 % (*MOE*), 71 % (*TSS*), and 23 % (*TSPS*) compared to control group of LVL produced by traditional production method applied in wood product industry. So, it can be said that poplar dLVLs can be used as structural elements and the production parameters of dLVLs can be optimized safely and effectively using Taguchi-based GRA.

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Criterion for Objective Determination of Threshold Value in Filtering Surface Roughness Signal of Solid Wood After Machining with Fast Fourier Transform (FFT) Based Filter

Kriterij za objektivno određivanje vrijednosti praga osjetljivosti pri filtriranju signala hrapavosti strojno obrađene površine masivnog drva filtrom utemeljenim na brzoj Fourierovoj transformaciji (FFT)

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ABSTRACT • *The article presents the method for an objective determination of threshold value, needed for filtering out the anatomical roughness signal, with filter based on fast Fourier Transform (FFT), from surface roughness profile after machining. The method includes experimental preparation of solid wood surface by cutting in such a way to get a surface that can be considered to represent only anatomical roughness, with no other influence. Experiments were performed on radial cross section of solid oak wood (Quercus robur L.) so that the results could be compared with roughness profiles that were previously obtained in experiments after sawing with circular saw. From these samples and based on frequency analysis of anatomical roughness signals, the threshold value was determined to be 1.6 μm. The average value of* R_a *parameter of anatomical roughness for specimens of radial cross section of solid oak wood was 2.1 μm with standard deviation of 0.3 μm. The importance of choosing adequate sampling length in threshold determination, based on frequency analysis of anatomical roughness signal, was also established.*

KEYWORDS: *machined surface roughness; anatomical roughness; circular saw; solid wood; signal analysis; threshold; FFT*

SAŽETAK • *U radu je prikazana metoda za objektivno određivanje vrijednosti praga osjetljivosti potrebnoga za filtriranje signala anatomske hrapavosti filtrom koji se temelji na brzoj Fourierovoj transformaciji (FFT), i to iz*

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profila hrapavosti površine nakon strojne obrade. Metoda obuhvaća eksperimentalnu pripremu površine masivnog drva rezanjem na način da se dobije površina za koju se može smatrati da je samo anatomska hrapavost, bez ikakvih drugih utjecaja. Eksperimenti su izvedeni na radijalnom presjeku masivnog drva hrastovine (Quercus robur L.) kako bi se rezultati mogli usporediti s profilima hrapavosti koji su prethodno dobiveni u eksperimentima piljenja kružnom pilom. Iz tih uzoraka i na temelju frekvencijske analize signala anatomske hrapavosti utvrđeno je da se za vrijednost praga osjetljivosti može uzeti vrijednost od 1,6 μm. Prosječna vrijednost R_a parametra anatomske hrapavosti za uzorke radijalnog presjeka hrastova drva bila je 2,1 μm, sa standardnom devijacijom od 0,3 μm. Također je utvrđena važnost odabira primjerene duljine uzorkovanja pri određivanju praga osjetljivosti na temelju frekvencijske analize signala anatomske hrapavosti.

KLJUČNE RIJEČI: *hrapavost obrađene površine; anatomska hrapavost; kružna pila; masivno drvo; analiza signala; prag osjetljivosti; FFT*

1 INTRODUCTION
1. UVOD

Quantification of wood surface roughness after machining is still a complex problem that is not satisfactorily resolved. In scientific research, quality of machined surface is usually quantified by parameters defined in ISO 4287 (1997), and in industry it is usually based on subjective standards that include visual inspection and sensing of the surface by hand. According to Hendarto *et al*. (2006), the lack of wood surface evaluation methods is mostly caused by the fact that wood roughness also depends on factors related to wood anatomy caused by its nonhomogeneous structure. The problem of determination of appropriate techniques and parameters for the evaluation of surface quality in wood machining is also further complicated by the fact that the resulting measured surface profile after machining is the result of the interaction of workpiece material, machine tool, measuring instrument used for the measurement of surface profile and analysis of surface profile data (Sandak, 2005; Sinn *et al*., 2009). Measuring instruments used for the determination of surface profile can be roughly divided into contact and non-contact instruments. In laboratory measurements, contact instruments are mostly used and their universal characteristics are defined by ISO 3274 (1996). They give more reliable surface profile traces, if the appropriate parameters and stylus tips are used, where ISO 4288 (1996) can be helpful. It must be noted here that, for the assessment of wood surface quality, due to its surface characteristics, recommendations given by Gurau *et al*. (2006) and Gurau and Irle (2017) should be consulted.

For industrial use in on-line surface quality assessment of machined surface of solid wood, due to high passing speeds of workpieces in relation to measurement speeds of contact type surface roughness testers, this type of instruments cannot be used. In industrial on-line control only non-contact type of instruments can be used and, in that area, there are studies on the appropriateness of use of different non-contact measurement methods for the determination of surface profile of solid wood after machining (Lemaster, 1999; Sandak and Tanaka, 2003; Sandak *et al*., 2004; Sandak *et al*., 2020). Although, non-contact type instruments have advantages over contact type instruments in an industrial environment, it should be noted that there will be differences between profiles measured by contact and non-contact methods (Gurau *et al*., 2001; Sandak and Tanaka, 2003).

The analysis of surface profile data is another factor in the assessment of surface quality of wood. As already said, measured surface profile is mostly analyzed by methods and parameters defined in ISO 4287 (1997) and before that, it is filtered with standard filters. Usually, Gaussian filter defined in ISO 16610-21 (2011) is used for filtering out the roughness (or waviness) of the profile, but it has been shown that materials with sharp peaks and valleys pose a problem for standard filtering technique (Mills and Yoshino, 2019). If the standard Gaussian filter is used for filtering the surface profile of large porous wood species, there is evident raising of the roughness profile in the immediate vicinity of the pore edges that affects the evaluation of the surface roughness. It has been found that the Robust Gaussian Regression Filter (RGRF), now proposed by ISO 16610-31 (2010), gives better results (Fujiwara *et al*., 2004; Gurau *et al*., 2006; Sharif and Tan, 2011), but most of the research in evaluating the surface quality of machined surface of solid wood still uses standard Gaussian filters.

If the surface profile of solid wood after machining is adequately measured, the traced signal, which represents surface roughness of wood, is superposition of anatomical roughness of wood and machining roughness, which consists of tool marks left on machined surface and other machining related effects, like chipped or raised grain, fuzziness, etc. (Csanády and Magoss, 2011; Csiha and Krisch, 2000; Gurau, 2019; Lemaster, 2004). It can be hard to distinguish the effect of each component on the overall roughness and, depending on the wood species component, anatomical structure can have a significant impact on the overall roughness (Gottlöber, 2014). According to Gurau *et al*. (2013), the proper evaluation of the quality of a machined wood surface implies that irregularities due to wood anatomy are excluded from the numerical characterization of the surface profile. There are different approaches to removing irregularities due to wood anatomy from surface roughness measurements (Westkämper and Riegel, 1993; Magoss and Sitkei, 1999; Schadoffsky, 2000; Fujiwara *et al*., 2003; Gurau *et al*., 2005; Tan *et al*., 2010).

One way of looking at this problem is to consider the measured surface profile as a signal composed of anatomical roughness signal that represents irregularities due to wood anatomy and which can be represented by a random signal (Lemaster and Taylor, 1999), machining roughness signal, which is a random signal that represents non-periodic effects on machined surface due to machining (chipped grain, fuzziness, etc.) and periodic signals (for most of the wood machining processes) that represent tool marks. Due to different signal characteristics of individual components, different signal processing techniques can be used to try to separate the above-mentioned signal components and evaluate them accordingly.

In previous research by Đukić *et al*. (2022), a simple method for filtering out the periodic signal components due to teeth marks on machined surface after sawing with circular saw was proposed based on filtering with Fast Fourier Transform (FFT). That method includes the use of a threshold value, which is used as a limit below which all the frequency components in the analyzed roughness signal are set to zero. From this filtered signal in frequency domain, which is assumed to satisfactorily describe the periodic signal due to teeth marks in frequency domain, the time domain representation is obtained by Inverse Fast Fourier Transform (IFFT). In this way signals are obtained that, according to our assumptions, represent the part of surface roughness signal (periodic) due to tool marks and the other part (non-periodic, random) that represents anatomical roughness and machining induced roughness (processing roughness).

As was recognized in that research, one of the shortcomings was that the determination of threshold value was subjective (threshold value was determined by trial and error) and after further quantification of obtained signals, by calculating R_a and R_q values for these signals, it was not possible to objectively assess the individual impact of anatomical roughness and machining induced roughness on the overall surface quality.

The main goal of this research was to try to determine the objective method for the determination of threshold value. The idea was to measure the surface profile of solid oak wood (*Quercus robur* L.), which was prepared in such a way that the obtained surface profile could represent the anatomical roughness signal that can be used with previously obtained data from sawing experiments. Then the FFT of that signal can be obtained and threshold value can easily be calculated as the first number greater than maximum value of FFT of an anatomical roughness signal. It is also possible to calculate R_a and R_q values of the anatomical roughness signal and individual impact of machining induced roughness can be evaluated more easily.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

To measure the surface profile of solid oak wood (*Quercus robur* L.) that would represent an anatomical roughness signal representative of the surfaces that are obtained by resawing, and which could be compared with data from previous research (Đukić *et al*., 2022), three samples with average dimensions *L* x *B* x *H* = (500 x 220 x 21) mm were prepared from plainsawn boards. The average moisture content of specimens was 9 %. Surfaces for the determination of surface profile were prepared by cutting on the test device for orthogonal cutting in the laboratory at Biotechnical Faculty, Department of Wood Science and Technology in Ljubljana, Slovenia. The cuts were made in 90°–0° direction (McKenzie, 1961). The resulting texture of these surfaces was a combination of radial and tangential, but it was mostly radial because the samples were prepared from plainsawn boards.

Cutting parameters were set up to obtain Type II - continuous chip (Koch and Woodson, 1970). The cuts were performed with a cutter made from high-speed steel (HS) with W – 18 %, with rake angle $\gamma = 50°$, clearance angle $\alpha = 8°$. Cutting edge radius was around $\rho_0 \cong 4$ µm. The chip thickness was $h = 0.03$ mm and cutting speed $v_c = 0.5$ m/s. Measurement setup can be seen in Figure 1.

Surface roughness was measured with surface roughness tester Mitutoyo SurfTest SJ-500 (Ser. No. B0007 1808). The measurements were done in accordance with ISO 4287 (1997). The stylus tip radius was 10 µm and in accordance with the recommendations in ISO 3274 (1996), the λ_s profile filter cut-off was 25 µm and λ_c profile filter cut-off was 8 mm. Evaluation length was 40 mm. Stylus traversing speed was set to $v_s = 0.1$ mm/s, which then corresponded to spatial resolution of 5 µm between two measurement points. The sampling frequency was $f_s = 20$ Hz, so the maximum analyzed frequency component of the surface roughness signal was 10 Hz. Gaussian filter was used for filtering the *R* profile of roughness signal. Although its filtering disadvantages when used on profiles of wood species with deep valleys have already been mentioned, this type of filter was used in previous research and is still commonly used in most of the scientific research dealing with roughness analysis of solid wood surfaces. The

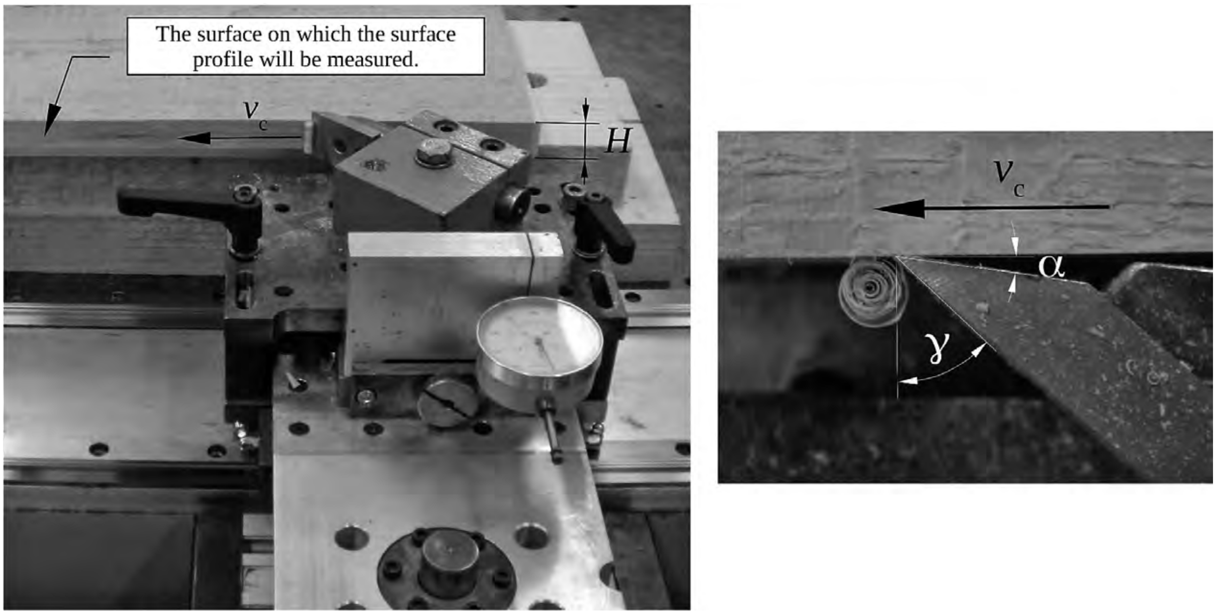


Figure 1 Cutting of test specimens of solid oak wood (*Quercus robur* L.) with average cutting width H = 21 mm in 90° – 0° direction with cutter made from high-speed steel (HS), with rake angle γ = 50°, clearance angle α = 8°, chip thickness h = 0.03 mm and cutting speed v_c = 0.5 m/s to obtain Type II - continuous chip
Slika 1. Rezanje ispitnih uzoraka od hrastovine (*Quercus robur* L.) na prosječnu širinu rezanja H = 21 mm u smjeru 90 – 0° oštricom od brzoreznog čelika (HS) te s prednjim kutom oštrice γ = 50°, stražnjim kutom oštrice α = 8°, debljinom odvajane strugotine h = 0,03 mm i brzinom rezanja v_c = 0,5 m/s kako bi se dobila strugotina tipa II (kontinuirana strugotina)

use of Robust Gaussian Regression Filter (RGRF) would be preferred here because the measurements were performed on solid oak wood that has deep valleys due to its anatomical characteristics, and raising of filtered profile in the immediate vicinity of wood pores is evident from measured profiles (Figure 2). The influence of distortions of filtered roughness profile introduced by using a Gaussian filter is assumed to represent systematic error in the evaluation of surface roughness parameters and it is assumed that on average it will affect all measured profiles in the same way, so the obtained parameter values can be compared. If the primary task is the accurate evaluation of the surface shape, then the use of RGRF filter is recommended.

Surface roughness measurement parameters were chosen to be the same as in previous research (Đukić *et al*., 2022), so that the results could be compared. Also, the recommendations specific to wood surface roughness evaluation, given by Gurau *et al*. (2006) and Gurau and Irle (2017), were considered.

Before the measurements, the roughness tester was calibrated with a working gauge that provides reference roughness profile with R_a = 2.97 μm (Mitutoyo, Ser. No. 393041807).

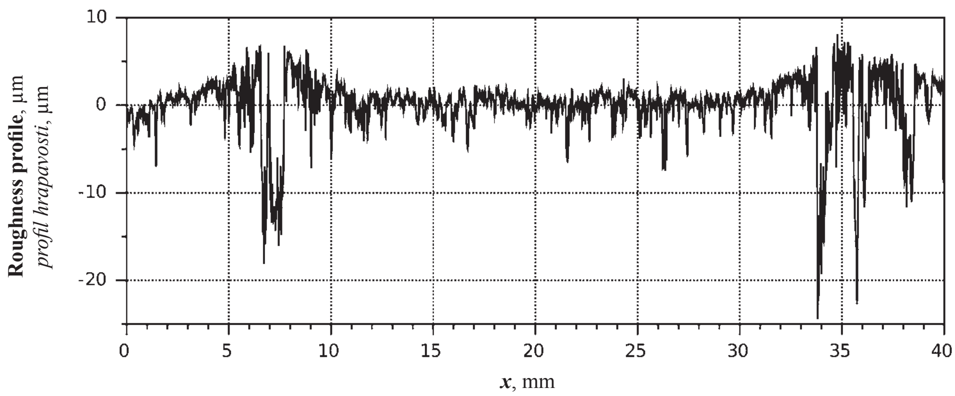


Figure 2 Anatomical surface roughness profile (R profile) sample of radial cross-section of solid oak wood (*Quercus robur* L.) measured with surface roughness tester Mitutoyo SurfTest SJ-500, with the λs = 25 µm, λc = 8 mm, evaluation length 40 mm, stylus traversing speed v_s = 0.1 mm/s and filtered with Gaussian filter
Slika 2. Profil anatomske hrapavosti površine (R-profil) uzorka radijalnog presjeka hrastovine (*Quercus robur* L.) izmjeren profilometrom Mitutoyo SurfTest SJ-500, s λs = 25 µm, λc = 8 mm, ocjenske duljine 40 mm, brzine pomicanja ticala v_s = 0,1 mm/s i filtrirano Gaussovim filtrom

During measurements, the stylus tip traversed the machined surface in the direction that would correspond to the direction of the feed movement vector (v_f) if these boards were to be sawn with circular saw along the grain with cutting height (H). It can be seen from Fig. 1 that the assumed direction of feed speed in sawing matches the direction of cutting speed vector (v_c) in preparation of the samples for the determination of anatomical roughness, which was conducted on the test device for orthogonal cutting.

On every test board, five measurements were taken on randomly selected measurement locations, which included wood pores. Measured surface profiles obtained in such a way were representative of the analyzed wood surfaces. In total fifteen roughness profiles with a total evaluation length of 600 mm were measured. Measured roughness profiles were exported to text files for further processing. For further analysis, scripts were written in Scilab software (https://www.scilab.org).

For every sample of measured anatomical roughness, signal R_a parameter was calculated as

$$R_a = \frac{1}{N} \cdot \sum_{i=1}^{N} |y_{ri}|$$

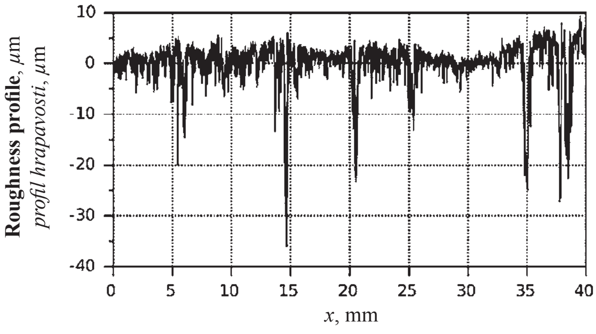
Where N is the number of samples in measurement and y_{ri} are the individual values of measured roughness in the given board sample.

Before frequency analysis, measured roughness profile signals were transformed from spatial to time domain, so that the results of frequency analysis could be obtained as a function of frequency instead as a function of wavelength.

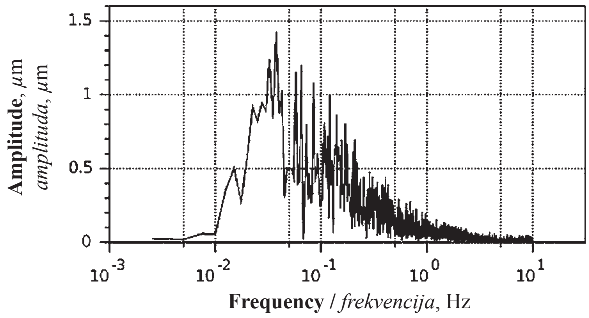
As mentioned above, stylus traversing speed was set to v_s = 0.1 mm/s and corresponding spatial resolution was 5 µm. Time difference between the two samples was s.

For every measured roughness profile, Fourier transform was obtained with fast Fourier transform and graphically presented (Figure 3). Scilab software uses FFTW open-source C subroutine library for computing the discrete Fourier transform (https://www.fftw.org). Frequency resolution was 2.5 mHz.

After all the frequency spectra of individual samples were obtained, the highest recorded amplitude value was recorded. According to the procedure for filtering of machined surface roughness signal with FFT based filter (Đukić *et al.*, 2022), the threshold value needed for filtering was set to the first higher number rounded to 0.1 µm. From the analyzed frequency spectra, the value of threshold was set to 1.6 µm (Figure 4). All the frequency components of analyzed frequency spectra from machined surface roughness signal after sawing with circular saw below this value are assumed to be due to anatomical roughness. This is not exactly



a)



b)

Figure 3 Sample of a) anatomical surface roughness profile (R profile) measured on radial cross-section of solid oak wood (*Quercus robur* L.) and b) frequency analysis of that signal

Slika 3. a) Uzorak profila anatomske hrapavosti površine (R-profil) izmjerenoga na radijalnom presjeku hrastovine (*Quercus robur* L.), b) frekvencijska analiza tog signala

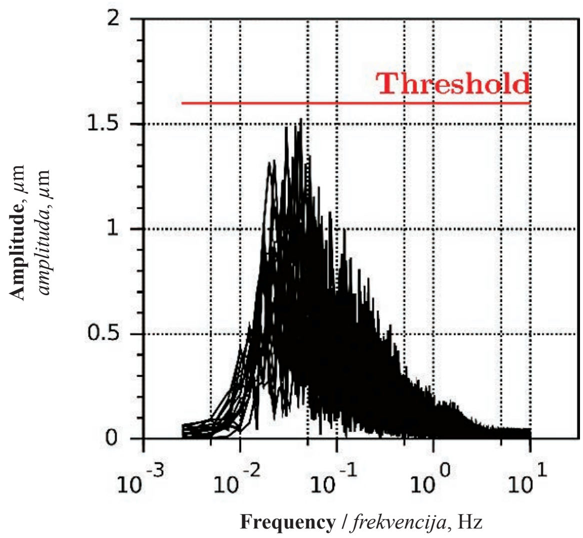


Figure 4 Frequency spectra of measured anatomical surface roughness signals and threshold used for further signal processing of surface roughness signals obtained from measurements on machined surface after sawing with circular saw

Slika 4. Frekvencijski spektri svih izmjerenih signala anatomske hrapavosti površine i prag osjetljivosti koji je služio za daljnju obradu signala hrapavosti dobivenih iz mjerenja na obrađenoj površini nakon piljenja kružnom pilom true, because certain frequency components can have lower values due to tool marks, but it is assumed that they will not contribute significantly to the overall value of roughness signals due to saw teeth marks, reconstructed after filtering.

Further signal processing and filtering of previously measured roughness signals, obtained by measurements on the machined surface after sawing with circular saw, was conducted in accordance with the procedure explained in Đukić *et al*. (2022). The component of roughness signal that, according to our assumptions, should correspond to saw teeth marks on machined surface after sawing with circular saw, was determined by filtering with filter based on fast Fourier transform with the threshold value determined in this experiment. From this filtered signal, R_a values were calculated for every measured profile according to Eq. 1.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

In the previous research (Đukić *et al*., 2022), surface roughness profiles were obtained from the experiments carried out during longitudinal sawing of solid oak wood with values of feed per tooth equal to f_z = (0.02, 0.04, 0.07 and 0.14) mm and feed per one revolution of the saw blade f_o = (0.52, 1.04, 1.69 and 3.39) mm. With respect to roughness measurement parameters, corresponding values of frequency of occurrence of tooth marks was f_{fz} = (4.6, 2.3, 1.4 and 0.7) Hz and frequency of occurrence associated with any tool related phenomena that is related to one revolution of the saw blade was equal to f_{fo} = (0.19, 0.10, 0.06 and 0.03) Hz.

The value of threshold used in the previous research was 2 µm and this value was subjectively determined by trial and error. The difference between this value and the threshold determined by objective criteria based on anatomical roughness was only 0.4 µm. Due to this fact, after filtering roughness signals of machined surfaces, after sawing and reconstructing the part of the signal that should correspond to the saw teeth mark contribution to the overall roughness, there was no significant difference compared to the results published in the previous research. All conclusions related to the machined surface roughness after sawing solid oak wood with circular saw, which were presented in the previous research, can also be applied here. Frequency components due to saw teeth marks, which would correspond to f_{fz} values, are not visible due to low values of feed per tooth used in the experiment. This would result in correspondingly low values of theoretical saw teeth marks, and this signal would be hard or impossible to distinguish from anatomical roughness. This can be justified by the data presented in Đukić *et al*. (2023).

Comparison of the frequency spectra of anatomical roughness signals and machined roughness after sawing with circular saw in relation to threshold value can be seen in Figure 5.

From the signals obtained by measurement of anatomical roughness profiles, R_a values were calculated according to Eq. 1. The average value was 2.1 µm with pooled standard deviation (Figliola and Beasley, 1991) equal to $s(R_a)$ = 0.3 µm. This value (or similarly determined R_q value of anatomical roughness profile) can be considered as a value that can be used for removing the influence of roughness component due to wood anatomy from the measured surface profile after machining. It can be used in a similar context as a method based on structure number ΔF (Magoss, 2008; Csanády and Magoss, 2011) or method of anatomical roughness removal based on the Abbot-curve, as described by Gurau *et al*. (2005). This value can be compared with the data presented in Gurau *et al*. (2005) where, among other parameters, the mean R_a value of surface roughness after sanding solid oak wood with grit P120 was 4.78 µm and for processing roughness it was 2.24 µm. Assuming that the value of R_a parameter associated with anatomical roughness can be represented by the difference between values of R_a parameter for total and processing roughness, its value would be around 2.5 µm. That value agrees well with the value obtained in our research.

Calculated values of R_a parameters for machined surface after sawing and for profiles filtered with filter based on FFT with threshold value obtained in this research can be seen in Figure 6. Values of R_a parameters obtained from filtered profiles should represent the influence of saw teeth marks (processing roughness) on total roughness. If the results are compared to the results obtained in the previous research, there is no significant difference in the obtained values of R_a parameters after filtering. This is because the threshold value determined by trial and error in the previous research was chosen close to the value determined in this research. The threshold determined in this research is more objective and it can easily be replicated for other wood species.

The difference between values of R_a parameters of the machined surface roughness profiles and processing roughness was on the average 3.3 µm with standard deviation of 0.6 µm with data for f_z = 0.04 mm as the only outlier. On the average, the impact of anatomical roughness on surface roughness after sawing for f_z = (0.02, 0.04, 0.07 and 0.14) mm was correspondingly (22, 19, 17 and 15 %). This is in line with the expectations, because with higher feed speeds, the traces of saw teeth on the surface are increasingly pronounced.

In our model, components of roughness profile are non-periodic due to anatomical roughness and pro-

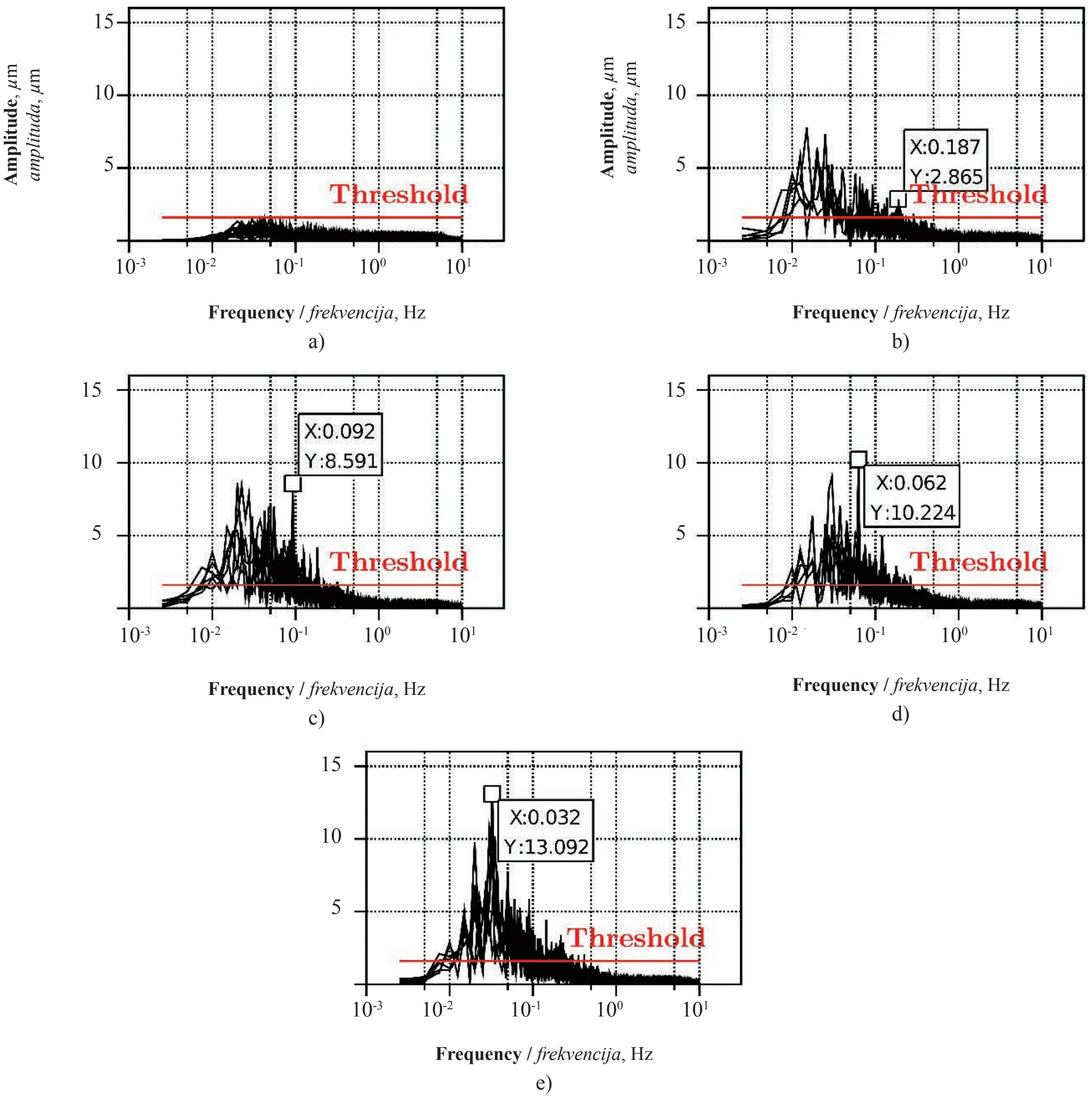


Figure 5 Comparison of frequency spectra for samples of measured surface roughness profiles against objectively determined value of threshold for: a) anatomical roughness, b) machined surface roughness after sawing with f_z = 0.02 mm, c) machined surface roughness after sawing with f_z = 0.04 mm, d) machined surface roughness after sawing with f_z = 0.07 mm and e) machined surface roughness after sawing with f_z = 0.14 mm

Slika 5. Usporedba frekvencijskih spektara za sve izmjerene uzorke profila hrapavosti površine u odnosu prema objektivno određenoj vrijednosti praga osjetljivosti: a) za anatomsku hrapavost, b) za hrapavost obrađene površine nakon piljenja s f_z = 0,02 mm, c) za hrapavost obrađene površine nakon piljenja s f_z = 0,04 mm, d) za hrapavost obrađene površine nakon piljenja s f_z = 0,07 mm i e) za hrapavost obrađene površine nakon piljenja s f_z = 0,14 mm

cessing roughness, which cannot be approximated by periodic functions, so they are represented as non-deterministic signals, e.g., noise. It is known that with averaging the noise is reduced (Smith, 1999). That proved to be the case even if the averaging is applied to the signals of anatomical roughness of solid oak wood (Đukić *et al*., 2023).

To test how averaging would influence the data obtained in this study and correspondingly the threshold level, averaged frequency spectra of anatomical roughness signals and roughness signals obtained on machined surface after sawing with circular saw were examined. The averaged frequency spectra were obtained by calculating the average values for each frequency component of individual samples. It included the calculation of an average for each row (amplitudes for each frequency component were represented as column vectors) of all the frequency spectra of anatomical roughness and each frequency spectra of processing roughness for different values of feed per tooth. In the end, one averaged frequency spectra was obtained for each type of roughness. The results are presented in Figure 7.

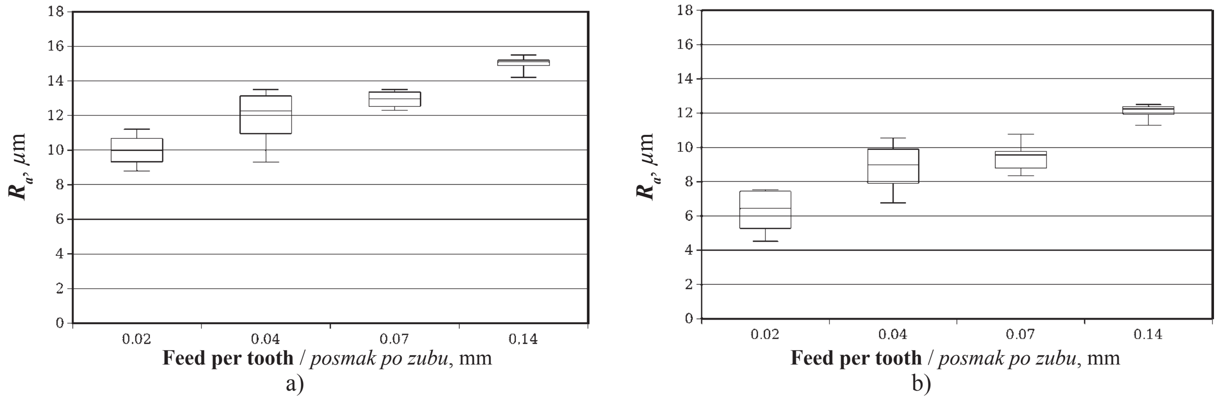


Figure 6 Machined surface roughness expressed through parameter R_a: a) after sawing solid oak wood with different values of feed per tooth and b) for processing roughness signal, after filtering total roughness signal with filter based on FFT and threshold obtained in this research

Slika 6. Hrapavost obrađene površine izražena putem parametra R_a: a) nakon piljenja hrastovine s različitim vrijednostima posmaka po zubu, b) za hrapavost zbog obrade, koja je dobivena filtriranjem ukupnog signala hrapavosti s filtrom na temelju FFT-a i praga osjetljivosti dobivenoga u ovom istraživanju

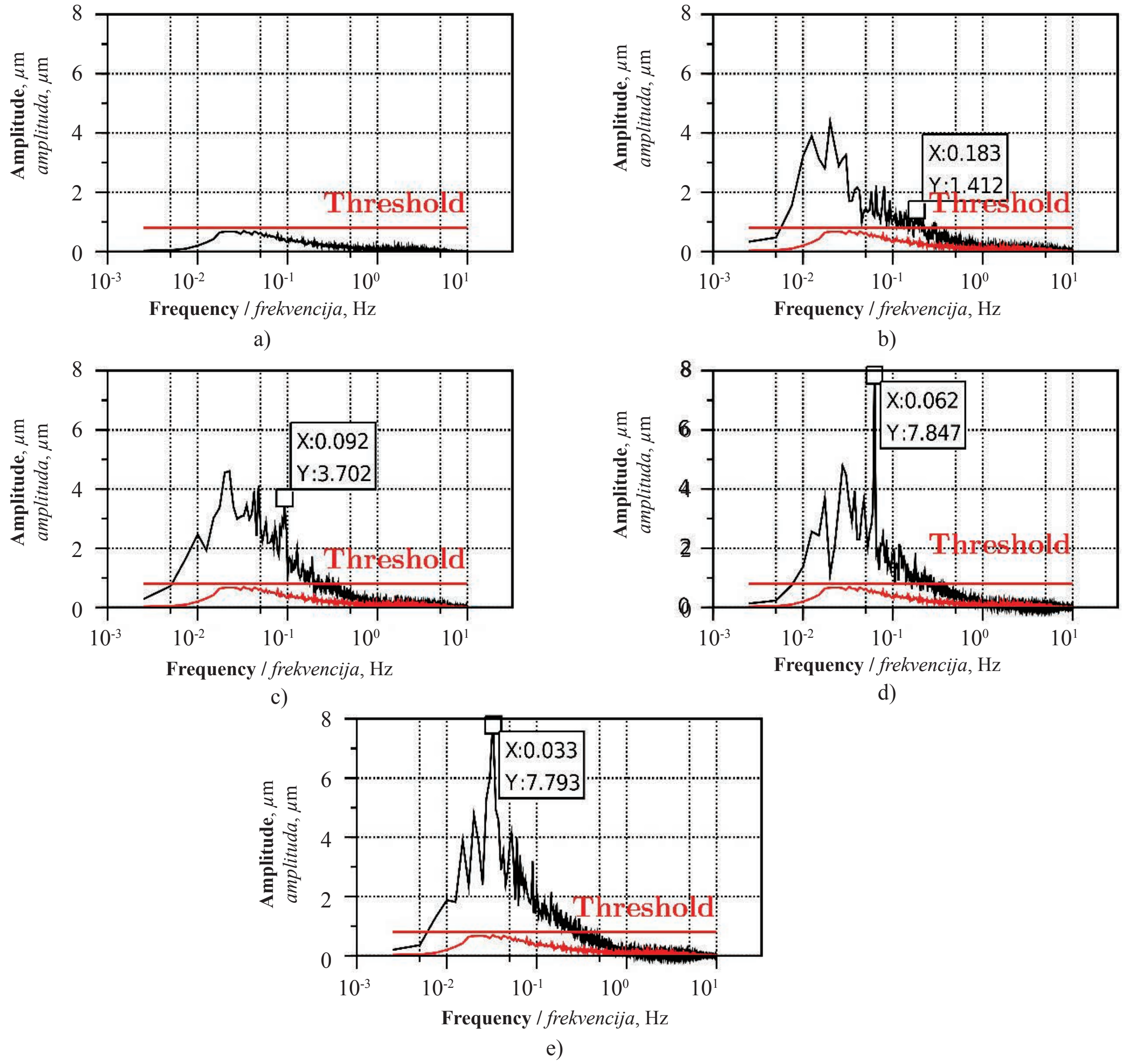


Figure 7 Comparison of frequency spectra of averaged surface roughness profiles for: a) anatomical roughness, b) machined surface roughness after sawing with f_z = 0.02 mm, c) machined surface roughness after sawing with f_z = 0.04 mm, d) machined surface roughness after sawing with f_z = 0.07 mm and e) machined surface roughness after sawing with f_z = 0.14 mm

Slika 7. Usporedba frekvencijskih spektara usrednjenih profila hrapavosti površine: a) za anatomsku hrapavost, b) za hrapavost obrađene površine nakon piljenja s f_z = 0,02 mm, c) za hrapavost obrađene površine nakon piljenja s f_z = 0,04 mm, d) za hrapavost obrađene površine nakon piljenja s f_z = 0,07 mm, e) za hrapavost obrađene površine nakon piljenja s f_z = 0,14 mm

If these results are compared with the results presented in Figure 5, it can be easily seen that, with averaging, the threshold level is twice smaller than previously determined. Also, with averaging, the periodic components are much easier to discern.

Therefore, it can be concluded that the threshold level should be determined from the similar roughness signal length to be used and with similar post-processing, because otherwise wrong conclusions could be reached.

4 CONCLUSIONS
4. ZAKLJUČAK

It can be concluded that the present experimental method can be used successfully for the determination of anatomical roughness of solid wood. From roughness profile obtained on such surface, threshold value for filtering out the anatomical roughness with filter based on fast Fourier transform (FFT) can be used. From the frequency analysis of anatomical roughness signals, obtained on the radial cross section of solid oak wood, a threshold of 1.6 µm was determined.

The average value of R_a parameter of anatomical roughness was 2.1 µm with standard deviation of 0.3 µm. The average impact of anatomical roughness on surface roughness after sawing for f_z = (0.02, 0.04, 0.07 and 0.14) mm was (22, 19, 17 and 15 %), respectively.

It should be kept in mind that the determination of the threshold based on frequency analysis must be carried out on the similar sampling lengths to be used in further analysis, due to the averaging effect. Otherwise, too low values of threshold could be used, and wrong conclusions could be reached.

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Chemical Content and Antimicrobial Activities of Essential Oils Obtained from Plant Parts of *Juniperus excelsa* M. Bieb.

Kemijski sastav i antimikrobno djelovanje eteričnih ulja dobivenih iz dijelova biljke *Juniperus excelsa* M. Bieb.

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ABSTRACT • *This study was conducted to determine the chemical content, antibacterial, and antifungal properties of essential oil obtained from the parts of the plant called Juniperus excelsa M. Bieb. In this study, essential oils of cone, needle, and twig of J. excelsa plant, which is a naturally grown plant in Gümüşhane province, were obtained by hydrodistillation method in a Clevenger type device. Chemical composition of essential oils was determined thanks to the analysis conducted with GC-MS/FID device. Besides, antimicrobial activity tests of essential oils were decided in contrast to 23 different microorganisms with the disc diffusion method. As a result of the essential oil analysis of J. excelsa, the percentage of essential oil yield in cones, needles, and twigs was found as 5.88 %, 2.00 %, and 0.62 %, respectively. α-pinene was confirmed to be the most abundant main compound found in the essential oils of cones, needles, and twigs. As a result of the essential oil analysis of the cone, needle, and twig of J. excelsa species, it was revealed that monoterpenes were the most abundant chemical class in terms of percentage. In the antimicrobial activity test performed on the essential oils of J. excelsa plant parts, it was found that cones, needles, and twigs have a strong antimicrobial effect against Bacillus subtilis, Enterococcus faecalis, Escherichia coli, Escherichia coli O157:H7, Listeria monocytogenes, Salmonella enteritidis, Salmonella typhimurium, Staphylococcus aureus, Candida albicans, Penicillum expansum, Saccharomyces cerevisiae bacteria, yeast, and molds.*

KEYWORDS *antimicrobial activity; chemical content; GC-MS; essential oil; Juniperus excelsa M. Bieb.*

SAŽETAK • *Ovo je istraživanje provedeno kako bi se utvrdio kemijski sastav te antibakterijska i antifungalna svojstva eteričnog ulja dobivenoga iz dijelova biljke Juniperus excelsa M. Bieb. Za potrebe ove studije eterična ulja češera, iglica i grančica biljke J. excelsa, koja je prirodno uzgojena u pokrajini Gümüşhane, dobivena su metodom hidrodestilacije u uređaju tipa Clevenger. Kemijski sastav eteričnih ulja određen je analizom na uređaju GC-MS/FID. Osim toga, disk-difuzijskom metodom ispitano je antimikrobno djelovanje eteričnih ulja na 23 različita mikroorganizma. Analizom eteričnog ulja biljke J. excelsa utvrđeno je da je postotak prinosa eteričnog ulja u češerima, iglicama i grančicama bio (redom) 5,88 % 2,00 % i 0,62 %. Najzastupljeniji glavni spoj pronađen u eteričnim uljima češera, iglica i grančica jest α-pinen. Rezultat analize eteričnih ulja češera, iglica i grančica biljke J. excelsa pokazao je da su postotno najzastupljenija kemijska klasa monoterpeni. U ispitivanju antimikrobne*

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aktivnosti provedenome na eteričnim uljima dijelova biljke J. excelsa ustanovljeno je da češeri, iglice i grančice imaju snažno antimikrobno djelovanje na bakterije, kvasce i plijesni Bacillus subtilis, Enterococcus faecalis, Escherichia coli, Escherichia coli O157:H7, Listeria monocytogenes, Salmonella enteritidis, Salmonella typhimurium, Staphylococcus aureus, Candida albicans, Penicillum expansum i Saccharomyces cerevisiae.

KLJUČNE RIJEČI: *antimikrobna aktivnost; kemijski sastav; GC-MS; eterično ulje; Juniperus excelsa M. Bieb.*

1 INTRODUCTION
1. UVOD

Forests are home to biodiversity of terrestrial ecosystems, and they play a main role in maintaining this biodiversity. Forests do not only meet important human functions such as shelter and food, but they also have important economic value based on products such as wood, food, fiber medicine, etc. Moreover, the fact that forests are home to medicinal and aromatic plants, whose value has greatly increased in recent years, adds additional importance to our forests (Başaran, 2012; Deniz *et al.,* 2014). Considering the flora of Türkiye in terms of its location, it is understood that 11466 plant species are distributed across 7 geographical regions. Related studies demonstrate that 3649 of these plants are endemic, 1700 of them have medicinal properties, and 500 of these plants have medicinal and aromatic properties (Fidan *et al.,* 2011; OGM, 2020). Türkiye is a country rich in coniferous forests due to its geographical location. About half of the country's total forest area consists of coniferous trees (Tumen *et al.,* 2009).

Plants are important potential sources of phytochemicals (Baltacı *et al.,* 2022a). Natural herbal products have been utilized as raw materials in hundreds of industrial products (Öz *et al.,* 2015). The biochemical ingredients found in plants are directly related to the medicinal and aromatic properties of plants. The fact that plants contain various volatile components is a special sign that the medicinal and aromatic properties of plants are quite high (Faydaoğlu and Sürücüoğlu, 2011). Essential oils are described as natural, volatile, complex compounds obtained from the essence of medicinal aromatic plants as secondary metabolites. In order to obtain essential oils, parts of the plant like flowers, needles (leaves), seeds, roots, stems, bark, wood can be utilized separately or used as a whole without parting. In order to determine the essential oil content of the plants, certain parts of the plants must go through the dry or wet distillation process. Following this process, methods such as GC and GC-MS are used to determine essential oil components (Başer, 2010; Baltacı *et al.,* 2022b). Other sectors such as medicine, food, and cosmetics use of essential oils as primary raw materials. These valuable oils have analgesic, wound healing, calming, bactericidal, and fungicidal, refreshing, stress-reducing, mind-opening, and sedative effects. Furthermore, many essential oils have strong antimicrobial properties. In the literature, a great number of studies can be found on the determination of the chemical composition of essential oils obtained from plants, and the determination of antioxidant and antimicrobial properties of these oils (Üçüncü *et al.,* 2010; Fidan *et al.,* 2022).

Yaglioglu *et al.* (2020) stated that the genus *Juniperus* (Cupressaceae) is represented by 8 taxa in Türkiye and approximately 68 species in the world, and that Juniperus species have been used in folk medicine since ancient times. *Juniperus excelsa* M. Bieb. is a tree species that spreads from the Eastern Mediterranean to the Caucasus, Greece, Macedonia, Türkiye, Iran, Afghanistan, and Pakistan. It has been reported that *J. excelsa* is the most dominant species among forests formed by juniper species with a distribution rate of 82 % in Türkiye (OGM, 2014; Saruhan, 2022). *J. excelsa* is one of the evergreen tree species that can grow up to 25 m, and its trunk diameter can be up to 2.5 m. The color of the trunk shell is gray-brown, and the trunk shell, which is in the form of cracked strips towards the length of the trunk, is brown in color when the tree is young, while the color of the shell turns gray when the tree ages (Gültekin and Gültekin, 2006). Juniper cone oil has been reported to be sedative, antiseptic, and analgesic, and is known to cure many other ailments such as jaundice, eczema, and tuberculosis (Khajjak *et al.,* 2012). Kakar *et al.* (2017) reported that *Juniperus* species are characterized by large amounts of essential oil in needles and cones, as well as in seeds and twigs.

J. excelsa (Crimean Juniper) species have been the focus of much research both around the world and in Türkiye due to their characteristics. Among different studies conducted in Türkiye, Topçu *et al.* (2005) investigated essential oil components of *J. excelsa* needles, Ünlü *et al.* (2008) looked into the composition of essential oils obtained from cones, and Nadir *et al.* (2013) examined the chemical composition of needle and cone essential oils. Lesjak *et al.* (2017) evaluated the bioactivity and chemical profile of the needles, and cones of *J.excelsa* in their study. Sela *et al.* (2015) carried out a study to determine the yield, chemical composition, and antimicrobial activity of the essential oils of the cones and needles of *J. excelsa*. In their study, Emami *et al.* (2011) examined antioxidant activity of essential oils obtained from different parts of the *Juniperus excelsa* M. Bieb. *subsp. excelsa* tree. Hojjati *et al.* (2019) reported a comparison of the leaf essential oil composition of ten populations of the *Juniperus excelsa* complex found in Iran.

No study was found on the chemical composition of the essential oils of cones, needles, and twigs of *Juniperus excelsa* grown in Gümüşhane region of Türkiye, nor were their antimicrobial properties investigated. The intent of this study was to contribute to the understanding of the medicinal importance of the plant by determining the chemical composition and antimicrobial properties of essential oils obtained from *J. excelsa* plant parts and by comparing it with other studies.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Plant materials
2.1. Biljni materijali

Cone, needle, and twig samples of the *J. excelsa* tree, which is the focus of the study, were gathered from the natural areas with no human activities in the central Hacıemin District of Gümüşhane province. Identification of plants species was performed by Prof. Dr. Sefa AKBULUT, Faculty Member of Department of Forestry Engineering, Faculty of Forestry at Karadeniz Technical University (Kato Herbarium No:19555). The samples were collected by hand on September 13, 2020 (autumn season) from the above region and dried in the shady and airy environment by mixing at regular intervals. Cone, needle, and twig samples were stored in a cool, dry place out of the sun until analysis. For analysis, the samples were ground and treated in the same way. The area where *J. excelsa* cone, needle, and twig samples were collected is shown in Figure 1.

2.2 Methods
2.2. Metode

2.2.1 Extraction of essential oils
2.2.1. Ekstrakcija eteričnih ulja

Essential oils were obtained by hydrodistillation process. Samples of 100 grams from dried cones, needles, and twigs of *J. excelsa* species were collected, and

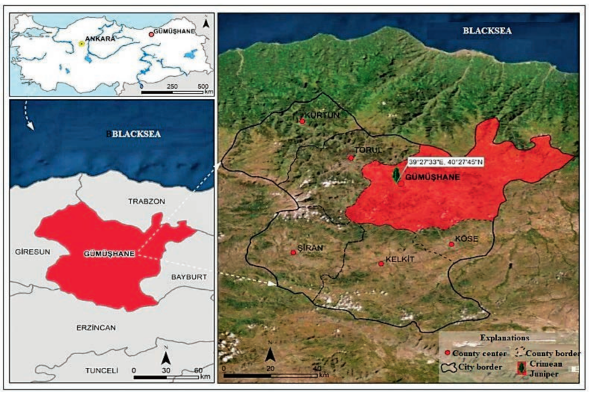

Figure 1 The area where *J. excelsa* cone, needle, and twig samples were gathered
Slika 1. Područje na kojemu su prikupljeni uzorci češera, iglica i grančica biljke *J. excelsa*

ground. 100 g of homogenized cones, needles, and twigs samples were weighed, respectively, in a 2000 mL round Clevenger device flask, by adding 1000 mL of distilled water. It was done with a modified Clevenger apparatus with +4 ℃ cooler for 4 hours. 2 mL of n-hexane was placed in the collection part of the Clevenger device. The temperature of the cooler was set to +4.0 ℃. Essential oils were collected by boiling at a low temperature for 4 hours (Öz *et al.,* 2021). Percentage yields of essential oils by weight were calculated for cone, needle, and twig, respectively. The essential oils obtained were put into colored bottles in n-hexane in GC quality and stored with the lid closed. Percentage of yields for essential oils by weight were calculated separately for cones, needles, and twigs (w/w) (Djordjevic *et al.,* 2021).

$$Yield\ \% = \frac{(Amount\ of\ extracted\ essential\ oil(g))}{(Amount\ of\ dry\ plant\ material)} x100 \quad (1)$$

2.2.2 Analysis of volatile oils components with GC-MS/FID
2.2.2. Analiza hlapljivih organskih spojeva uređajem GC-MS/FID

The volatile oils obtained by hydrodistillation in the Clevenger apparatus were filtered as dissolved in hexane and then placed in the autosampler by putting them in dark colored bottles. Essential oil component analyses were carried out in the Gas Chromatography Mass Detector-Flame Ionization Detector (GC-MS/FID) (GC-FID Agilent-7890A, MS Agilent 5975C model). HP-5MS model nonpolar capillary column (30 m × 0.32 mm, film thickness 0.25 µm) was used for analysis. The injections were applied in splitless mode at 240 °C using helium as the carrier gas with a flow rate of 1 mL / min. 1 µL volatile oil solution in hexane (GC class) was injected, and was initially stored at 60 °C for 2 minutes, and then spectra were taken by raising to 240 °C with an increase of 3 °C/min. After the volatile compounds were separated on a gas chromatography column, mass spectra of each compound were obtained in the mass spectrophotometer one by one. The identification of components of essential oils was performed based on a comparison of retention indices (*RI*) with reference to a homologous series of n-alkanes (C_6-C_{32}), under identical experimental conditions. The mass spectrum of each component was identified through the structure clarification by comparing with the reference compounds of the NIST and Willey libraries, as well as by making a comparison of their retention time either with retention times of authentic compounds or with the literature data (Adams, 2007).

2.2.3 Determination of antimicrobial activity
2.2.3. Određivanje antimikrobne aktivnosti

The antimicrobial activities of essential oils extracted from *J. excelsa* plant parts were carried out in accordance with the disc diffusion method (Matuschek *et al.,* 2014). Essential oil samples were prepared by dissolving them in hexane. According to the method, the samples must be in different concentrations depending on their essential oil densities; values were determined against 23 different microorganisms activated using cones (100 ppm, 50 ppm, 25 ppm), needles (100 ppm, 50 ppm, 25 ppm), and twigs (6240 ppm, 1000 ppm, 500 ppm).

The disc diffusion method was performed in two stages: the preparation of microorganisms and the preparation of samples. All test microorganisms were obtained from Gümüşhane University, Faculty of Engineering, and Natural Sciences, Department of Food Engineering. For this purpose, bacteria were used for antibacterial analysis in Nutrient Broth medium after 24 hours of first activation at 36 ºC, and after 18 hours of second activation at 36 ºC. After the second activation, the prepared sterile Nutrient Agar was smeared on the nutrient media with the swap method. For antifungal analysis, yeasts and molds were used in Malt Extract Broth medium at 27 ºC for 48 hours after the first activation followed by a second activation for 24 hours. The yeasts and molds which had been activated twice were smeared on Malt Extract Agar with the swap method, as was the case with bacteria, and prepared for analysis. The obtained essential oils were absorbed into sterile antimicrobial discs with 20 µL and placed on the previously prepared petri dishes. Petri dishes containing bacteria were incubated for 24 hours at 36 ºC, and petri dishes containing yeast and mold were incubated for 48 hours at 27 ºC. When the specified time expired, the transparent zones around the discs were measured in millimeters, and antimicrobial activity results were specified.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 The amount of obtained essential oil
3.1. Količina dobivenoga eteričnog ulja

Essential oils were obtained as a result of the hydrodistillation process applied to 100 g of dried cones, needles, and twigs. As a result of the analysis, it was found that the amount of essential oil obtained from the cones was 5883.4 mg with a yield of 5.88 % (w/w), the amount of essential oil obtained from the needles was 1988.7 mg with a yield of 2.00 % (w/w), and the amount of essential oil obtained from the twigs was 0.62 % (w/w) with a yield of 618.9 mg. When the amount and percentage of essential oil detected in cones, needles, and twigs were compared, it was decided that the amount and percentage of essential oil in the cones were higher.

Al Hafi *et al.* (2015) reported that the highest yields of essential oils were obtained by Clevenger from *J. excelsa* cone needles and twigs (1.20-2.50 %), followed by needles (1.00-1.50 %), and twigs (0.20-0.50 %). Asili *et al.* (2008), as a result of their study to determine the essential oil in the cones and needles of *J. excelsa*, indicated that the essential oil yields were 1.66 % (v/w) from cones and 1.50 % (v/w) from needles. Furthermore, in a different study carried out in Macedonia, Sela *et al.* (2015) pointed out that the essential oil yields obtained from the needles of this species were between 0.89 % and 1.39 %. In his study, Öncel (2016) obtained the highest amount of essential oil from cones of 1.69 %. It was reported that the amount of essential oil was found to be 1.04 % from the needles, and 0.45 % from the twigs. The literature shows that the results of the present study are compatible with other studies of the same topic and it is supposed that small differences seen may be caused by the method applied, collection time, and genetic factors.

3.2 GC/MS and GC/FID analysis results of essential oil components obtained from cones, needles, and twigs
3.2. Rezultati GC/MS i GC/FID analize komponenata eteričnog ulja dobivenoga iz češera, iglica i grančica

On account of GC-MS/FID analysis, the structure of 93 compounds from the cones of *J. excelsa*, 113 compounds from the needles, and 111 compounds from the twigs were identified. When the essential oil samples are compared in terms of the number of compounds, the number of compounds identified in needle essential oils is seen to be higher than the number of compounds identified in cone and twig essential oils. The results of GC-MS/FID analysis of essential oils obtained from the cones, needles, and twigs of *J. excelsa* are presented in Table 1.

When all components in essential oils obtained from cones were studied, it was determined that the main compounds were *α*-pinene (85.78 %), cedrol (2.62 %), and *β*-myrcene (2.11 %) (Table 1). When all the components in the essential oils obtained from the needles were examined, the main compounds were found to be α-pinene (67.52 %), cedrol (7.98 %), and *β*-myrcene (1.65 %) (Table 1). When all the components in the essential oils obtained from the twigs were examined, it was decided that the main compounds were *α*-pinene (69.92 %), cedrol (2.49 %), and Germacrene B (2.47 %) (Table 1).

Al Hafi *et al.* (2015) stated that the main component detected in essential oils obtained from the needles, twigs, and cones of *J. excelsa* was α-pinene in cones (86.80-95.20 %), in needles (30.60-68.80 %), and in twigs (78.30-89.80 %). In their study on the composition of the essential oil obtained from the cones of the same species, Ünlü *et al.* (2008) found that the essential oil consists of 44 components and that α-pinene (55.50 %), α-cedrol (7.70 %), and sabinene

Table 1 Results of chemical constituents of volatile oil obtained from plant parts (cones, needles, and twigs) of J. excelsa
Tablica 1. Rezultati analize kemijskog sastava hlapljivog ulja dobivenoga iz biljnih dijelova (*češera*, iglica i grančica) *J. excelsa*

No	RI	LRI	Compounds / *Spojevi*	Percent composition / *Postotni udio*		
				JeC	JeL	JeT
1	706	700	Heptane	0.02	0.04	0.06
2	725	725	Cyclohexylmethane	0.03	0.03	0.06
3	802	802	Hexanal	0.02	0.03	0.01
4	825	832	Methyl hexyl ether		0.05	
5	851	850	2-Hexenal		0.09	
6	923	923	Tricyclene	0.20	0.18	0.20
7	931	931	*α*-Thujene			0.06
8	946	946	***α*-Pinene**	85.78	67.52	69.92
9	951	951	*α*-Fenchene			0.08
10	958	958	Camphene	0.37	0.44	0.20
11	961	961	Verbenene	0.02	0.03	0.03
12	975	975	Sabinene	0.27	0.06	0.59
13	982	982	*β*-Pinene	0.87	0.79	1.16
14	997	997	***β*-Myrcene**	2.11	1.65	
15	1007	1007	*α*-Phellandrene	0.02	0.02	0.02
16	1011	1011	3-Carene	0.01	0.76	0.26
17	1019	1019	*α*-Terpinene	0.05	0.04	0.05
18	1027	1027	*o*-Cymene	0.06	0.12	0.09
19	1033	1033	Limonene	0.79	1.49	0.72
20	1034	1034	Eucalyptol	0.01		0.01
21	1038	1038	*cis*-*β*-Ocimene	0.01	0.01	0.02
22	1039	1039	*trans*-*β*-Ocimene	0.01	0.03	
23	1047	1054	Butyric acid, isopentyl ester		0.03	
24	1061	1061	*γ*-Terpinene	0.48	0.44	0.13
25	1069	1069	Sabinene hydrate	0.06	0.02	0.02
26	1091	1091	Terpinolene	0.84	0.72	1.35
27	1100	1100	*trans*-Sabinene hydrate	0.05		
28	1102	1102	Linalool		0.16	0.02
29	1104	1104	Nonanal			0.03
30	1106	1106	Solusterol		0.07	
31	1109	1107	Umbellulol	0.01	0.01	
32	1113	1113	*p*-Mentha-1,5,8-triene	0.01		0.01
33	1115	1115	Fenchol	0.01	0.08	0.01
34	1118	1118	3-Methyl-3-butenyl isovalerate		0.03	
35	1128	1128	*α*-Campholenal	0.02	0.05	0.13
36	1137	1137	1-Terpinenol	0.01	0.01	0.01
37	1141	1141	L-Pinocarveol	0.05	0.06	0.12
38	1144	1144	*trans*-Verbenol	0.03	0.03	0.03
39	1147	1147	Camphor	0.16	0.27	0.12
40	1151	1151	*α*-Phellandren-8-ol	0.01	0.03	0.05
41	1160	1149	*cis*-*p*-Ment-2,8-dien-1-ol		0.03	
42	1163	1163	Pinocamphone	0.01	0.05	0.02
43	1165	1165	Pinocarvone	0.01	0.02	0.03
44	1169	1169	Borneol	0.12	0.11	0.15
45	1175	1177	(*E,E*)-1,3,5-Undecatriene	0.01	0.01	
46	1180	1180	Terpinen-4-ol	0.08	0.04	0.13
47	1188	1188	p-Cymen-8-ol	0.01	0.02	0.02
48	1193	1193	*α*-Terpineol	0.05	0.05	0.16
49	1195	1191	*trans*-Undec-4-enal		0.05	
50	1199	1199	Myrtenol	0.03	0.03	0.09
51	1206	1212	Homomyrtenol	0.01	0.02	0.02
52	1212	1204	Levoverbenone	0.02	0.04	0.03
53	1222	1222	*trans*-Carveol	0.02	0.03	0.03
54	1243	1243	Hexyl isovalerate		0.07	0.02
55	1246	1246	*p*-Cymene-2-ol methyl ether	0.01		0.04

Table 1 (Continuation)
Tablica 1. (Nastavak)

No	RI	LRI	Compounds / *Spojevi*	Percent composition / *Postotni udio*		
				JeC	JeL	JeT
56	1259	1259	Piperitone	0.01	0.05	0.01
57	1263	1257	Myrtanol	0.01		0.03
58	1289	1289	*L*-bornyl acetate	0.29	0.19	0.21
59	1295	1295	(*E,E*)-2,4-Decadienal		0.03	0.03
60	1342	1342	*δ*-Elemene	0.12	0.42	0.70
61	1353	1353	*α*-Cubebene		0.01	0.01
62	1376	1376	Ylangene		0.01	0.01
63	1380	1380	*α*-Copaene		0.08	0.03
64	1384	1385	*α*-Funebrene		0.01	0.01
65	1388	1387	Hexanoic acid hexyl ester		0.06	
66	1389	1389	2-epi-*α*-Funebrene			0.03
67	1397	1397	*β*-Elemene	0.12	0.34	0.34
68	1410	1410	*α*-Cedrene	0.03	1.13	0.04
69	1419	1415	(+)-*β*-Funebrene	0.28		
70	1421	1421	*β*-Cedrene		0.84	0.12
71	1428	1428	Caryophyllene	0.32	0.76	0.73
72	1439	1445	Elixene	0.31		1.45
73	1452	1447	Alloaromadendrene		0.02	0.01
74	1459	1458	*cis*-*β*-Farnesene			0.06
75	1461	1461	Humulene		0.35	
76	1471	1469	*α*-Elemene			0.02
77	1473	1474	Acoradien	0.02	0.06	0.01
78	1478	1483	*γ*-Cadinene		0.03	
79	1481	1481	*γ*-Muurolene	0.02		0.40
80	1484	1484	*α*-Amorphene	0.01	0.27	
81	1494	1494	*β*-Selinene		0.14	0.22
82	1488	1488	Germakren D	0.31	0.99	0.72
83	1496	1493	*β*-Cadinene			0.05
84	1498	1497	*δ*-Selinene		0.12	
85	1501	1501	*α*-Farnesene	0.05		
86	1503	1503	*α*-Selinene	0.04	0.31	0.25
87	1507	1507	*α*-Muurolene	0.02	0.22	0.11
88	1514	1514	*β*-Bisabolene	0.27	0.14	
89	1523	1523	*γ*-Cadinene	0.18	0.83	
90	1533	1533	*δ*-Cadinene	0.09	1.24	0.74
91	1535	1535	Germacrene B	0.48	1.50	2.47
92	1538	1538	*γ*-Bisabolene	0.06	0.10	0.04
93	1541	1539	Cadine-1,4-diene		0.08	
94	1542	1532	*γ*-Selinene		0.29	0.18
95	1544	1542	Cubenene	0.02	0.05	0.04
96	1551	1551	Selina-3,7(11)-dien	0.02	0.11	0.15
97	1557	1557	Elemol	0.19	0.50	0.71
98	1572	1576	Selin-4,7(11)-diene		0.07	
99	1584	1584	Salvial-4(14)-en-1-one			0.26
100	1586	1586	Spatulenol	0.14	0.91	0.36
101	1593	1593	Caryophyllene oxide	0.03	0.05	0.10
102	1596	1596	Globulol		0.02	0.02
103	1599	1599	Epiglobulol			0.16
104	1600	1600	Humulene epoxide	0.16		
105	1602	1601	Viridiflorol		0.51	
106	1617	1612	Isoaromadendrene epoxide			0.05
107	1625	1625	Cedrol	2.62	7.98	2.49
108	1629	1629	Caryophylla-4(12),8(13)-dien-5α-ol	0.02	0.23	
109	1642	1642	*γ*-Eudesmol	0.01	0.17	0.22
110	1653	1653	*τ*-Muurolol	0.02	1.17	0.49

Table 1 (Continuation)
Tablica 1. (Nastavak)

No	RI	LRI	Compounds / *Spojevi*	Percent composition / *Postotni udio*		
				JeC	JeL	JeT
111	1659	1659	*β*-Eudesmol	0.03	0.10	0.62
112	1664	1664	*β*-Selinenol	0.05	0.22	1.03
113	1674	1674	Caryophyllenol-II	0.01	0.08	0.11
114	1679	1674	Eudesma-4(15),7-dien-1-b-ol		0.03	
115	1688	1688	Cinnamyl valerate		0.04	
116	1689	1689	8-Cedren-13-ol	0.01	0.03	0.09
117	1702	1702	14-Hydroxy-α-humulene	0.15	0.50	0.63
118	1707	1709	Juniper camphor		0.04	0.04
119	1713	1714	6-Isopropenyl-4,8a-dimethyl-1,2,3,5,6,7,8,8a-octahydronaphthalene-2-ol	0.01	0.03	0.05
120	1719	1720	epi-α-Bisabol-1-one		0.02	
121	1726	1724	*β*-Santalol			0.08
122	1756	1748	1,10-seco-1-hydroxycalamenen-10-one			0.04
123	1776	1776	*α*-Muurolene-14-ol		0.02	
124	1778	1775	Isovalencenol			0.05
125	1785	1781	*cis*-Lanceol	0.01	0.01	0.06
126	1940	1933	(*E,E*)-5,9-Farnesyl acetone	0.01		0.01
127	1975	1975	Sandaracopimaradiene	0.02	0.01	0.03
128	1991	1991	Manoyl oxide	0.02	0.20	0.13
129	2004	2005	Epimanoyl oxide			0.31
130	2023	2023	Kaurene	0.04	0.01	0.40
131	2032	2032	Kaur-16-ene	0.07	0.01	0.03
132	2068	2069	Dehydroabietan		0.01	0.17
133	2094	2080	2-Phenylethyl geranoate	0.39	0.07	0.04
134	2148	2134	1-Adamantanecarboxylic acid, 3-methylphenyl ester	0.02		0.04
135	2162	2163	Dodecyl benzoate	0.04		0.01
136	2199	2199	*p*-Methoxybenzoic acid, 2-isopropoxyphenyl ester	0.02	0.03	1.31
137	2234	2227	Pimara-7,15-dien-3-one		0.07	2.18
138	2246	2252	Methyl sandaracopimarate		0.01	0.05
139	2285	2288	Neoabietal			0.06
140	2319	2319	Totarol	0.04	0.10	0.19
141	2336	2336	Ferruginol	0.01		1.85
142	2403	2391	Abietinol	0.01	0.01	

RI: Retention indices calculated against C_6-C_{32} n-alkanes on HP 5MS column / *indeksi retencije izračunani u odnosu prema C_6-C_{32} n-alkanima na HP 5MS koloni;* LRI: Retention Indices reported in Literature (Adams, 2007; Wiley and NIST) / *indeksi retencije navedeni u literaturi* (Adams, 2007.; Wiley and NIST); JeL: *Juniperus excelsa* needles / *iglice Juniperus excelsa*; JeC: *Juniperus excelsa* cones / *češeri Juniperus excelsa*; JeT: *Juniperus excelsa* twigs / *JeT: grančice Juniperus excelsa*

(3.50 %) were the main components, as a result of the GC-MS analysis. Lesjak *et al.* (2017) reported that the main components of essential oils obtained from *J. excelsa* cones are α-pinene (77.00 %), cedrol (8.00 %), and limonene (6.00 %), while the main components of the essential oil obtained from the needles are α-pinene (31.00 %), cedrol (37.00 %), and limonene (15.00 %). Gülsoy and Merdin (2017) identified 41 different components in the needles in their study. They determined the essential oil in the needles of *J.excelsa* and its properties. They confirmed that α-pinene (81.28 %) was the main component among these components, followed by myrcene (5.19 %), and limonene (4.52 %). In another study, α-pinene (36.00 %), β-pinene (30.20 %), limonene (12.60 %), β-phellandrene (3.90 %) were specified as the main components of the needles of *J. excelsa* (Nadir *et al.,* 2013). There are some differences between the results of this study and the studies in the literature, although they are similar in general. The chemical classification of the compounds detected in *J. excelsa* cone, needle, and twig essential oils is shown in Table 2.

After the amounts of essential oil components obtained from cones were examined, the most common compound class was found to be monoterpenes with 91.90 % (17 compounds), followed by sesquiterpenoids with 3.47 % (16 compounds), and sesquiterpenes with 2.77 % (20 compounds). It is understood

Table 2 Chemical classification of compounds detected in *J. excelsa* cone, needle, and twig essential oils
Tablica 2. Kemijska klasifikacija spojeva otkrivenih u eteričnim uljima *češera*, iglica i grančica *J. excelsa*

Chemical classification *Kemijska klasifikacija*	**Number of compounds in cones** *Broj spojeva u češerima*	**% amount** *Postotna količina, %*	**Number of compounds in needles** *Broj spojeva u iglicama*	**% amount** *Postotni udio, %*	**Number of compounds in twigs** *Broj spojeva u grančicama*	**% amount** *Postotni udio, %*
Aldehydes	2	0.04	5	0.25	5	0.26
Alcohols	1	0.01	1	0.08	1	0.01
Esters	5	0.76	10	0.60	7	1.68
Ethers	1	0.01	1	0.05	1	0.04
Hydrocarbons	3	0.06	3	0.08	2	0.12
Ketones					1	0.04
Monoterpenes	17	91.90	16	74.32	18	74.90
Monoterpenoids	21	0.77	20	1.06	19	1.09
Sesquiterpenes	20	2.77	29	10.52	27	8.94
Sesquiterpenoids	16	3.47	20	12.62	21	7.63
Diterpene	3	0.13	4	0.04	5	0.95
Diterpenoids	4	0.08	4	0.38	4	4.34
Total	93	100	113	100	111	100

that the total ratio of other compounds was quite low with 1.86 %. Considering the amounts of essential oil components obtained from the needles, the most common compound class was monoterpenes with 74.32 % (16 compounds), followed by sesquiterpenoids with 12.62 % (20 compounds), and sesquiterpenes with 10.52 % (29 compounds). The total ratio of other compounds was found to be very low with 2.54 %. Regarding the amounts of essential oil components obtained from the twigs, the most common compound class was found to be monoterpenes with 74.90 % (18 compounds), followed by sesquiterpenes with 8.94 % (27 compounds), and sesquiterpenoids with 7.63 % (21 compounds). The total ratio of other compounds was found to be 8.53 %.

3.3 Antimicrobial test results of essential oil extracts
3.3. Rezultati antimikrobnih ispitivanja ekstrakata eteričnih ulja

The results of antimicrobial activity tests of pine cone, needle, and twig essential oil samples are presented in Table 3.

Essential oil obtained from cones was detected to have a strong antibacterial effect against *Aeromonas hydrophila*, *Enterococcus faecalis*, *Escherichia coli* O157:H7, *Escherichia coli*, *Salmonella typhimurium*, *Klebsiella pneumoniae*, *Pseudomonas aeruginosa*, *Yersinia enterocolitica* from gram-negative bacteria; and *Bacillus cereus*, *Bacillus subtilis*, *Staphylococcus aureus,* and *Listeria monocytogenes* from gram-positive bacteria. It should be noted that cone essential oils show activity in 15 of 23 different test microorganisms analyzed, being effective at different concentrations. Essential oil obtained from needles was found to have a strong antibacterial effect against *Escherichia coli*, *Escherichia coli* O157:H7, *Salmonella enteritidis*, *Klebsiella pneumoniae*, *Yersinia enterocolitica,* and *Enterococcus faecalis* from gram-negative bacteria, and *Staphylococcus aureus*, *Bacillus cereus*, *Bacillus subtilis,* and *Listeria monocytogenes* from gram-positive bacteria. Needle essential oils were found to show activity in 13 of 23 different test microorganisms analyzed. Essential oil from twigs was found to have a strong antibacterial effect against *Escherichia coli* O157:H7, *Escherichia coli*, *Salmonella typhimurium*, *Salmonella enteritidis,* and *Enterococcus faecalis* from gram-negative bacteria, and *Bacillus cereus*, *Staphylococcus aureus*, *Bacillus subtilis,* and *Listeria monocytogenes* from gram-positive bacteria. It was observed that twig essential oils did not demonstrate antifungal activity against analyzed yeast and mold fungi.

In a study, in which the antimicrobial activity of *J. excelsa* essential oils is evaluated clinically and against gram-positive and gram-negative bacterial strains that cause foodborne illness, while the antimicrobial activity of essential oils against gram-negative bacteria is moderate, it has been stated that gram-positives examined in essential oil of both needles and cones are more effective in growth inhibition (Lesjak *et al.,* 2017). Moein *et al.* (2010) reported in their study that the most sensitive bacteria of the essential oil of *J. excelsa* are *Staphylococcus epidermidis,* and *Pseudomonas aeruginosa*. In their study, Asili *et al.* (2008) investigated the antimicrobial activities of essential oil obtained from the cones and needles of *J. excelsa*, and it showed antimicrobial activity against *Bacillus subtilis*, *Candida albicans*, *Escherichia coli*, *Pseudomonas aeruginosa,* and *Staphylococcus aureus*. Ünlü *et al.* (2008) stated that Crimean Juniper cones showed

Table 3 Results of antimicrobial activity test conducted on cone, needle, and twig essential oils of *J. excelsa*
Tablica 3. Rezultati ispitivanja antimikrobnog djelovanja eteričnih ulja *češera*, iglica i grančica *J. excelsa*

	Cones / *Češeri*			**Needles** / *Iglice*			**Twigs** / *Grančice*			**Penisilin G (10 mg)**
	100 ppm	50 ppm	25 ppm	100 ppm	50 ppm	25 ppm	6240 ppm	1000 ppm	500 ppm	
Gram-negative bacteria *Gram-negativne bakterije*	**Diameter of inhibition zones** / *Promjer zona inhibicije*, mm									
Aeromonas hydrophila	16.75±0.10	12.45±0.10	6.15±0.10	-	-	-	-	-	-	34±0.01
Enterococcus faecalis	14.50±0.10	10.35±0.10	5.15±0.10	8.75±0.10	-	-	11.50±0.10	5.50±0.10	-	32±0.01
Escherichia coli O157:H7	14.45±0.10	9.50±0.10	4.60±0.10	16.50±0.10	9.75±0.10	-	16.35±0.10	10.65±0.10	5.60±0.10	34±0.01
Escherichia coli	15.55±0.10	7.80±0.10	-	16.70±0.10	10.25±0.10	-	15.40±0.10	9.80±0.10	4.50±0.10	34±0.01
Salmonella typhimurium	16.80±0.10	9.75±0.10	-	-	-	-	15.70±0.10	8.70±0.10	-	34±0.01
Klebsiella pneumoniae	10.50±0.10	5.10±0.10	-	10.50±0.10	-	-	-	-	-	30±0.01
Pseudomonas aeruginosa	15.50±0.10	8.45±0.10	-	-	-	-	-	-	-	30±0.01
Yersinia enterocolitica	14.50±0.10	8.90±0.10	-	8.90±0.10	-	-	-	-	-	30±0.01
Salmonella enteritidis	-	-	-	15.60±0.10	9.85±0.10	-	14.10±0,10	8.40±0.10	-	34±0.01
Proteus vulgaris	-	-	-	-	-	-	-	-	-	30±0.01
Vibrio parahaemolyticus	-	-	-	-	-	-	-	-	-	26±0.01
Shigella flexneri	-	-	-	-	-	-	-	-	-	30±0.01
Gram-positive bacteria *Gram-pozitivne bakterije*										
Bacillus cereus	15.30±0.10	9.50±0.10	4.30±0.10	15.40±0.10	9.70±0.10	-	16.40±0.10	10.10±0.10	5.45±0.10	30±0.01
Bacillus subtilis	14.60±0.10	8.90±0.10	-	10.50±0.10	5.50±0.10	-	15.50±0.10	9.40±0.10	-	34±0.01
Staphylococcus aureus	14.50±0.10	8.50±0.10	-	15.50±0.10	9.85±0.10	-	16.50±0.10	10.75±0.10	5.35±0.10	38±0.01
Listeria monocytogenes	12.70±0.10	6.90±0.10	-	9.70±0.10	-	-	15.70±0.10	8.90±0.10	-	30±0.01
Micrococcus luteus	-	-	-	-	-	-	-	-	-	28±0.01
Yeast-Molds *Kvasac – Plijesni*										
Candida albicans	15.50±0,10	10.70±0.10	-	8.90±0.10	-	-	-	-	-	22±0.01
Saccharomyces cerevisiae	12.30±0,10	7.30±0.10	-	6.50±0.10	-	-	-	-	-	14±0.01
Penicillum expansum	10.70±0,10	5.65±0.10	-	6.75±0.10	-	-	-	-	-	16±0.01
Fusarium oxysporum	-	-	-	-	-	-	-	-	-	20±0.01
Aspergillus flavus	-	-	-	-	-	-	-	-	-	25±0.01
Zygosaccharomyces bailii	-	-	-	-	-	-	-	-	-	10±0.01

(-): No activity, Penisilin G (10 mg) was used as the standard for bacteria, yeast, and molds / (-): *nema aktivnosti, penicilin G (10 mg) uzet je kao standard za bakterije, kvasce i plijesni* strong activity against *Clostridium perfingens* bacteria in terms of antimicrobial activity, while it showed weaker activity against *Staphylococus aureus*, *Streptococus pvogenes*, *Candida albicans*, *Streptococus ppneumoniae*, *Mycobacterium smegnatis,* and *Candida crusei*. The results of the present study and these studies in the literature are parallel, and it is understood that the essential oils obtained from the plant parts of *J. excelsa* have a strong antimicrobial effect.

4 CONCLUSIONS
4. ZAKLJUČAK

Within the scope of this study, the amount of essential oil, and chemical components, antibacterial and antifungal properties of the cones, needles, and twigs of *J.uniperus excelsa* were examined. In this study, 93 compounds were identified from the cone part, 113 compounds from the needle part, and 111 compounds from the twig part of the plant. As a result of the essential oil analyses of the plant parts, it was seen that the essential oil ratio of the cones was higher than the others, and the number of compounds in the needles was found to be higher. The most abundant main compound in essential oils was found to be α-pinene with (85.78 % in cones), (67.52 % in needles), and (69.92 % in twigs) in all three parts of the plant. In terms of chemical classification of essential oils, the most abundant compound class by percentage in all three parts of the plant was found to be monoterpenes with (91.90 % in cones), (74.32 % in needles), and (74.90 % in twigs). Essential oils obtained from *J. excelsa* plant parts have the highest sensitivity against *B. cereus*, *B. subtilis*, *E. faecalis*, *E. coli*, *E. coli* O157:H7, *L. monocytogenes*, *S. enteritidis*, *S. typhimurium*, *S. aureus*, *C. albicans*, *P. expansum*, *S. cerevisiae*, and they can replace synthetic antibiotics against diseases caused by these organisms.

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Anatomical Characteristics and Fibre Quality of Grapevine (*Vitis vinifera* L.) Stem Wood

Anatomska obilježja i kvaliteta vlakana stabljike vinove loze (*Vitis vinifera* L.)

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ABSTRACT • *This study investigated the anatomical characteristics and fibre quality for papermaking indices of Vitis vinifera L. (grapevine) stem wood, which is extracted as agricultural waste. Two grapevine trunks were collected from the Gülnar region in Turkey. Observations on microscopic anatomical characteristics were carried out on sectioned and macerated wood samples. According to the measurements conducted, the following mean anatomical characteristics were determined: earlywood vessel tangential diameter 258.81 µm, latewood vessel tangential diameter 35.52 µm, ray width 197.19 µm, ray height 4618.67 µm, vessel length 498.85 µm, fibre length 1.03 mm, fibre diameter 22.05 µm, and fibre wall thickness 4.23 µm. Based on the determined characteristics linked to the fibre quality, the fibres of the grapevine can be placed in Quality Class III for pulp and paper processing. All derived indices of grapevine met the acceptable threshold except for the flexibility ratio. Examining the anatomical structure of the grapevine will enable a database to be created for further studying of wood anatomy and these characteristics can be evaluated with respect to other possible areas of use.*

KEYWORDS: *wood anatomy; fibre quality; fibre dimensions; fibre derivative indices*

SAŽETAK • *U ovom su radu istražena anatomska obilježja i indeksi kvalitete vlakana za proizvodnju papira od stabljika vinove loze (Vitis vinifera L.) koje se ekstrahiraju kao poljoprivredni otpad. Dva uzorka stabljike vinove loze prikupljena su iz regije Gülnar u Turskoj. Promatranja mikroskopskih anatomskih obilježja provedena su na presječenim i maceriranim uzorcima vinove loze. Prema provedenim mjerenjima, utvrđene su ove srednje anatomske vrijednosti: tangentni promjer traheje ranog drva 258,81 µm, tangentni promjer traheje kasnog drva 35,52 µm, širina drvnog traka 197,19 µm, visina drvnog traka 4618,67 µm, duljina traheje 498,85 µm, duljina vlakana 1,03 mm, promjer vlakana 22,05 µm i debljina stijenke vlakana 4,23 µm. Na temelju utvrđenih obilježja vezanih za kvalitetu vlakana, vlakna vinove loze mogu se svrstati u III. razred kakvoće materijala za preradu celuloze i papira. Svi proizvodni indeksi vinove loze, osim omjera fleksibilnosti, zadovoljili su prihvatljivi prag. Ispitivanje anatomske strukture vinove loze omogućit će stvaranje baze podataka za daljnje proučavanje anatomije vinove loze i na taj će se način navedena obilježja moći proučavati u drugim područjima uporabe.*

KLJUČNE RIJEČI: *anatomija drva; kvaliteta vlakana; dimenzije vlakana; proizvodni indeksi vlakana*

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1 INTRODUCTION
1. UVOD

Nowadays, obtaining a continuous supply of wood raw materials is becoming difficult because of limited wood resources and high demand, resulting in the gradual reduction of forest resources. The need for wood and wood-based composites will definitely increase in the future. The most obvious factor for this demand is the population growth, which is increasing rapidly. In addition, many different sectors have begun to use wood raw material. Therefore, the imbalance between the demand for wood raw materials and the current supply is inevitable. It is predicted that instead of wood fibre, the use of agricultural and other sourced alternative fibres will play an important role in the future wood supply-and-demand table (Cooper and Balatineez, 1999).

The paper and paperboard industry is one of the most important sectors in which wood fibre is used, with global paper and paperboard production reaching 401 million metric tons in 2020 (FAO, 2022). In the future (by 2050), worldwide paper production is expected to increase to approximately 700 million metric tons (low estimate) - 900 million metric tons (high estimate) (Bajpai, 2016). One ton of paper is produced from around 2.5 tons of wood. The limited wood resources are being increasingly used not only for papermaking, but also for production of furniture, plywood, and many other products, and therefore, other types of fibrous biomass must be considered for paper manufacturing (Przbysz *et al.*, 2018).

The decrease in the availability of raw materials for pulp and paper production have led papermakers to search for new raw material resources. Several studies have been carried out to identify these resources (Sabharwal and Young, 1996; Chandra, 1998). As an alternative to wood-based raw materials, annual plants and agricultural wastes are the most important raw material resources for pulp and paper production. In fact, they provide excellent specialty paper and constitute the sole source of paper raw materials in some regions (Jiménez *et al*., 1997).

Vitis L. (grapevine) is one of the 16 genera of the *Vitaceae* family distributed in the tropics and subtropics and has 84 species distributed in the temperate regions of the northern hemisphere. One of the important species, *Vitis vinifera* L., has a natural distribution in the Caucasus region and is widely cultivated today. The grapevine is cultivated for its fruit in Turkey and in many Mediterranean regions and has a high economic value and deep-rooted history (Kayacik, 1982; Akkemik, 2020). The grapevine is a woody plant that sheds its leaves in winter and climbs by using its tendrils to twine around a support.

Turkey has produced 3 670 000 tons of grapes in an area of 4 170 410 decares. With this amount, grapes constitute 19.26 % of Turkey's fruit production and rank first among fruits in terms of quantity (TUIK, 2021). Accordingly, the amount of leftover stem wood and pruning waste is quite high during the year. It is known that pruning residues amount to approximately 2 420 000 tons per year. A study conducted by Yeniocak *et al.* (2014) on the production of particleboard from grapevine pruning residues determined that these residues are suitable for particleboard production. The suitability of composite production from vine pruning has been explored by several researchers (Ntalos and Grigoriou, 2002; Yaşar *et al.,* 2009; Mancera *et al.,* 2011; Yeniocak *et al.,* 2016; Auriga *et al.,* 2022; Santos *et al.,* 2022). Studies conducted on the use of vine pruning residues in pulp production have concluded that the pulp is of lower quality than that produced from other agricultural wastes (Jiménez *et al*., 1990). In another study, experiments were carried out with other agricultural wastes (olive trimmings, wheat straw, and sunflower) using different pulp production techniques to increase the quality (Jiménez *et al.,* 2006).

The use of vine pruning residues in pulp and composite board production has been investigated by many researchers, but the information available in terms of grapevine stem wood is insufficient. This study aimed to preliminarily investigate the anatomical characteristics of grapevine stem wood, which contains a great deal of woody material compared to pruning residues, and its suitability for papermaking in terms of fibre morphology and fibre quality.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

Wood samples of *Vitis vinifera* L. were collected from the Gülnar District of Mersin Province. Located in the Taşeli section of the Mediterranean Region, Gülnar is at an altitude of 950 m a.s.l. It is located between 36º - 37º North parallels and 33º-34º East meridians. Most of the land in the district land is forested, followed by agricultural areas, pastures, and unused land. The typical Mediterranean climate prevails in the region. In the higher areas, winters are cold and snowy and summers are cool and relatively rainy. The area experiences a mean annual rainfall of 627 mm and annual temperature of 14.3 ºC (Anonymous, 2022).

The anatomical characteristics of two previously felled grapevine trunks (15 cm) were studied based on the International Association of Wood Anatomists (IAWA) hardwood list (Wheeler *et al*., 1989). Two small wood samples from each trunk were obtained from the outer region of cross-sections in order to eliminate juvenile wood. Samples in dimensions of 10 (R) × 10 (T) ×

20 (L) mm were prepared for wood anatomical measurements. These samples were boiled to soften them and then cut into thin sections (about 20-30 µm) using a Leica SM 2010R sliding microtome. The sections were stained with 1 % safranin for 5 min. and dehydrated in ethanol series (50 %, 70 % and 96 %). Then, they were transferred to xylene in two steps of pure xylene, each step lasting for 10 min. The sections were transferred to glass slides and mounted with Entellan.

For measuring vessel and fibre lengths, grapevine wood was separated with a razor blade in the size of half a matchstick. Maceration was performed according to Schultz's method, which was adopted by Merev (1998). Accordingly, the samples placed in test tubes were treated with potassium chlorate ($KClO_3$) and nitric acid (HNO_3) (1:1). The set up was allowed to react in a fume cupboard while standing on a test-tube rack until the chips were softened and bleached. When the reactions were slow, the racks were heated to 60 °C until the maceration of the chips occurred. Macerated chips were washed with distilled water several times until they became clear. The resultant samples were transferred into well-labelled specimen bottles. Glycerine was added to each bottle, and the samples were stained with safranin to highlight the thickness of the cell wall and lumen.

The earlywood and latewood vessels were evaluated separately according to the IAWA list (Wheeler *et al*., 1989). Tangential and radial earlywood/latewood vessel diameter (µm), ray width (µm), multiseriate ray width (number of cells), ray height (µm), and ray frequency (number of rays per millimetre) were measured. From the macerated samples, fibre length (FL in mm), fibre diameter (FD in µm), fibre lumen diameter (FLD in µm), and fibre cell wall thickness (FCWT in µm) were measured. Twenty-five measurements were taken for vessels and rays and fifty measurements for fibres. An Olympus BX51 microscope connected to an Olympus DP71 camera was used to acquire images, and measurements were taken via BAB Bs200Pro Image Processing and Analysis Software.

The fibre morphology measurements were used to calculate the slenderness (felting) ratio (*FL*/*FD*), flexibility (elasticity) ratio [(*FLD*/*FD*)x100], Runkel ratio (2×*FCWT*/*FLD*), rigidity coefficient (2×*FCWT*/*FD*), Luce's shape factor [$(FD^2\text{-}FLD^2)/FD^2$], F-factor [(*FL*/<u>*FCWT*</u>)x100], and Muhlsteph ratio [(cell wall thickness area/fibre cross-sectional area)x100] of the samples (Saikia *et al.,* 1997; Ohshima *et al.,* 2005; Mousavi *et al.,* 2013; Pirralho *et al.,* 2014; Saeed *et al.,* 2017; Afrifah *et al*., 2022). The derived values were evaluated in order to determine fibre quality class following Rachman and Siagian (1976).

Descriptive statistical analysis of anatomical characteristics and derived values were performed using the SPSS 21 software package. Descriptive statistics include mean values, standard deviation values, minimum and maximum values and coefficients of variation, and these values are shown in tables.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Anatomical characteristics
3.1. Anatomska svojstva

The examination of the grapevine wood sections and the evaluation of the measurements were performed. Microscopic transverse, radial, and tangential wood sections are shown in Figures 1A, 1B, 1C, respectively. A summary of the descriptive statistics is given in Table 1. The annual ring boundaries are distinct due to the difference in diameter of the earlywood and latewood vessels and the flattening of the fibres at the end of the annual ring, which has a porous structure. The earlywood vessels were quite large and often solitary (Fig 1D). On the other hand, the latewood vessels consisted of 3 to 6 cells radially spaced (Figure 1E). The tangential diameter of the thin-walled, earlywood vessels was 258.81 µm, while that of the latewood vessels was 35.52 µm. The radial diameter of the earlywood vessels was 260.18 µm, while that of the latewood vessels was 33.01 µm. The vessel elements were 498.85 µm long (medium length) (Wheeler *et al.,* 1989), the perforation plates were simple, and the intervessel pits were large, elongated, and mostly scalariform types (Figure 1F). The vessel-parenchyma and vessel-ray pits were similar, and half bordered. The narrow vessel members occasionally exhibited irregular spiral thickenings. Tyloses occurred frequently in the earlywood vessels.

The paratracheal axial parenchyma cells were solitary, scattered, and irregularly arranged (Figures 2A, 2B). The fibres were of the libriform type, septate and 1.03 mm long. The libriform fibres had very small simple pits on radial and tangential walls. There were vascular tracheids with irregular, very fine spiral thickenings in the latewood. The multiseriate rays consisted of nine cells on average and were 197.19 µm wide and 4.62 mm high (Figure 2C). The rays were heterogeneous, with procumbent body ray cells and 1-4 rows of upright square marginal cells (Figure 2D, arrows 1 and 2). Secretory cells were associated with the ray parenchyma.

The mean values and standard deviation values for each wood anatomical characteristic are summarized in Table 1. In the present study, the tangential vessel diameter ranged from 35.52 to 258.81 µm. The literature reports on the tangential vessel diameter ranged from 60 to 220 µm in Merev *et al.* (2005), and from 30 to 300 µm in Yaşar *et al.* (2009). The vessel

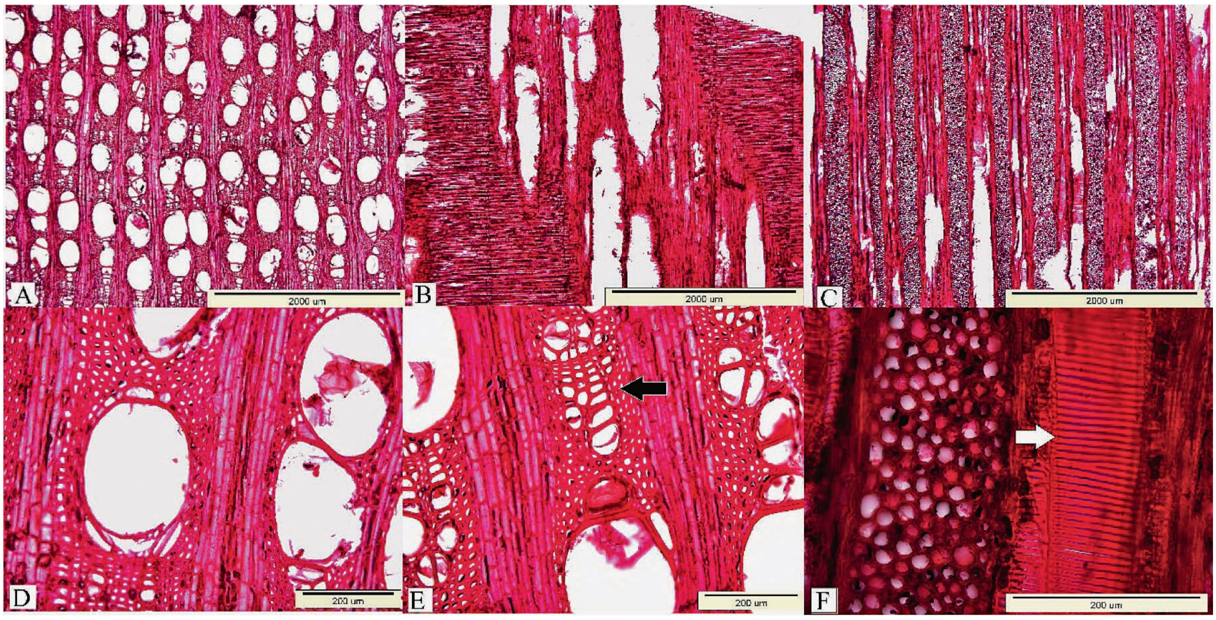

Figure 1 Microscopic view of grapevine wood: A) cross-section, B) radial section, C) tangential section, D) scattered solitary earlywood vessels, E) latewood vessels arranged in radial rows (indicated by arrow), F) elongated intervessel pits (indicated by arrow)
Slika 1. Mikroskopski prikaz vinove loze: A) poprečni presjek, B) radijalni presjek, C) tangentni presjek, D) pojedinačne traheje ranog drva, E) traheje kasnog drva u radijalnim nizovima (označene strelicom), F) izdužene intervaskularne jažice (označene strelicom)

length of the grapevine was 498.85 μm in the present study, similar to the vessel length of 500 μm in Crivellaro and Schweingruber (2013) and the vessel length of 476.7 μm in Hashemi and Tabei (2015). The multiseriate ray width (9 cell) was similar to the values (7 to 13 cell) in the study by Merev *et al.* (2005).

Grapevine fibre lengths belong to the medium length class (900-1600 μm). Fibres were longer in comparison with Hashemi and Tabei (2015) (0.96 mm) and shorter in comparison with Merev *et al.* (2005) (1.25 mm) (Table 1). Despite this, the length class remained the same in all the above sources. Gra-

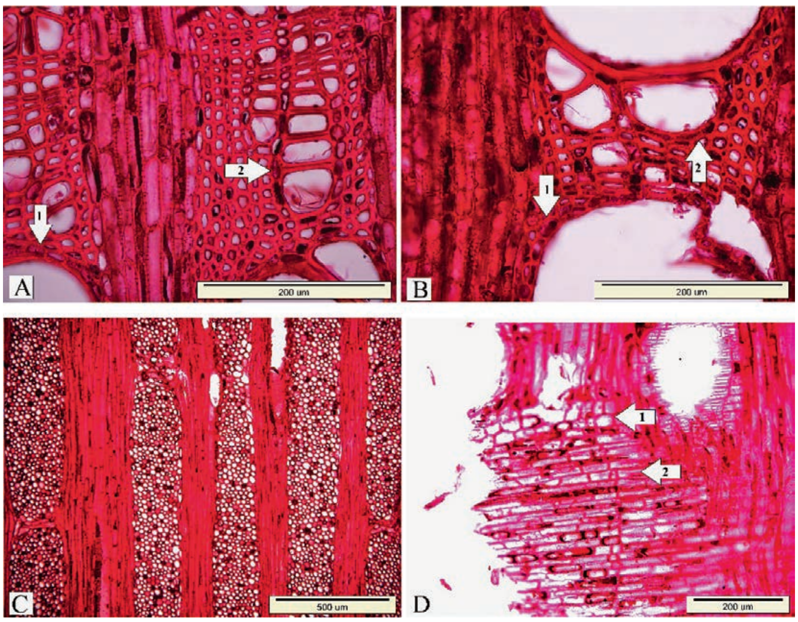

Figure 2 A-B) solitary longitudinal parenchymas (indicated by arrows 1-earlywood, 2-latewood) located irregularly around vessels, C) multiseriate rays in tangential sections, D) square ray cells (arrow 1) and procumbent ray cells (arrow 2)
Slika 2. A) – B) Pojedinačni aksijalni parenhimi nepravilno raspoređeni oko traheja (označeni strelicama: 1 – rano drvo, 2 – kasno drvo), C) višeredni drvni traci na tangentnom presjeku, D) kvadratične parenhimske stanice (strelica 1) i pravokutne parehnimske stanice (strelica 2)

Table 1 Statistical values of wood anatomical characteristics of grapevine wood
Tablica 1. Statističke vrijednosti anatomskih svojstava vinove loze

Features / *Svojstva*	**Mean** *Srednja vrijednost*	**Std. Dev.**	**Max.**	**Min.**	**Coefficient of variation, %** *Koeficijent varijacije, %*
Earlywood vessel tangential diameter, µm *tangentni promjer traheide ranog drva,* µm	258.81	59.87	393.04	155.44	23.13
Earlywood vessel radial diameter, µm *radijalni promjer traheide ranog drva,* µm	260.18	55.99	386.51	174.47	21.52
Latewood vessel tangential diameter, µm *tangentni promjer traheide kasnog drva,* µm	35.52	10.64	58.18	13.77	29.96
Latewood vessel radial diameter, µm *radijalni promjer traheide kasnog drva,* µm	33.01	13.28	63.41	11.11	40.23
Ray width, µm / *širina drvnog traka,* µm	197.19	42.28	284.89	66,62	21.44
Multiseriate ray width (number of cells) *širina višerednoga drvnog traka (broj stanica)*	9.24	1.60	12	4	17.32
Ray height, µm / *visina drvnog traka,* µm	4618.67	2292.49	12115.45	1552.28	49.64
Rays per mm / *broj trakova po* mm	3.5	0.54	5	3	15.43
Vessel length, µm / *duljina traheje,* µm	498.85	136.73	785.16	243.57	27.41
Fibre length, mm / *duljina vlakana,* mm	1.03	0.19	1.54	0.55	18.45
Fibre diameter, µm / *promjer vlakana,* µm	22.05	4.58	33.25	13.25	20.77
Fibre lumen diameter, µm / *promjer lumena vlakana,* µm	13.59	3,38	24.11	7.35	24.87
Fibre wall thickness, µm / *debljina stijenke vlakana,* µm	4.23	1.17	7.35	2	27.66

pevine fibres are in the same length category as the fibres of many hardwoods, e.g., *Quercus robur* (Gülsoy *et al.,* 2005), *Alnus glutinosa* (Tırak Hızal and Erdin, 2016), *Populus alba* (Ištok *et al*., 2019), *Fraxinus angustifolia* (Tırak Hızal and Erdin, 2020), *Fagus orientalis* (Gülsoy *et al.,* 2021) and *Eucalyptus globulus* (Gominho *et al*., 2014; Rahmanto *et al.,* 2021). Compared with other agricultural fibres, some species, e.g., *Corylus avellana* L. (Gençer and Özgül, 2016), *Diospyros lotus* (Topaloğlu *et al.,* 2019), *Persea americana* (Altunışık Bülbül and Gençer, 2021) and *Crataegus azarolus* (Nazari *et al.,* 2021) have a similar fibre length, whereas others, such as *Actinidia deliciosa* (Yaman and Gençer, 2005; Vaysi and Yosefi, 2008), *Gossypium hirsutum* (Tutuş *et al*., 2010), and *Diospyros lotus* (Kiaei and Bakhshi, 2014) showed longer fibres. The grapevine has a longer fibre length than *Persea americana (*Ajuziogu *et al.,* 2010), *Olea europaea* (Ali and Ali, 2014), *Rosmarinus officinalis* (Serin *et al*., 2017), *Populus alba* (Ištok *et al*., 2017), *Prunus armeniaca* (Gençer *et al.,* 2018), and *Crataegus azarolus* (Dong *et al.,* 2021) (Table 2). Fibre length has been reported to play an important role in the processing and mechanical performance of fibre-based products such as paper and fibreboard (Migneault *et al.,* 2008). Ogunkunle and Oladele (2008) reported that long fibres produce paper with higher tear resistance, although Kırcı (2000) and Eroğlu and Usta (2004) reported that long fibres may cause formation defects. The opacity, printability, and stiffness properties improve when short fibres are mixed with longer fibres (Sadiku and Abdukareem, 2019).

The mean fibre diameter value was measured as 22.05 µm and the mean fibre lumen diameter as 13.59 µm. These values are lower than the values found in the study by Hashemi and Tabei (2015) (Table 2). Recent studies have determined that the fibre diameter, fibre lumen diameter, and fibre cell wall thickness are important factors affecting paper properties. To ensure easy collapsibility and provide an effective surface, a thinner cell wall is more appropriate in papermaking (Panshin and de Zeeuw, 1980). The walls of the fibres were classified as having medium thickness (Wheeler *et al*., 1989), and although the thickness class did not change, their values were much lower than those in the literature. The cell wall thickness values of grapevine fibres are given in Table 2. The values indicate that fibres have thinner cell walls in comparison with *Actinidia deliciosa* (Yaman and Gençer, 2005), *Actinidia deliciosa* (Vaysi and Yosefi, 2008), *Olea europaea* (Topaloğlu *et al.,* 2019), *Quercus robur* (Gülsoy *et al*., 2005), *Eucalyptus globulus* (Gürboy and Özden, 1994; Gominho *et al.,* 2014), *Eucalyptus* sp. (Rahmanto *et al*., 2021), *Salix alba* (Eroğlu and Usta, 2004), *Fraxinus angustifolia* (Tırak Hızal and Erdin, 2020), and *Alnus glutinosa* (Tırak Hızal and Erdin, 2016), but thicker in comparison with *Populus alba* (Ištok *et al*., 2017). Fibre cell wall thickness is similar to that of *Gossypium hirsutum* (Tutuş *et al.,* 2010), *Prunus armeniaca (*Tajik *et al.,* 2015), *Corylus avellana* (Gençer and Özgül, 2016) and *Rosmarinus officinalis* (Serin *et al.,* 2017). Hashemi and Tabei (2015) determined the grapevine fibre cell wall thickness to be 5.49 µm, which is considerably higher than the value found in this study.

Table 2 Fibre morphology of grapevine wood and other species in literature
Tablica 2. Morfologija vlakana vinove loze i drugih vrsta u literaturi

Species / *Vrsta*	***FL*** mm	***FD*** µm	***FLD*** µm	***FCWT*** µm	**References** / *Reference*
Vitis vinifera L.	1.03	22.05	13.59	4.23	This study
Vitis vinifera L.	0.58-1.25	-	-	-	Merev *et al.,* 2005
Vitis vinifera L.	0.96	26.45	15.48	5.49	Hashemi and Tabei, 2015
Actinidia deliciosa (A. Chev.)	1.59	35.97	22.30	6.84	Yaman and Gençer, 2005
Actinidia deliciosa (A. Chev.)	1.37	30.04	14.17	7.93	Vaysi and Yosefi, 2008
Corylus avellana L.	1.04	22.20	13.66	4.30	Gençer and Özgül, 2016
Corylus avellana L.	1.06	23.8	14.08	4.8	Merev, 1998
Crataegus azarolus L.	0.84	20.93	-	5.76	Dong *et al.,* 2021
Crataegus azarolus L.	0.93	18.91	-	5.89	Nazari *et. al.,* 2021
Diospyros lotus L.	1.13	12.05	4.89	3.58	Kiaei and Bakhshi, 2014
Diospyros lotus L.	0.94	16.59	6.17	5.21	Topaloğlu *et al*., 2019
Gossypium hirsutum L.	0.81	24.98	16.75	4.12	Tutuş *et al*., 2010
Olea europaea L.	1.11	25.12	14.36	5.38	Topaloğlu *et al*., 2019
Olea europaea L.	0.81	15.60	10.94	2.33	Ali and Ali, 2014
Persea americana Mill.	1.06	25.78	16.18	4.87	Altunışık Bülbül and Gençer, 2021
Persea americana Mill.	0.89	23.00	12.00	5.00	Ajuziogu *et al.,* 2010
Rosmarinus officinalis L.	0.36	12.84	4.22	4.31	Serin *et al*., 2017
Alnus glutinosa L. Gaertner	1.20	26.46	17.32	4.57	Tırak Hızal and Erdin, 2016
Eucalyptus globulus Labill.	0.93	21.4	9.1	6.1	Gominho *et al*., 2014
Eucalyptus globulus Labill.	0.69	20.78	6.42	7.18	Gürboy and Özden, 1994
Eucalyptus sp.	1.16			5.2	Rahmanto *et al.,* 2021
Fagus orientalis Lipsky	1.16	0.60	20.20	5.70	Gülsoy *et al.,* 2021
Fraxinus angustifolia L.	1.20	23.62	18.74	4.88	Tırak Hızal and Erdin, 2020
Populus alba L.	0.87	-	13.38	3.72	Ištok *et al*., 2017
Populus alba L.	0.91	-	-	-	Ištok *et al*., 2019
Prunus armeniaca L.	0.69	12.08	5.69	3.19	Gençer *et al.,* 2018
Prunus armeniaca L.	1.18	14.75	6.11	4.30	Tajik *et al.,* 2015
Quercus robur L.	1.35	18.60	7.40	5.60	Gülsoy *et al*., 2005
Salix alba L.	0.92	20.8	-	5.0	Eroğlu and Usta, 2004

3.2 Derived morphological quality parameters
3.2. Proizvodni morfološki parametri kvalitete

Fibre characteristics are one of the most important parameters in determining paper properties. In order to evaluate paper properties with an objective approach, the slenderness (felting) ratio, flexibility ratio, coefficient of rigidity, Muhlsteph ratio, and F-factor ratio determined from the fibre dimensions should be considered (Kırcı, 2000). The fibre length and fibre parameters of grapevine wood are shown in Table 3.

The slenderness ratio, which is one of the important factors, has a positive effect on the strength, tear, burst, tensile and double-folding resistance of paper (Ekhuemelo and Udo, 2016; Takeuchi *et al*., 2016). The slenderness ratio of the grapevine was 48.57, which is higher than that of *Punica granatum* (35.8) (Gülsoy *et al.,* 2015), *Persea americana* (41) (Altunışık Bülbül and Gençer, 2021), *Olea europea* (44.76) (Topaloğlu *et al*., 2019) and *Rosmarinus officinalis* L. (27.77) (Serin *et al*., 2017) and lower than that of *Eriobotrya japonica* (69.17) (Topaloğlu *et al.,* 2019), *Cornus australis* (52.84) (Gençer and Aksoy, 2017) and *Prunus armeniaca* (55.09) (Gençer *et al.,* 2018). The preferable slenderness ratio for papermaking fibre should exceed 33 (Xu *et al*., 2006). Considering that the slenderness ratio value required for the best papermaking is 70-90 for softwoods and 40-60 for hardwoods, it was concluded that grapevine wood should produce paper with good properties.

Bektas *et al*. (1999) and Ogunleye *et al.* (2017) determined four groups for fibre elasticity: high elastic fibres (flexibility ratio>0.75), elastic fibres (flexibility ratio=0.50-0.75), rigid fibres (flexibility ratio=0.30-0.50), and highly rigid fibres (flexibility ratio<0.30). The flexibility ratio of grapevine fibres was found to be 0.33; therefore, the fibres were determined to be rigid and thus not suitable for paper production. The flexibility ratio value of the grapevine was lower than that of hardwoods and softwoods (0.55–0.75) (Smook, 1997). A lower value on this index gives a higher possibility of paper tearing, collapse, and opacity (Foelkel *et al*., 1978). As the fibres of grapevine wood are rigid, they would not be suitable for paper production.

The acceptable Runkel ratio for papermaking fibres is close to or higher than 1 (Xu *et al*., 2006). Fibres with a Runkel ratio less than 1 are considered as thin-walled fibres (Oluwadare and Sotannde, 2007), while fi-

Table 3 Fibre length and derived values of grapevine wood
Tablica 3. Duljina vlakana i proizvodne vrijednosti vinove loze

Derivative fibres *Proizvedena vlakna*	**Mean** *Srednja vrijednost*	**Std. Dev.**	**Max.**	**Min.**	**Coefficient of variation, %** *Koeficijent varijacije, %*
Fibre length, mm / *duljina vlakana,* mm	1.03	0.19	1.54	0.55	18.45
Slenderness ratio (Felting power) *omjer vitkosti (brzina filcanja)*	48.57	13.45	92.64	19.57	27.69
Flexibility ratio / *omjer fleksibilnosti*	0.33	0.10	0.61	0.15	30.30
Runkel ratio / *Runkelov omjer*	0.65	0.21	1.21	0.3	32.31
Rigidity coefficient / *koeficijent krutosti*	38.47	7.35	54.83	22.83	19.11
Luce's shape factor / *Lucein faktor oblika*	0.61	0.09	0.8	0.4	14.75
F-factor	260.76	83.97	571.60	97.37	32.20
Muhlsteph ratio / *Muhlstephov omjer*	61.61	9.03	79.6	40.45	14.66

bres with a Runkel ratio above 1 are considered as thick-walled fibres (Ezeibekwe *et al.,* 2009). The Runkel ratio is related to paper conformity, pulp yield, and digestibility (Ohshima *et al.,* 2005). The Runkel ratio of grapevine wood fibres was 0.65 and therefore, the value was below 1. This value is similar to that of *Olea europea* (0.78) (Topaloğlu *et al.,* 2019) and *Eucalyptus* sp. (0.70) (Rahmanto *et al.,* 2021), and lower than that of *Cornus australis* (1.16) (Gençer and Aksoy, 2017) and *Prunus armeniaca* (0.99) (Gençer *et al.,* 2018). The morphological characteristics of the grapevine indicate that it possesses good fibre felting power since all the Runkel ratio values were below 1. According to this parameter, the fibres could be satisfactory for papermaking.

A rigidity coefficient value of ≤50 increases the collapsibility of the fibres and thus flexible and strong papers are obtained (Tamolang and Wangaard, 1961). The average rigidity coefficient was 38.47 %. This value is higher than that of other species, and it affects the tensile, tear, burst, and double-fold resistance of paper (Huş *et al.,* 1975). This implies that the low rigidity coefficient of grapevine wood should make it suitable as a raw material for pulp and papermaking.

The Luce's shape factor is related to paper sheet density (Kaur and Dutt, 2013), and a low value could be significantly correlated to the breaking length of paper (Ona *et al.,* 2001). A low Luce's shape factor value has been reported to indicate decreased resistance to beating in paper production (Luce, 1970). The mean value of Luce's shape factor for grapevine wood was 0.61. The determined value is similar to the range of values (0.50 - 0.60) for *Eucalyptus* spp. used in cellulose and paper production (Pirralho *et al.,* 2014; Baldin *et al*., 2017).

The Muhlsteph ratio determines the effect of the cell wall on the physical properties of paper. Thin-walled fibres are easily crushed during paper production and this positively affects paper density or resistance properties (Akgül and Tozluoğlu, 2009). The reason for the low pulp sheet density with low pulp strength is the low Muhlsteph ratio (Przybysz *et al.,* 2018).

For paperboard and corrugated board production, a higher Muhlsteph ratio would be more suitable (Elmas *et al.,* 2018). The Muhlsteph ratio of grapevine wood was 61.61, which was higher than that of *Populus tremula* (47.4) (Atik, 1995), *Acer platanoides* (48.4) (Durmaz and Ateş, 2016), and *Salix excelsa* clones (8.4) (Elmas *et al.,* 2018), but lower than that of *Fagus orientalis* (76.7) (Akgül and Tozluoğlu, 2009) and *Elaeagnus angustifolia* (70.45) (Akgül and Akça, 2020).

The F-factor, which is one of the important parameters for the papermaking industry, indicates the flexibility of fibres. Higher F-factor values yield usable fibres (İstek *et al.,* 2009). The F-factor of grapevine wood was calculated to be 260.76. Thus, the grapevine has a higher F-factor than *Eucalyptus camaldulensis* (249.1) (Huş *et al.,* 1975), *Fagus orientalis* (140.4) (Akgül and Tozluoğlu, 2009), *Acer platanoides* (141.7) (Durmaz and Ateş, 2016), and *Salix excelsa* clones (231.4-233.9) (Elmas *et al.,* 2018), and a lower F-factor than *Populus tremula* (415.1) (Atik, 1995).

According to the Rachman and Siagian (1976) criteria, grapevine fibres had a score of 175, which places the fibre in Class III quality of pulp and paper. Fibres in Quality Class III have moderate to heavy density with a thick wall and narrow lumen. During the sheet forming, fibres do not flatten easily, and felting and bending among fibres are poor, producing low quality in tear, burst and tensile strength.

4 CONCLUSIONS
4. ZAKLJUČAK

In this preliminary study, the suitability of the wood fibres of *Vitis vinifera* L. based on fibre dimensions and fibre parameters was investigated. The anatomical characteristics and fibre quality parameters were compared with those of the same and some other species. Grapevine wood has porous rings, with large vessels. The grapevine wood vessel would be classified as medium-long, having simple perforation plates, large intervessel pits (mostly scalariform type), and scattered paratracheal parenchyma cells.

The fibre length and fibre wall thickness are considered to be two important parameters for pulp and papermaking. Grapevine fibres were classified as medium-long (1.03 mm) and medium thick (4.23 μm). Except for the flexibility ratio, the derived indices of slenderness, Runkel, and Muhlsteph ratios, coefficient of rigidity, Luce's shape factor, and F-factor met the acceptable threshold. In general, the derivative indices of grapevine wood fibres were ranked in Quality Class III. The preliminary results indicate that grapevine wood fibres could be used in the production of paper by blending them with other fibres. However, in order to reach a definite conclusion on this issue, it is necessary to determine the contents of cellulose, hemicellulose, lignin, and extractive substances in grapevine fibres (Eroğlu and Usta, 1989). The fibres might be used in paperboard and corrugated board production and they could be used for papermaking by mixing them with fibres of other suitable species or recycled fibres.

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Analysis of the Effect of Gypsum-Based Fire Protection Cover on Charring of Wooden Bearing Elements

Analiza utjecaja protupožarne obloge na bazi gipsa na pougljenje drvenih nosivih elemenata

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ABSTRACT • *Fire resistance of load-bearing wooden structures is evaluated by the charring of surface layers of wooden bearing elements. The degree of resistance is determined by the onset and course of charring; these basic parameters are affected by the cover structure as well as by cavity fillers between the individual bearing elements. This article focuses on analysing the structure of fire protection covers based on gypsum boards, namely gypsum plasterboards and gypsum fibreboards. The foundation for the current analysis, based on the ignition temperature of the load-bearing timber element under the fire cover, includes the results of experimental fire tests carried out on test specimens in a fire furnace with a standard time-temperature curve set to simulate a fire. In addition, a numerical analysis of the temperature behaviour in the structure was carried out in order to compare the experimental results with the values obtained by mathematical analysis. Finally, the influence of the cover layers on the resulting fire resistance of the analysed structures is evaluated.*

KEYWORDS: *fire resistance; ignition temperature; gypsum plasterboard; gypsum fibreboard; fire test*

SAŽETAK • *Vatrootpornost nosivih drvnih konstrukcija ocjenjuje se prema pougljenjenim površinskim slojevima. Stupanj otpornosti određen je na početku i tijekom pougljenjivanja, a na nj utječe pokrovna konstrukcija, kao i ispune šupljina između pojedinih nosivih elemenata. Ovaj je rad fokusiran na analizu strukture protupožarnih obloga na bazi gipsanih ploča, odnosno gips-kartonskih i gips-vlaknatih ploča. Analiza temperature zapaljenja nosivoga drvnog elementa ispod protupožarne obloge obuhvaća rezultate eksperimentalnog simuliranja požara sa standardnom krivuljom vrijeme – temperatura na uzorcima na kojima je provedeno ispitivanje gorivosti. Osim toga, provedena je numerička analiza kretanja temperature u konstrukciji kako bi se eksperimentalni rezultati usporedili s vrijednostima dobivenim matematičkom analizom. Na kraju je ocijenjen utjecaj obloga na vatrootpornost analiziranih konstrukcija.*

KLJUČNE RIJEČI: *vatrootpornost; temperatura zapaljenja; gips-kartonska ploča; gips-vlaknata ploča; ispitivanje gorivosti*

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1 INTRODUCTION
1. UVOD

The use of wooden elements in construction is subject to strict regulations regarding their fire resistance, as fire safety is a major concern in any building. Increasing the fire resistance of timber structures is a frequent subject of research. The aim of this paper is to highlight the importance of gypsum-based boards as a fire protection cover on the overall fire resistance of a timber structure and on the possible delay of the charring process of load-bearing timber elements. This paper brings a new perspective to this area of investigation in comparing the benefit to the fire resistance of the structure using a fire protection cover of either gypsum plasterboards or gypsum fibreboards. The findings obtained from the fire tests were further illustrated by numerical analysis.

The onset of charring is an important factor in determining the fire resistance (Bisby and Frangi, 2015; Östman, Brandon and Frantzich, 2017; White, 2016) of a timber element. When exposed to fire, wood undergoes a process of charring, which is the partial burning or carbonization of the wood due to high heat in the absence of oxygen (Kuklík and Kuklíková, 2010). The charring process creates a protective layer of char that acts as an insulator, slowing down the rate of heat transfer from the flames to the wood. As the char layer forms, the wood becomes progressively more resistant to fire. Therefore, the onset of charring and the rate at which it progresses are key factors in determining how long a wooden element can resist the effects of fire. The aim of our research was to determine the time difference in the onset of charring of a load-bearing timber element when gypsum plasterboard or gypsum fibreboard was used as a fire protection cover.

Gypsum plasterboards and mineral fibreboards are the most commonly used materials for passive fire protection covers. Academic literature (Fitzgerald, 2004; Thomas, Buchanan and Fleischmann, 1997; Just, Schmid and König, 2010; Piloto, Rodriguez-del-Rio and Vergara, 2022) provides information and relationships for determining the charring rate, charring onset, and cover failure time of these fire protection covers. However, these characteristics may be more difficult to determine for less commonly used compositions.

This article focuses on fire protection covers consisting of two layers of gypsum plasterboards mounted on a steel profile grid or oriented strand boards, as well as two layers of gypsum fibreboards mounted on particle board or oriented strand board.

Figure 1 Experimental fire furnace with test specimen TS1 (photo: authors)
Slika 1. Eksperimentalno ložište s ispitnim uzorkom TS1 (autorska fotografija)

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Test specimens
2.1. Ispitni uzorci

The present analysis investigates how fireproofing affects the onset of charring of timber load-bearing elements. The results obtained by this analysis are based on experimental fire tests of material specimens carried out in accordance with European testing and classification standards for ensuring the fire safety of structures. Modifications were made to these tests to adapt them for use in an experimental fire furnace with an exposure area of 2 m², as shown in Figure 1.

The figures below (Figure 2, Figure 3, Figure 4, Figure 5) provide explicit information about the test specimens, including their individual compositions. In all cases, the fire protection cover had a surface layer directly exposed to high temperatures simulating fire.

The test specimens had dimensions of 1 m × 2 m to fit the test furnace and consisted of wooden prisms with the top covered with gypsum-based boards and wood-based boards. The space between the wooden beams was filled with EPS 100 foam polystyrene for test specimens TS1 and TS2 and mineral wool with a density of 148 kg·m^{-3} for test specimens TS3 and TS4. The test fire protection covers were attached from the underside of the exposed fire and had the following composition (described from above):

(1) Test specimen TS1 (see Figure 2) - oriented strand board with a thickness of 15 mm, steel grid with a thickness of 60 mm (an air gap with a thickness of 10 mm and mineral wool with a density of 148 kg/m^3 mm and a thickness of 50 mm are inside) and two gypsum plasterboards, each with a thickness of 12.5 mm.
(2) Test specimen TS2 (see Figure 3) - oriented strand board with a thickness of 15 mm and two gypsum plasterboards, each with a thickness of 12.5 mm.
(3) Test specimen TS3 (see Figure 4) - oriented strand board with a thickness of 15 mm and two gypsum fibreboards, each with a thickness of 10 mm.
(4) Test specimen TS4 (see Figure 5) – particleboard of Euroclass B with a thickness of 16 mm and two gypsum fibreboards, each with a thickness of 10 mm.

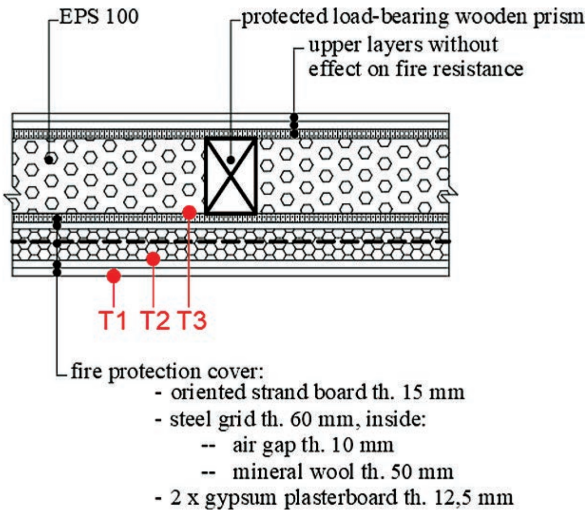

Figure 2 Configuration of test specimen segment TS1
Slika 2. Konfiguracija segmenata ispitnog uzorka TS1

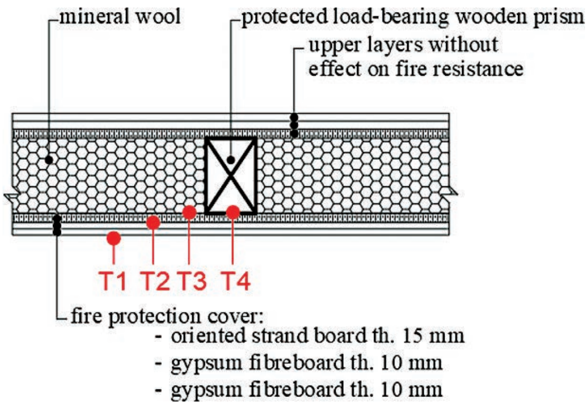

Figure 4 Configuration of test specimen segment TS3
Slika 4. Konfiguracija segmenata ispitnog uzorka TS3

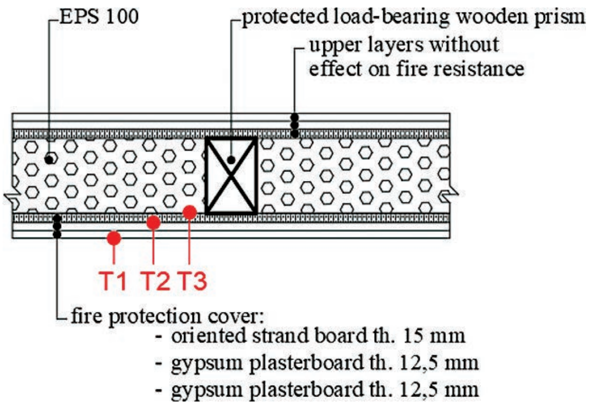

Figure 3 Configuration of test specimen segment TS2
Slika 3. Konfiguracija segmenata ispitnog uzorka TS2

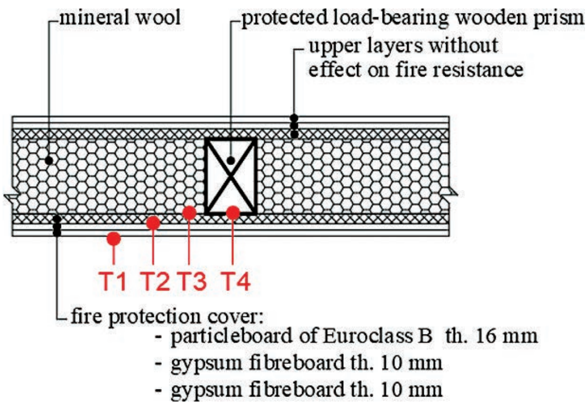

Figure 5 Configuration of test specimen segment TS4
Slika 5. Konfiguracija segmenata ispitnog uzorka TS4

The test specimens were equipped with K-type thermocouples. The T1 thermocouples were placed on a heated surface. The T2 thermocouples were placed on gypsum-based board and the T3 thermocouples on wood-based board (oriented strand board or particleboard). The test specimens TS3 and TS4 still had T4 thermocouples in the joint under the wooden prisms.

2.2 Test procedure
2.2. Tijek ispitivanja

The testing of specimens was carried out in a natural gas fire furnace, using a standard set time-temperature curve (EN 1363-1, 2012), with consideration given to the methodological procedures outlined in the standards for suspended ceilings (EN 1364-2, 1999; EN 1365-2, 2014). The test specimens were placed horizontally in the fire furnace and exposed to the fire from below.

The exposed surface area of each specimen was 1.92 m^2, and the maximum operating temperature limit of the fire furnace according to the time-temperature curve was 1100 °C. Thermocouples T1 to T4 were used to monitor temperatures at 1-minute intervals.

The heating curve in the furnace was continuously monitored and controlled to ensure that the specimen was exposed to temperatures in accordance with the standard time-temperature curve specified by the standard expression (ISO 834-1, 1999):

$$Q_g = 20 + 345 \log (8 \cdot t + 1) \quad (1)$$

Where Q_g is the temperature of gases in the fire compartment under consideration (°C), and t is time (min). In the graphs presented below, this temperature is referred to as T_d.

The deviations from the prescribed temperature values based on the standard time-temperature curve were initially high during the beginning of the test, but after 6 to 7 minutes, they became negligible. It can be assumed that this faster start-up of temperatures did not significantly affect the experiment. Both the temperature and the pressure in the modified testing furnace were checked and found to correspond to the prescribed standard values.

2.3 Charring onset temperature
2.3. Temperatura početka pougljenjivanja

Wood is composed of cellulose, hemicellulose, and lignin that decompose when heated and release flammable gases. If the temperature is high enough, these gases can ignite and sustain a fire.

The ignition temperature of wood is the minimum temperature required to start a combustion reaction in wood although the heat source is removed. The ignition temperature of wood can vary depending on several factors. Generally, the ignition temperature of dry wood is around 300 - 350 °C (Shi and Chew, 2013). For the above fire tests, the isotherm of 300 °C was chosen as the ignition temperature, which means that the test meets the requirements of the standard.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Experimentally obtained results
3.1. Eksperimentalno dobiveni rezultati

3.1.1 Results for test specimen TS1
3.1.1. Rezultati za ispitni uzorak TS1

Based on the temperature data recorded by thermocouple T2, positioned above 12.5 mm thick gypsum plasterboards (Figure 6), it can be concluded that the protective effect of the two layers of gypsum plasterboards diminishes gradually between 39 and 50 minutes. The temperature of 300 °C in thermocouple T3 was reached in the 66th minute.

3.1.2 Results for test specimen TS2
3.1.2. Rezultati za ispitni uzorak TS2

According to the temperature readings from thermocouple T2 placed above the 12.5 mm thick gypsum plasterboard (Figure 7), it can be concluded that within the time interval of 37 to 45 minutes, the protective effect of both layers of gypsum plasterboards gradually decreases. This leads to a sudden increase in temperature in this thermocouple. The temperature of 300 °C in thermocouple T3 was reached at the 54th minute.

3.1.3 Results for test specimen TS3
3.1.3. Rezultati za ispitni uzorak TS3

Based on the temperature monitored in the thermocouple T2 placed above the gypsum fibreboards of 10 mm thickness (Figure 8), it can be concluded that in the interval of 36 to 53 minutes the protection effect of both layers of gypsum fibreboards is gradually lost. Consequently, sudden increase in the speed of temperature growth occurs in this thermocouple.

The isotherm of 300 °C reached the thermocouple T3 at the upper side of the oriented strand board in the 57th minute. The 300 °C temperature in the thermocouple T4 placed in the joint between the wooden prisms and the oriented strand board occurred in the 59th minute, i.e. with a delay of 2 minutes. This delay is caused by the curvature of the isotherm due to the effect of the different properties of the wooden prisms and the cavity filler (Figure 11).

3.1.4 Results for test specimen TS4
3.1.4. Rezultati za ispitni uzorak TS4

After monitoring the temperature using thermocouple T2 placed above 10 mm thickness gypsum fibreboards (Figure 9), it can be concluded that between 33 minutes and 52 minutes, the protective effect of both layers of gypsum fibreboard gradually diminishes. As a result, there is a sudden increase in temperature in this thermocouple.

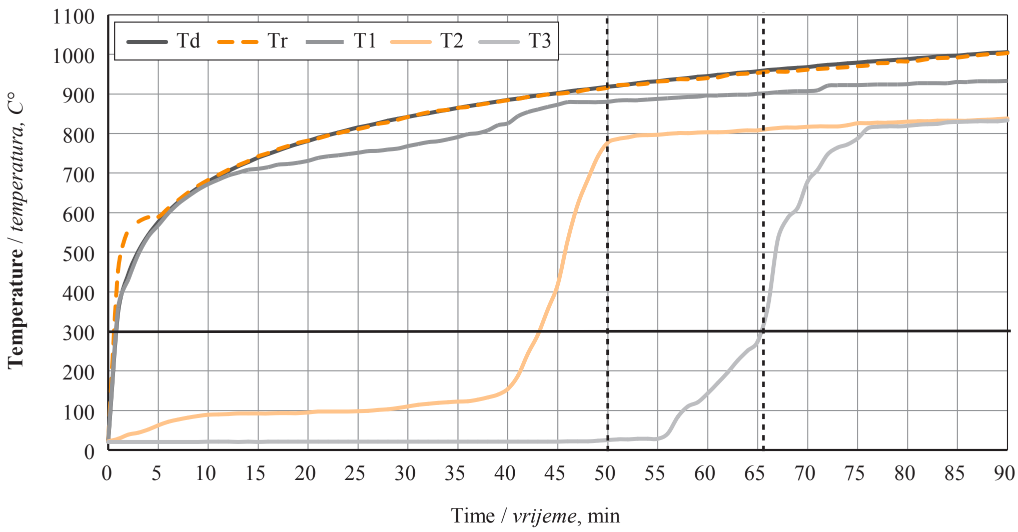

Figure 6 Temperature trends of test specimen TS1 in thermocouples T1 to T3 depending on actually reached temperature T_r in fire furnace with marked significant time points, T_d is fire furnace reference temperature
Slika 6. Temperaturni tokovi ispitnog uzorka TS1 u termoparovima od T1 do T3 ovisno o stvarno postignutoj temperaturi T_r u ložištu s označenim značajnim vremenskim točkama; T_d je referentna temperatura u ložištu

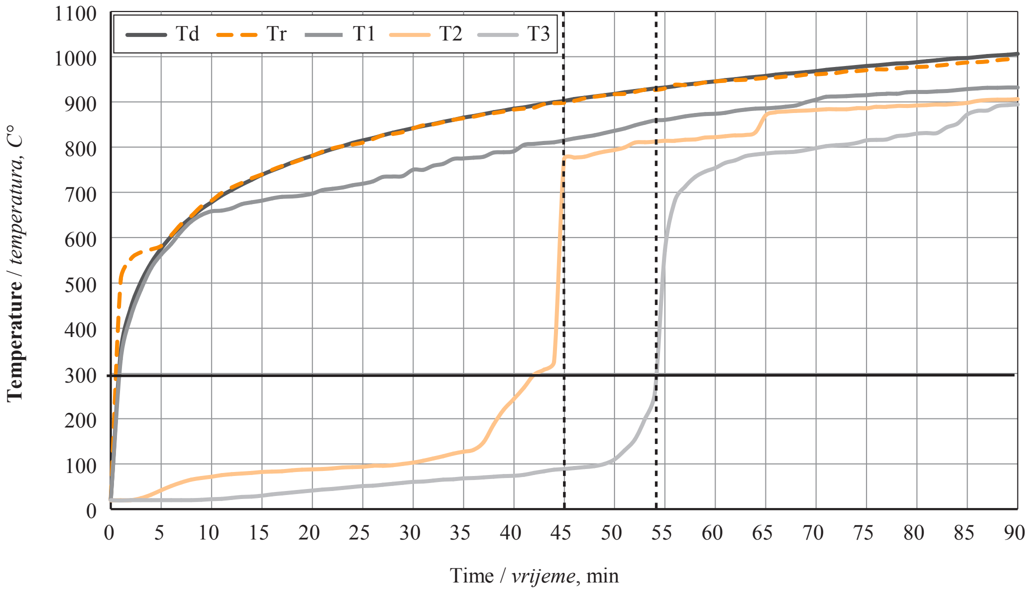

Figure 7 Temperature trends of test specimen TS2 in thermocouples T1 to T3 depending on actually reached temperature T_r in fire furnace with marked significant time points, T_d is fire furnace reference temperature
Slika 7. Kretanje temperature ispitnog uzorka TS2 u termoparovima od T1 do T3 ovisno o stvarno postignutoj temperaturi T_r u ložištu s označenim značajnim vremenskim točkama; T_d je referentna temperatura u ložištu

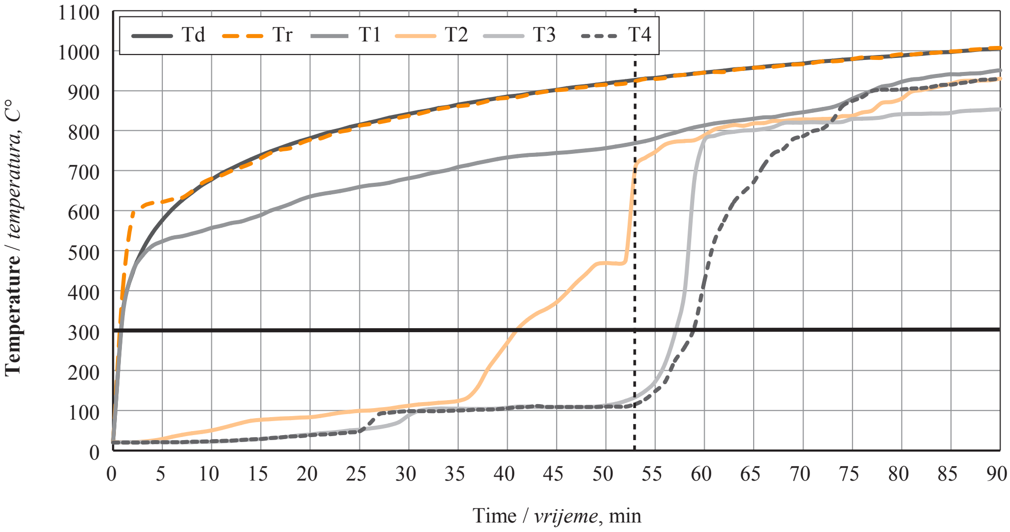

Figure 8 Temperature trends of test specimen TS3 in thermocouples T1 to T3 depending on actually reached temperature T_r in fire furnace with marked significant time points, T_d is fire furnace reference temperature
Slika 8. Temperaturni tokovi ispitnog uzorka TS3 u termoparovima od T1 do T3 ovisno o stvarno postignutoj temperaturi T_r u ložištu s označenim značajnim vremenskim točkama; T_d je referentna temperatura u ložištu

A temperature of 300 °C was reached in the T3 thermocouple on top of the particleboard (Euroclass B) in the 62th minute. This temperature in the T4 thermocouple located in the joint between the wooden prisms and the particleboard occurred with a delay of about one minute.

3.1.5 Other data obtained by testing of specimens
3.1.5. Ostali podatci dobiveni ispitivanjem uzoraka

Figures 6 to 9 provide other data on the behaviour of the test specimens. Temperature points with the sudden temperature change indicate the times of breaking of individual layers of the test specimens. The time interval between the termination of faster temperature increases in the T2 thermocouples and reaching the temperature of 300 °C in the T3 thermocouples reflects the effect that the layers placed over the gypsum-based panels have on the time of onset of charring in the individual specimens.

A comparison of the termination of faster temperature increases in T2 thermocouple and the time to reach the temperature of 300 °C in T3 for the test specimens TS3 and TS4 shows that the fire resistance of

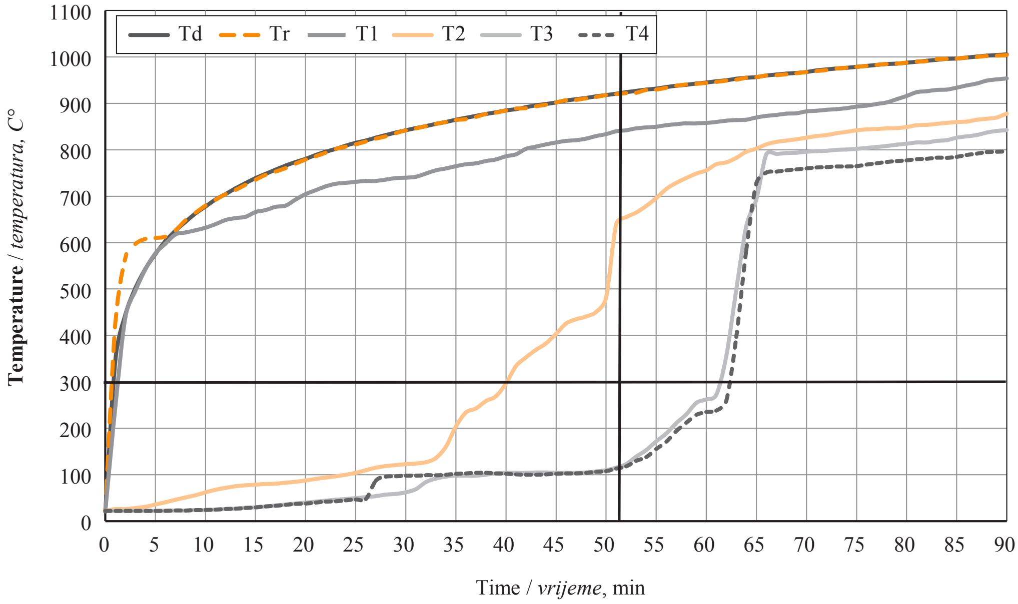

Figure 9 Temperature trends of test specimen TS4 in thermocouples T1 to T3 depending on actually reached temperature T_r in fire furnace with marked significant time points, T_d is fire furnace reference temperature
Slika 9. Temperaturni tokovi ispitnog uzorka TS4 u termoparovima od T1 do T3 ovisno o stvarno postignutoj temperaturi T_r u ložištu s označenim značajnim vremenskim točkama; T_d je referentna temperatura u ložištu

the particleboard of Euroclass B is clearly higher than that of the oriented strand board.

3.2 Analysis of fire test results in terms of fire protection
3.2. Analiza rezultata ispitivanja gorivosti u smislu zaštite od požara

3.2.1 Analysis of temperature trends measured by thermocouples
3.2.1. Analiza temperaturnih tokova mjerenih termoparovima

It is presumed that, when the temperature curves show a sudden rise in temperature, the beneficial effect of the fire protection cover is lost due to its collapse. It can then be considered that $t_f = t_{ch}$, which means that the time of failure of the fire protection cover t_f is equal to the time of charring onset t_{ch}.

The time of charring onset t_{ch} of gypsum plasterboard can be determined according to the formula in Eurocode 5 (EN 1995-1-2, 2004):

$$t_{ch} = 2.8\ h_p - 14. \qquad (2)$$

The variable h_p represents the thickness of fire protection cover in millimetres. If the protection cover consists of two layers of gypsum plasterboard, h_p is defined as the thickness of the bottom layer plus 80 % of the thickness of the top layer. However, for gypsum fibreboards, this formula does not apply.

The charring onset time calculated according to the standard procedure for a fire protection cover made of two layers of 12.5 mm gypsum plasterboard is t_{ch} = 49 minutes. This value is consistent with the results obtained from test specimens TS1 and TS2, whose fire protection cover failure time was $t_f = t_{ch}$ = 50 minutes and 45 minutes, respectively.

Test specimens consisting of two layers of 10 mm gypsum fibreboard had longer charring onset times than test specimens with two layers of 12.5 mm gypsum plasterboard. The failure time of fire protection cover of test specimens TS3 and TS4 was $t_f = t_{ch}$ = 53 minutes and 52 minutes, respectively. The temperature measurements taken by thermocouples on these specimens show that the composition of layers above the gypsum-based boards affects the results.

For test specimen TS1, which had 2 mm × 12.5 mm gypsum plasterboard cladding and mineral fibre thermal insulation on a steel grid, a 10 minute interval was recorded between the time the temperature suddenly started to rise (56 minutes) and the time when the 300 °C isotherm reached the top of the 15 mm thick oriented strand board (66 minutes).

For specimen TS2 with a fire protection cover made of 2 mm × 12.5 mm gypsum plasterboard and of 15 mm of oriented strand board, a 5-minute interval was recorded between the start of fast temperature increase (49 minutes) and the time when the 300 °C isotherm reached the upper side of the oriented strand board (54 minutes).

For test specimen TS3 with a fire protection cover made of 2 mm × 10 mm gypsum fibreboard and a 15 mm of oriented strand board, a 6-minute interval was recorded between the start of temperature rise (53 minutes) and the time when the 300 °C isotherm reached the upper side of the oriented strand board (57 minutes).

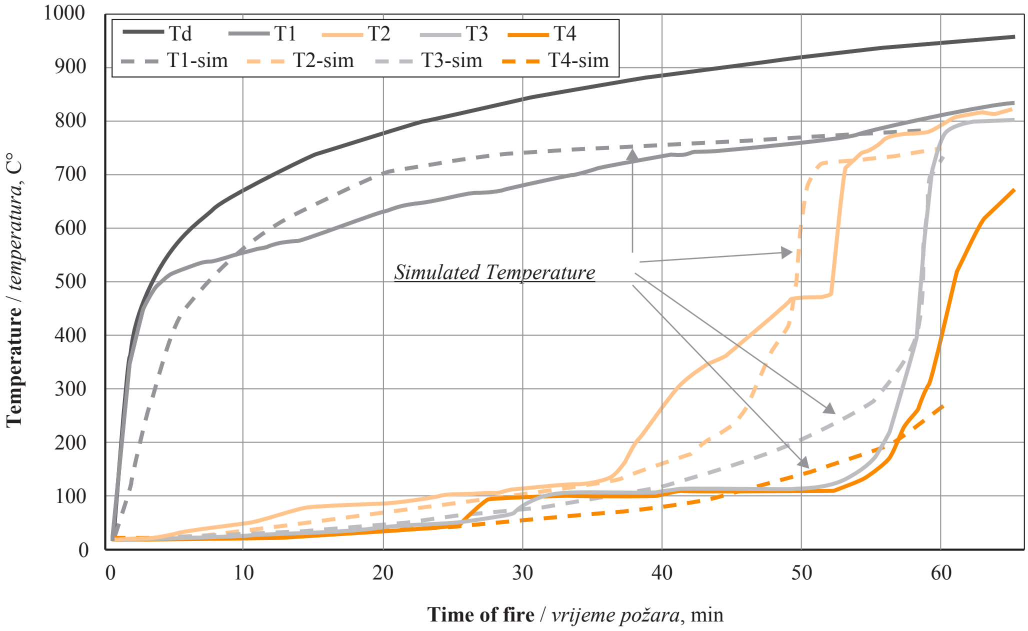

Figure 10 Comparison of numerical analysis and fire test results of test specimen TS3, where T_d is fire furnace reference temperature, T1 to T4 are temperatures measured by thermocouples, T1-sim to T4-sim are temperatures obtained by numerical analysis
Slika 10. Usporedba numeričke analize i rezultata ispitivanja gorivosti ispitnog uzorka TS3, pri čemu je T_d referentna temperatura u ložištu, T1 do T4 temperature izmjerene termoparovima, a T1-sim do T4-sim temperature dobivene numeričkom analizom

For test specimen TS4 with a fire protection cover made of 2 mm × 10 mm gypsum fibreboard and a 16 mm particleboard of Euroclass B, a 10-minute interval was recorded between the start of temperature increase (52 minutes) and the time when the isotherm of 300 °C reached the top side of the oriented strand board (62 minutes).

3.2.2 Numerical analysis
3.2.2. Numerička analiza

The application of the fire test knowledge is presented here (Figure 10) with a numerical analysis that simulates a real fire test, for a selected specimen TS3, which has a fire cover of gypsum fibreboard and oriented strand board.

The numerical analysis was performed using the boundary temperature conditions of the actual test and the material properties of the individual layers (Vimmrová, Krejsová, Scheinherrová, Doleželová and Keppert, 2020; Norgaard and Othuman Mydin, 2013). The dependence of the heat transfer coefficient, specific heat capacity and density of each material on temperature was respected for accurate simulations (Bergmann, Lavine, Incropera and DeWitt, 2018; Ezekoye, 2016). The simulation was performed using ANSYS software, which allows the solution of non-stationary and non-linear heat transfer problem using the finite element method (FEM). The first 60 minutes of the test were modelled with a step interval of 1 minute and a sub-step of 1 second.

The formula used to determine the heat transfer coefficient was taken from the literature (Peter, Veloo and Quintiere, 2013), according to which the heat transfer coefficient includes a radiative component that becomes more significant with higher temperatures. The effect of dropping the charred layers was expressed by increasing the thermal conductivity and decreasing the density and specific heat of the layer in order not to impede heat transfer. Overall, it can be concluded that the simulation is a suitable predictor of fire resistance results, despite some deviations from the actual test results.

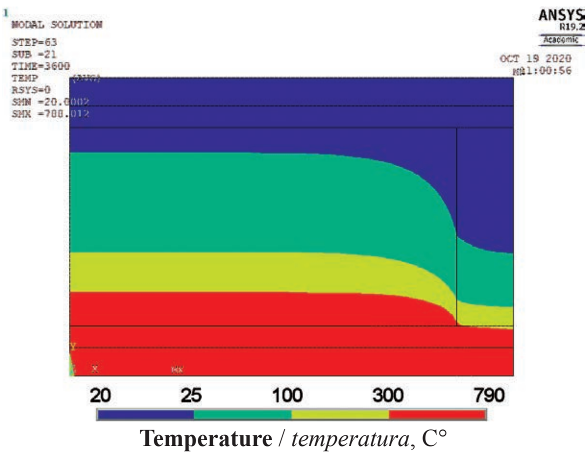

Figure 11 Temperature distribution in cross-section in °C at the end of simulation (after 1 hour)
Slika 11. Raspodjela temperature po presjeku (u °C) na kraju simulacije (nakon jednog sata)

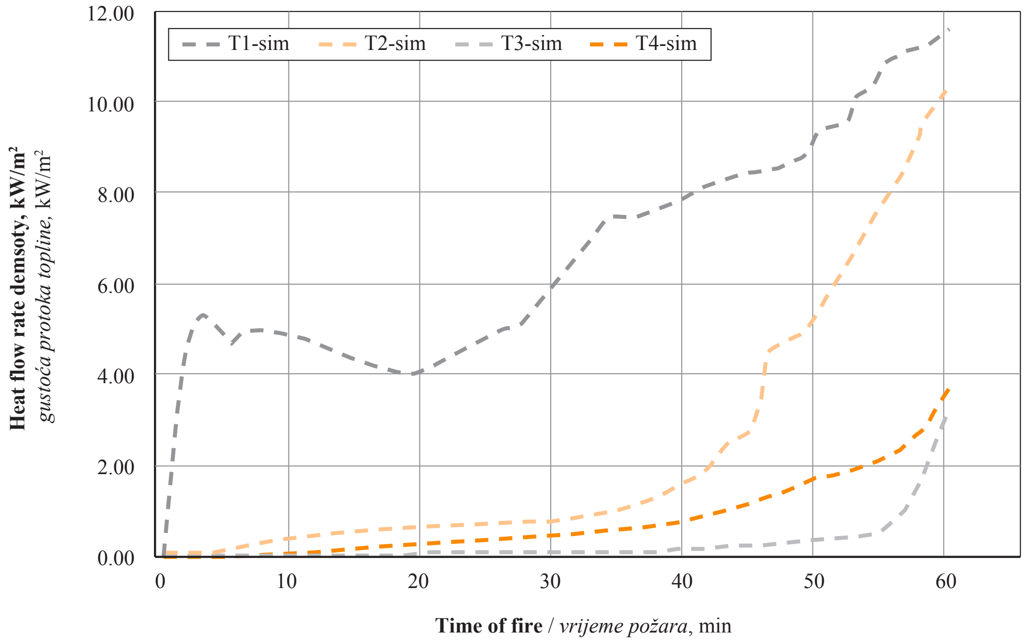

Figure 12 Heat flow rate density in measured points (simulation)
Slika 12. Gustoća protoka topline u izmjerenim točkama (simulacija)

Additionally, Figure12 displays the thermal loading of specimen surface in terms heat flow density (flux) with the maximum values reaching 11.6 kW·m^{-2}. For fires or larger scale experiments, thermal flow density values of 20 to 50 kW·m^{-2} are typically achieved.

The temperature distribution in the cross-section at the end of the simulation (after 1 hour) is illustrated in Figure 11.

4 CONCLUSIONS
4. ZAKLJUČAK

Assuming that the time of onset of charring t_{ch} occurs at the time of failure of fire protection t_f (König, 2004), the time of onset of charring can be determined as the moment when the 300 °C isotherm reaches the surface of the wooden structure, as mentioned above.

Table 1 provides an overview of the critical time points identified by the tests at the locations of the T2 thermocouples and the thermocouples above the wood-based boards.

The high temperature behaviour of the individual fire protection layers was derived from the temperature profiles of the test specimens, and it was assumed that the time point considered as the time of fire protection failure corresponds to the end of the faster temperature increase in the thermocouples T2. A comparison of the termination times of the faster temperature increase on the top side of gypsum-based boards shows that the time of failure of fire protection cover is shorter for gypsum plasterboard than for gypsum fibreboard. The average time to terminate the faster temperature increase in the T2 thermocouple for the gypsum plasterboard test specimens TS1 and TS2 was 47.5 minutes. Gypsum fibreboard test specimens TS3 and TS4 had an average time for the faster temperature increase to cease in thermocouple T2 of 52.5 minutes, which is approximately 10 percent longer than the gypsum plaster board test specimens. Considering that the thickness of gypsum fibreboard (10 mm) was less than that of gypsum plasterboard (12.5 mm), the fire resistance of gypsum fibreboard comes out approximately 12.5 percent higher than that of gypsum plasterboard. The higher fire resistance of the gypsum fibreboards is attributed to the fact that the reinforcement of the gypsum matrix is internal and therefore more protected against

Table 1 Overview of crucial time points
Tablica 1. Pregled ključnih vremenskih točaka

Test specimen *Ispitni uzorak*	**Time points in minutes** / *Vremenske točke u minutama*	
	Termination of faster temperature increase in T2 thermocouple / *Prestanak bržeg porasta temperature u termoelementu T2*	**Reaching temperature of 300 °C in T3 thermocouple** / *Postizanje temperature od 300 °C u termoparu T3*
TS1	50	66
TS2	45	54
TS3	53	57
TS4	52	62

high temperatures than the external reinforcement of the gypsum matrix of the plasterboards.

These numerical values should be considered indicative because the rate of reaching the isotherm of 300 °C in the thermocouples T3 and T4 is influenced by the thermal properties of the layers that are placed on the gypsum-based boards. This is especially evident from the difference in values between test specimen TS1 (50 minutes), where the layer is composed of mineral wool and an air gap, and test specimen TS2 (45 minutes), where the layer is composed of oriented strand boards.

Although the test results showed that gypsum fibreboards have better fire resistance than plasterboards, as can be seen in Table 1, the composition of gypsum plasterboards on a steel grid with mineral wool thermal insulation had the longest time to reach the temperature of 300 °C in the T3 thermocouple. It can be interpreted as the longest time needed to the onset of charring, i.e. the highest fire resistance. This confirms the fact that it is not only the material of the fire protection, but also the composition of its individual layers that matters.

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Positive Aspects of Using Solid Wood in Interiors on Human Wellbeing: A Review

Pozitivni aspekti primjene masivnog drva u interijerima na dobrobit ljudi – pregled literature

REVIEW PAPER
Pregledni rad
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ABSTRACT • *The paper provides an overview of research conducted in the field of wood application in the interior environment and on how solid wood as a material affects human behavior and sense of wellbeing. The analyzed literature includes articles published in the period 1989-2021 in Pub-Med, Google Scholar, Scopus, and Web of Science (WoS) databases using keywords: wood; wellbeing; psychological and physiological responses; indoor environment. Thirty-one articles were processed. Results from the studies confirmed that people have a strong connection and positive behavioral reactions in relation to the use of solid wood in interiors. Wood visually and tactilely affects the mental state of users, and affects physical state, productivity, and stress. Selected studies were reviewed to better understand the impact of the solid wood application on user behavior, health, and wellbeing using objective and subjective test methods. All the findings can be a potential guide for greater future implementation of wood in the sustainable interior design of timber buildings by wood processors, manufacturers, architects, and interior designers, as well as a more vital branding of sustainable and healthy wooden products and buildings with the aim of increasing the wellbeing in interior environments with an emphasis on furnishing sustainable public facilities.*

KEYWORDS: *solid wood in interior, sustainable environment; wood product design; user behavior; wellbeing*

SAŽETAK • *Rad donosi pregled istraživanja provedenih na području primjene drva u interijeru te utjecaja masivnog drva kao materijala na ljudsko ponašanje i osjećaj ugode. Analizirana literatura obuhvatila je članke objavljene u razdoblju 1989. – 2021. u bazama podataka Pub-Med, Google Scholar, Scopus i Web of Science (WoS), a pretražene su korištenjem ključnih riječi: drvo, blagostanje, psihološke i fiziološke reakcije, unutarnje okruženje. Obrađen je trideset i jedan članak. Rezultati istraživanja potvrdili su da ljudi osjećaju snažnu povezanost s masivnim drvom u interijeru i pozitivno reagiraju na njegovu primjenu. Drvo vizualno i taktilno utječe na psihičko stanje korisnika, na njihovo fizičko stanje te na produktivnost i stres. Odabrane su studije pregledane primjenom objektivnih i subjektivnih metoda ispitivanja kako bi se bolje razumio utjecaj upotrebe masivnog drva na ponašanje, zdravlje i dobrobit korisnika. Sva otkrića mogu biti potencijalni vodič za opsežniju buduću primjenu drva u održivom dizajnu interijera drvenih zgrada, na što uvelike mogu utjecati prerađivači drva, proizvođači, arhitekti*

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i dizajneri interijera, kao i aktivnije brendiranje održivih i zdravih drvenih proizvoda i zgrada radi povećanja dobrobiti u interijerima, s naglaskom na opremanje održivih javnih objekata.

KLJUČNE RIJEČI: *masivno drvo u interijeru; održivi okoliš; dizajn proizvoda od drva; ponašanje korisnika; dobrobit*

1 INTRODUCTION
1. UVOD

A vast majority of the population today spends more time indoors (Allen and Macomber, 2020), mostly at home and at the workplace and in other public spaces such as restaurants, schools, shopping centers, office spaces, public institutions and other interiors (Kosonen and Tan, 2004; Höppe, 2002). COVID-19 pandemic has profoundly changed people's ability to recreate outdoors, keeping most of them indoors (Dzhambov *et al*., 2021).

Spaces significantly influence human behavior and feelings, and thus the feeling of wellbeing and health. The results of many studies prove that symbiosis with natural, biophilic design and wood is closely related to better mental and physical health, and contributes to the wellbeing of the individual (Wilson, 2003). Historically, wood has always been used as a traditional building material, but the impact of wood on human wellbeing in interiors is only beginning to be taken seriously and has been researched more intensively in recent decades (Ritter *et al*., 2011).

A number of designers and architects state that the feeling of nature and warmth is the reason why they use wood as the main material in their works, and many will agree that wood is visually more pleasant and calming than other materials (Kaplan and Kaplan, 1989; Fell, 2010).

Despite the claim that wood is one of the preferred natural materials in interiors, there is not much data and research on how wood as a material affects the cognitive perception and behavior of users in interiors, which would provide guidelines for the design of defined products from the appropriate type of wood - not only from a technological but also a visually cognitive point of view.

The aim of this review is to contribute to unifying the knowledge of previous researchers who have investigated the influence of wood in interiors through different products (floors, ceilings and walls) and the influencing parameters of wood on human behavior in interiors. The goal is to enable further research into the impact of certain wood products (e.g., wooden panels) and certain types of solid wood (e.g., oak), the new design which would improve the user's perception of the environment, health, and sense of wellbeing when staying in public spaces.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

The paper follows the structure of a literature review according to the approach of Grant and Booth (2009), which includes three main steps: i) a comprehensive literature search; ii) synthesizing the material in a narrative form; iii) analysis of the paper's contribution in a thematic way. In order to accurately and reliably summarize the evidence, the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) statement is used for reporting systematic reviews (Liberati *et al*., 2009), which helped in the correct methodological approach.

2.1 Search strategy
2.1. Strategija pretraživanja

A close connection between wood products and surfaces in the interior and their psychological and physiological impact on humans has been investigated through literature research and review.

The paper presents an original research obtained by searching articles published from 1989 to 2021 found on Google Scholar, Scopus, PubMed and Web of Science (WoS) online databases. The literature was collected in the period from December 2021 to July 2022, by using the list of keywords "wood"; "wellbeing"; "psychological responses"; "physiological responses"; "indoor environment"; "material properties" and similar terms and combinations related to wood and indoor environment. These terms are combined using the Boolean operators AND and OR (between key terms). Keywords and search combinations were "wood" AND "interior" OR "indoor environment"; "wood" AND "wellbeing" OR "psychological responses" OR "physiological responses"; "wood" AND "stress" OR "productivity".

An additional search of reference lists was manually conducted for the identification of additional, relevant studies, which resulted in finding a couple of articles that were not originally written in English but could also be found in one of the observed databases. To make comprehensive research in this field, relevant research reports, conference papers, books and doctoral dissertations have been included in the search as well.

2.2 Selection of studies
2.2. Odabir studija

In publication research, the main focus was on wood products used in the indoor environment such as

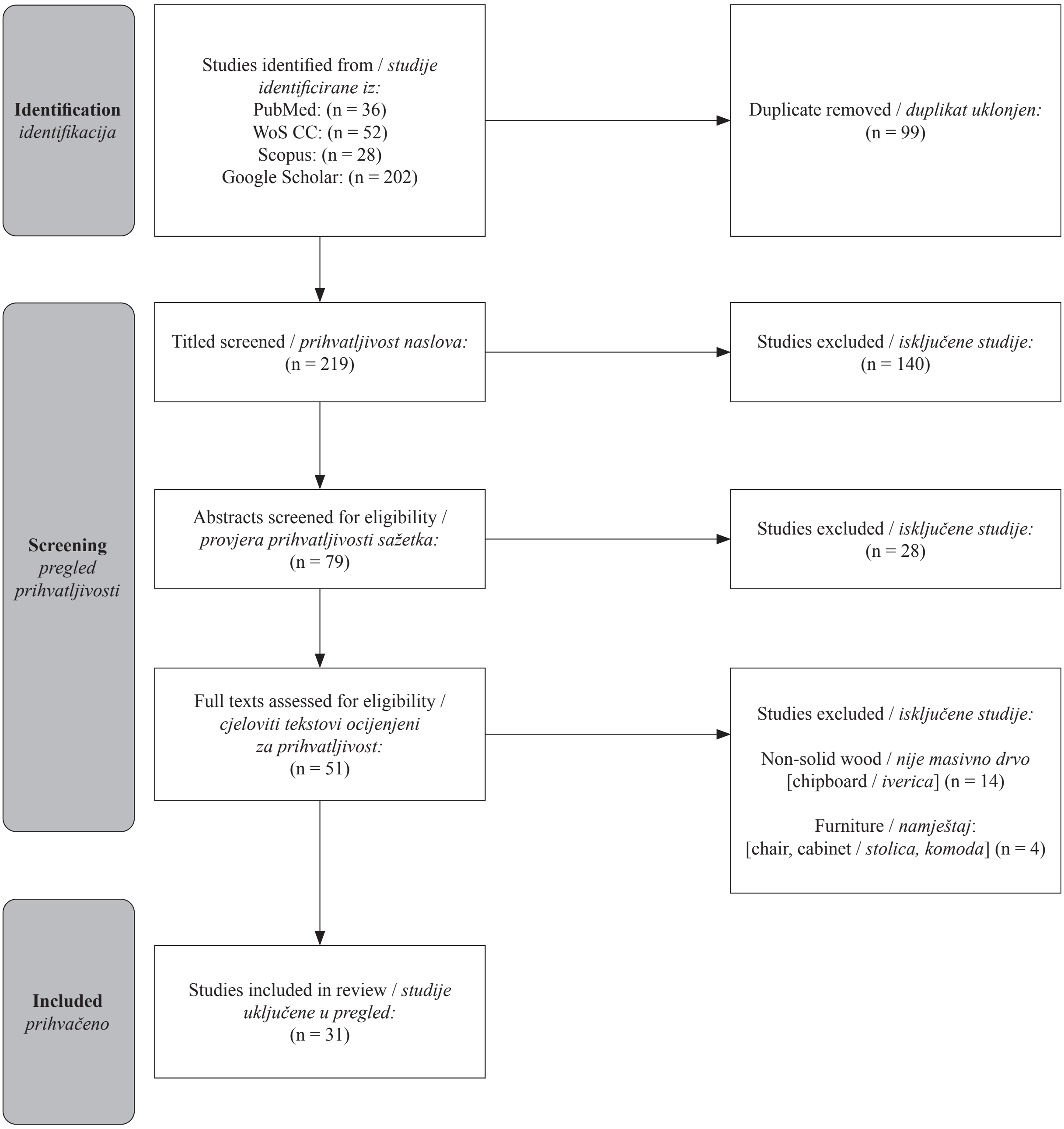

Figure 1 PRISMA-ScR flow diagram showing the process of selecting articles
Slika 1. PRISMA-ScR dijagram toka koji prikazuje proces odabira članaka

wall and ceiling paneling, solid wood surface material used in indoor applications such as veneers. Wood products such as chipboards and all wood based engineered products were eliminated because of their chemical (emissions of volatile organic compounds) and physical (visual appearance) properties, which are different from those of solid wood. Furthermore, the influence of wood furniture in the indoor environment was excluded as it was not within the scope of research.

After the primary results of search terms and addition investigation, 318 studies were chosen for further evaluation. Reading the articles, checking the quality of studies, and relevance of study topics, a total of 31 publications published between 1998-2019 were selected. The publications covered topics of air quality – humidity, antibacterial effects, productivity, stress relief, tactile and visual impacts of wood and wooden surfaces on human behavior.

Figure 1 presents a flow diagram adapted from PRISMA (Liberati *et al*., 2009) of the search process.

3 RESULTS
3. REZULTATI

The results obtained after the literature search were reviewed in detail, and for easier understanding of the cause and effect relationship, they are conceptually presented in Figure 2. The analyzed results imply a division into two groups, subjective and objective. These two groups of results together influence human

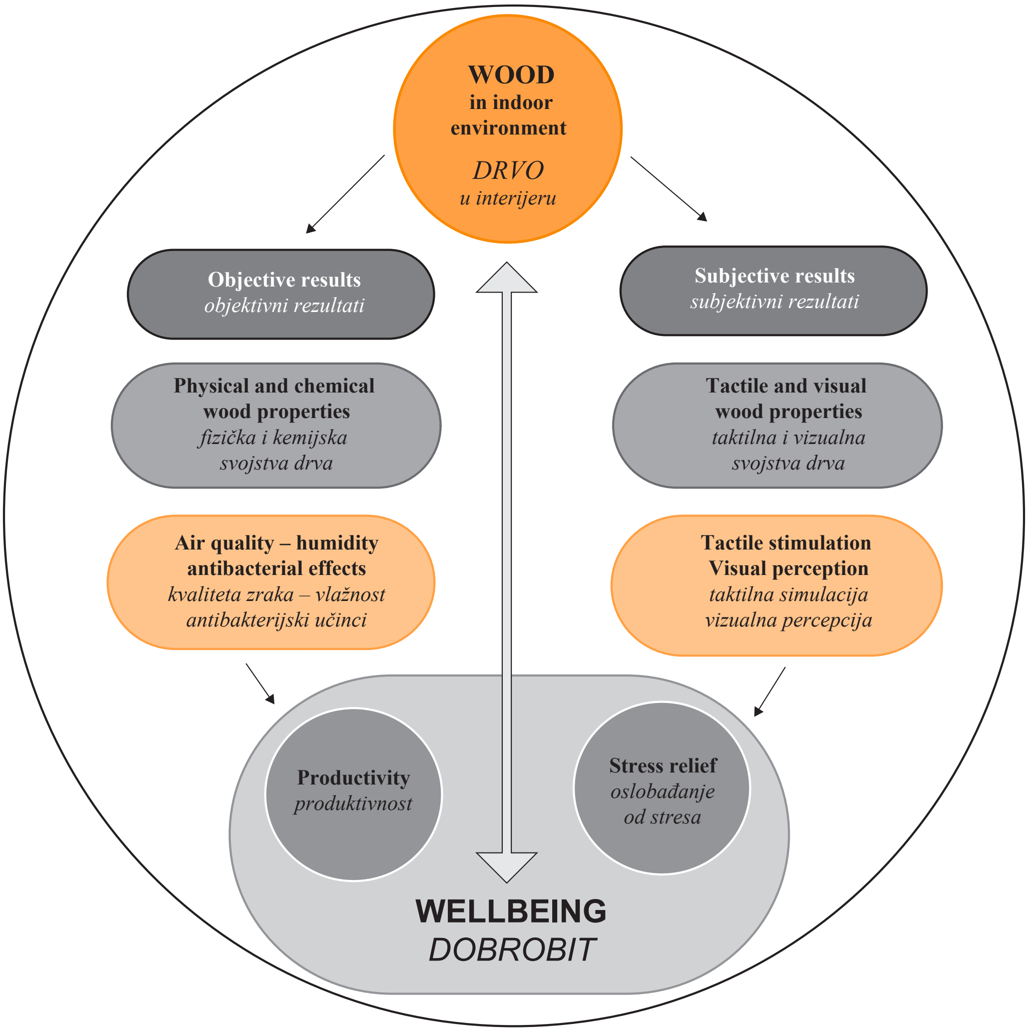

Figure 2 Mind map of identified relationships among results
Slika 2. Mentalna mapa identificiranih odnosa među rezultatima

behavior in terms of productivity and stress relief, which results in a positive outcome or wellbeing.

3.1 Objective results
3.1. Objektivni rezultati

These results are factual and independent, obtained by systematically measurable methods and are not prone to variations and other influences based on the physical and chemical properties of wood (Tsoumis, 1991). The results related to wood and its influence on humidity and air quality, as well as on antibacterial properties, were studied.

3.1.1 Air quality – humidity
3.1.1. Kvaliteta zraka – vlažnost

Wood is a hygroscopic material capable of absorbing and releasing water (Mortensen *et al*., 2005). Various studies have shown that the presence of wooden elements in the room can improve air quality by affecting the humidity of the room (Simpson, 1998; Hameury and Lundström, 2004**)**. When there is a higher amount of moisture in the room than in the wood, the wood will absorb excess water from the air, and also maintain the balance with the relative humidity in the surrounding air.

This characteristic of wood is a particularly important factor that affects the quality of workplaces. Productivity is reduced by 12 % in situations where employees are dissatisfied with the air quality in their workplaces (Bergs, 2002). Wooden products also increase the quality of life in residential areas because wood reduces the relative humidity in the living rooms and has a potential of up to 35 % compared to non-hydroscopic structure (Simonson *et al*., 2002).

Li *et al*. (2012) conducted a room-level test measuring the moisture buffering performance of wood wall coverings and their impact on the interior. The authors noted that wood is not the only material that lowers the moisture content of the air in the room and concluded that the room itself can cause up to 8 % change in relative humidity. Wooden wall coverings can mitigate this change by up to 30 %.

The absorption of wood moisture from the air depends on various factors such as the surface area of the wood exposed to the air from the interior, vapor permeability, surface treatment, thickness and sorption capacity (Osanyintola and Simonson, 2006).

3.1.2 Antibacterial effects
3.1.2. Antibakterijski efekti

With various well-known characteristics of wood such as the previously mentioned ability to bind moisture, scientists are becoming increasingly interested in the antibacterial properties of wood. So far, it has been discovered that wood repels bacteria better than plastic or glass (Hedge, 2015).

Kotradyova and co-authors (2019**)** performed microbiological testing on oak and pine in three different surface treatments (on a wooden surface without finishing, on acrylic varnish, and on a hard-wax oil finish). The results of the analysis showed the presence of antimicrobial action of wood, in contrast to the laminate used for comparison. More bacteria survived on wood treated with varnish than oil. Oak and pine have also been shown to have higher antimicrobial activity even without any chemical treatment because of the presence of tannins and terpenoids.

Another similar study was conducted by Vainio-Kaila and co-authors (2017), who compared the survival of bacteria on untreated wooden surfaces and glass surfaces. Bacteria such as *Escherichia coli* and *Listeria bacteria* were grown in the laboratory and died completely on wooden surfaces during the test, while surviving on glass plates. It has been found that wood is effective in fighting hospital bacteria. Wood contains various extracts that are characterized by their antibacterial properties.

Not every wood species has the same antibacterial properties. This depends on the species of wood. Sapwood and hardwood have different antibacterial properties within each species (Kavian-Jahromi *et al.*, 2015). Extremely strong antibacterial effects have been found in Scots pine wood (*Pinus sylvestris*) (Vainio-Kaila *et al*., 2017, Schönwälder *et al.*, 2002; Milling *et al*., 2005; Laireiter *et al.*, 2013) and European oak wood (*Quercus robur*) (Milling *et al*., 2005). The back of a larch wood (*Larix decidua*) also showed antibacterial properties (Laireiter *et al*., 2013).

3.2 Subjective results
3.2. Subjektivni rezultati

These results were obtained by subjective methods (survey questionnaire, interview and observation), but also by measuring heart rate and blood pressure. The results show the impressions, opinions, feelings and attitudes of users towards wood, which is viewed from the side of visual perception and tactile stimulus.

3.2.1 Visual perception
3.2.1. Vizualna percepcija

The visual impression of wood can vary due to various factors such as species, number of knots, color, structure and surface finish. Examining the visual perception of wood can provide insight into how people perceive different properties of wood. Wood species most often differ based on the visual perception of wood characteristics such as the difference between dark wood and light wood. Likewise, adjectives like exclusive, modern, and inexpensive are used to indicate differences in wood species. Some of the most commonly used factors when describing wood are exclusive – modern vs inexpensive, ecofriendly – natural, and dark – rough. Research has shown that hardwood floors are more often perceived as more expensive and exclusive, while softwoods are considered more environmentally friendly than hardwood (Roos *et al.*, 2013). There are two components that play a major role in the separation of different wooden decking materials (Nyrud *et al.*, 2008). The first component includes unevenness, roughness and knots in relation to flat surfaces, and the second component is the degree of whiteness.

Bowe and Bumgardner (2004) concluded that darker-colored wood is perceived as more expensive than lighter-colored wood. According to Roos and colleagues (2013), this thesis was partly confirmed since their study showed that the results of Nyrud and co-authors (2008) were correct in stating that exclusivity and great value can be perceived both in light wood (maple) and dark wood (ash, elm, and oak). A positive correlation was found in relation to the number of wooden elements in the room, in such a way that respondents perceive rooms as warmer if there is more wood in them (Masuda, 2004). The feeling of warmth in the room was related to the color of the wood, where the colors from the yellow-red spectrum dominated. However, an environment with a high percentage of wood is perceived in more cases as a "natural environment" than a "warm environment".

According to Nakamura and Kondo (2008), knots on wood are a proof that they came from a living tree, which can give the wood used in the interior a more natural look. Knots can also present difficulties in terms of reducing the mechanical strength of wood, which is why too many knots are still undesirable in wooden materials (Broman*,* 2001). Therefore, sawn wood with few knots is sold at higher prices as opposed to that with more knots. Broman (2001) argues that there is a difference in the way people perceive wood materials with knots compared to those without knots. It was proved that the number of knots negatively affects customer preferences. Nakamura and Kondo (2007; 2008) investigated why knots are often perceived as a poor visual feature of a wooden surface. Their research results (Nakamura and Kondo, 2007) showed that there is a linear relationship between subjective visibility and the number of knots and concluded that participants were more relaxed when looking at clean surface wood than the one with knots.

Coherence is another important property of the visual quality of wood. Coherence defines the unity of a material and can be changed and improved by repeating texture and color patterns (Tveit *et al*., 2006). Several studies have shown that coherent or visually harmonious surfaces are important for preferences (Broman, 1995a; Broman, 1995b; Broman, 1996; Broman, 2001; Nyrud *et al.*, 2008). It has been observed that people prefer surfaces with a homogeneous texture that provide the experience of the whole (Kaplan and Kaplan, 1989).

Nakamura *et al.* (2019) conducted two types of psychological measurements (Semantic Differential (SD) and Profile of Mood State (POMS 2)), where subjects were shown two virtual square images of a wooden wall, where the direction of the wood was turned vertically on one side, and on the other horizontally. The interpretation of the results of the SD method on the vertically displayed image of a wooden wall showed a higher level of relaxation among the participants and significantly lower tension and anxiety. In the POMS 2 test, the vertical image had better results on the "strength, activity and friendship" scale. This research suggests that visual stimulation depends on the direction, i.e. the horizontal and vertical position of wooden elements in the interior.

An interesting study was conducted in hospitals, where the experience of the amount of installed wood in the interior was compared using questionnaires with employees (Nyrud *et al.*, 2014). Respondents were shown ten different images of hospital rooms of unique design. The difference was based and measured on the amount of wood in each of the hospital rooms, from a room without wood to a room covered entirely in wooden wall coverings. The results showed that the most desirable design of a hospital room was the one with medium wood use, while a room completely covered with wood paneling was the least desirable. This research has shown that, despite the fact that wood is a natural material, its application is limited and it should be carefully balanced in application and not overdone.

3.2.2 Tactile stimulation
3.2.2. Taktilna stimulacija

Several studies have proven that wood is used not only for furniture but also as a building material in the indoor environment that has a big impact on people's mood (Rice *et al*., 2006; Fleming *et al*. 2013; Lindberg *et al.,* 2013; Strobel and Nyrud, 2017). It makes people feel comfortable and relaxed not only visually, but also by touch. People react positively to wood because it creates a warm atmosphere.

Tactile research of wood has shown that wood has an advantage over other materials because it is characterized by various textures, and due to its constant temperature, it creates a feeling of comfort and security in people. This comparison was shown through the observation of the temperature of a metal handrail that varies depending on the seasons, being hot in summer and cold in winter, which is not the case with wooden handrails whose temperature is consistent throughout the year (Obata *et al.,* 2005).

Wang and colleagues (2000) studied the influence of tactile stimuli on physiological characteristics by comparing skin temperature in relation to the heat flux of the material. These two parameters are related because heat flux affects the thermal conductivity of a material in relation to the temperature difference between the skin and the material with which the skin is in contact. It was noted that wood has significantly lower thermal conductivity than other materials such as tile, marble and concrete. In a separate Finnish study, natural and smooth wooden surfaces were found to be more pleasant than those coated (Bhatta *et al*., 2017).

A similar study was conducted investigating the effect of tactile contact with wood on two indices of physiological stress responses: pulse rate and blood pressure (Morikawa *et al*., 1998). Two types of wood were used - Japanese cedar with a planed and sawn surface and Japanese cypress with a sawn surface. Other materials used in the research were trapper, silk, stainless steel plate and a vinyl bag filled with cold water. The results showed that the tactile experience with denim and stainless-steel plate resulted in a high rate of pulse fluctuation and systolic blood pressure, while the contact with wood caused a low rate of fluctuation.

A study conducted by Sakuragawa and co-authors (2008) examined the effects of tactile contact with various wood materials in relation to changes in blood pressure (the body's response to stress) and subjective evaluation of materials. Wooden samples used Japanese cedar wood, Japanese cypress, oak and urethane-coated oak. Other materials used were aluminum and plastic. The research showed the following: contact with the wooden samples caused the participants to feel comfortable and no increase in blood pressure was observed; contact with chilled wood caused uncomfortable but at the same time natural feeling in the participants, and no increase in blood pressure was observed; and contact with room temperature aluminum or chilled plastic led to artificial and uncomfortable sensations and an increase in blood pressure was observed. The authors concluded that the temperature of the material has a significant effect on increasing blood pressure, while wood, unlike artificial materials, did not cause a psychophysiological reaction to stress.

Tactile stimulation research by Ikei and colleagues (2017a) was conducted in such a way that the sensors for physiological measurements were placed on the forehead of the participants. Eighteen female students participated in the study, and the materials used were white oak, tile, marble, and stainless steel. The results turned out that the only natural material used (white oak) reduced activity in the prefrontal cortex, while other materials did not. A significant increase in parasympathetic nerve activity was also observed, which proves that tactile contact with wooden materials leads to physiological relaxation.

Another study by the same authors (Ikei, *et al.,* 2017b) aimed to examine the cause-and-effect relationships of touching wood samples with different coatings (uncoated wood, vitreous-finished wood, oil-finished wood, mirror-finished wood, urethane-finished wood) on left and right prefrontal cortex activity and autonomic nerve activity. The results showed that uncoated wood and wood treated with oil had the most positive effect on tactile stimulation. These measurements showed that parasympathetic activity increased while prefrontal cortex activity decreased. In other words, these results explain why wood has a physiologically relaxing effect on humans. Some explanations were based on the difference between warm-cold, dry-wet and flat-uneven. However, for "pleasant" and "relaxed" feelings, there was no significant difference in subjective assessment.

The tactile sensation of touching three different surfaces, oiled parquet, lacquered parquet and laminate flooring, was investigated in Austria (Berger *et al*., 2006). After participants tactilely explored the floors with their hands and feet, the results showed that a floor with a naturally oiled surface was perceived as warm, rough and quite soft. Laminate floors were perceived as cool, smooth and hard, and the experience of varnished parquet was quite cold, quite smooth and quite hard. The majority of respondents preferred flooring with a natural oiled surface.

Wood is considered warmer in visual and tactile research compared to stone, plaster, and steel or stone (Wastiels *et al*., 2012). It is interesting to note that in the comparison of different types of wood, it is more difficult to make an estimate of heat, even tactilely (Fujisaki *et al*., 2015).

3.3 Wellbeing
3.3. Dobrobit

Findings from the literature show that the properties of wood affect human behavior in everyday situations. The connection between wood as a material used in the interior with productivity and stress was analyzed in order to find a potential connection to a sense of comfort and wellbeing.

3.3.1 Productivity
3.3.1. Produktivnost

The link between job satisfaction and productivity has been well established in several academic papers. The happier the worker, the more efficient he will be at work (Browning *et al*., 2012). Therefore, it is crucial for organizations to focus on worker satisfaction in order to maximize productivity (Westover *et al*., 2010). Wood in interior design is being increasingly used to achieve diversity in design styles. The use of wood in the interior can make the atmosphere of space from informal, rustic, contemporary to elegant and sophisticated. Nowadays, there is an increasing trend of biophilic design that can be seen in various places such as restaurants, offices, classrooms, and universities (Xue *et al*., 2019). Research has shown that there is a correlation between wood in the interior and productivity. This can be seen in the results that describe reduced stress levels, better cognitive functions, greater creativity and general wellbeing of respondents while staying in rooms equipped with a variety of wooden details. In the design of offices and other workplaces, the role of wood is of great importance as it can improve employees satisfaction and productivity in their workspace (Fell, 2010).

Knox and Parry-Husbands (2018) investigated the existence of a connection between nature, that is the practice of biophilic design in the interior, and the psychophysical reactions of workers who work and act in such an environment. The results showed that there is indeed a link between the use of wood and overall employee satisfaction at work, lower absenteeism, higher levels of concentration and improved productivity. Workers who worked in an environment with less than 20 % wood surfaces were 30 % less satisfied with their jobs than workers who worked in an environment with a high proportion of natural wood surfaces.

A similar study was conducted in New Zealand (Ridoutt *et al*., 2002), where subjects were shown pictures of ten different office interiors that contained wood elements or no wood at all in the space. Respondents had to choose the interior that they can imagine most and least as their workspace and describe it with three different adjectives (out of a total of 24 offered). Offices with a wooden interior were characterized as places that create a sense of comfort and innovation, while offices without wood were described as places of discomfort and unimaginativeness.

Several studies have been conducted in Japan on the impact of wood on quality of life. One study (Anme *et al*., 2012) examined the health and behavior of elderly Japanese residents of a nursing home. Part of the respondents were in daily contact with wooden elements, while the other part used plastic products. The final results showed that those who used wooden products were much more talkative and sociable, and that there was a greater number of interactions that improved their emotional state.

There is a strong argument for the use of wood in the construction of schools and school interiors (Think Wood, 2021). Due to the properties of wood, cost-effective constructions and high-performance buildings are made that are safe, resistant, attractive and enable a healthy environment and the wellbeing of students. Thus, in Japan, a reduced incidence of influenza outbreaks among students in schools with wooden interiors has been reported.

Wood interior design is associated with higher occupant satisfaction. To illustrate, Watchman and colleagues (2017) conducted a post-occupancy survey of occupants in two multifunction rooms. One room had extensive wood finishing, while the other was devoid of wood. The rooms were otherwise similar. Occupants in the room with wood finishing were more satisfied with lighting, noise and temperature. The occupants described the wood room as bright, pleasant, modern and warm. These results obtained by studying the reactions of respondents in an environment where wood is present show that the human physical condition measured by criteria such as blood pressure and human mental condition measured by stress levels are greatly improved. Psychophysiological results on wood in the interior show that the response of the human body to stress is lower and productivity is higher than in the interior where wood is not present.

3.3.2 Stress relief
3.3.2. Ublažavanje stresa

Stress is one of the greatest causes of health problems in humans. It can manifest in many different ways and can also affect the way people function. When people are under stress, they may have difficulty focusing or socializing because they feel more anxious (Kemeny, 2003**)**. Research on the physiological effects of wood is relatively new, but there is now a growing number of studies on this topic.

In a Japan study (Ohta *et al*., 2005**),** the focus was to investigate the impact of wood on physiological and psychological implications on humans. One of the experiments was a comparison in the reactions of the respondents when looking at a wooden or white steel wall. In people who prefer wood as a finishing material, blood pressure dropped significantly when looking at a wooden wall, while in people who are not prone to wood as a building material there were no significant changes in blood pressure. When the viewing of the steel wall was examined, the results were somewhat different. Blood pressure remained unchanged in subjects who liked steel, but in subjects who did not like it an increase in blood pressure was observed.

Tsunetsugu and co-authors (2002) investigated people's reactions to the use of wood in residential areas. Heart rate and blood pressure were measured on subjects who were in rooms with or without wooden surfaces. The blood pressure and heart rate of the participants in the rooms with wood were lower than the values measured before entering the room, while the blood pressure and heart rate of those in the room without wood increased compared to levels measured before entering the room.

A few years later, the same group of researchers (Tsunetsugu *et al*., 2007) investigated changes in respondents who stayed in different rooms. The rooms were specific in that their surface was covered with a different percentage of wood (0 %, 45 % or 90 %). Heart rate and blood pressure were lower in subjects who stayed in a room with 90 % wood than in a room with 0 % wood. The survey found that a room with 45 % coverage was the most optimal because respondents said they felt most comfortable there.

One example is the addition of wood cedar boards and rice straw paper to the walls in hospital rooms (Ohta *et al*., 2008). It was measured that the level of stress (the level of cortisol was measured) was reduced in patients who stayed in these rooms, in contrast to patients who stayed in rooms with concrete walls.

Vast research was conducted in places such as hospitals. One study was conducted in a newly renovated waiting room in Bratislava hospital (Kotradyova *et al*., 2019**),** where respondents' heart rate, cortisol level and blood pressure were measured before, during and after their stay in the wooden room. Respondents described their emotions as predominantly satisfied or very satisfied, and their cortisol levels decreased by 7.5 %, which is evidence of reduced stress due to staying in rooms containing wood elements.

Several studies have also been conducted in schools where the level of stress in students has been examined. One such study took place in Austria, where Kelz and colleagues (2011) studied stress levels in students who attended wood-dominated classrooms and wood-free classrooms. Research has shown that heart rate variability increased during the school year in students who stayed in classrooms where there was no wood. It is an indicator of the activation of the parasympathetic nervous system, which acts in a way that reduces stress levels and promotes the body's healing and recovery functions.

Another one-year Austrian study (Grote *et al*., 2010) also examined stress levels in classrooms with linoleum floors and plasterboard walls and in classrooms with wooden interior on a sample of 52 high school students in a school equipped with two types of classrooms. Students in wooden classrooms had significantly lower heart rates and lower perceptions of stress.

Fell (2010) based his study on the role of wood and plants in reducing stress in the context of an office environment by measuring the two branches of the autonomic nervous system responsible for physiological responses to stress in humans. The respondents were assigned to one of four rooms: a room with wood and plants, a room with only wood but no plants, a room without wood but with plants, and a room without any wood or plants. The results of this study are similar to those conducted by Kelz *et al.* (2011) as it was found that the activation of the sympathetic nervous system was lower in the room where there was wood. The rate of measurable divergent stress thoughts in a wood-based office was half that of a wood-free office (Fell, 2010). Another study carried out in Slovakia (Vavrinsky *et al*., 2019**)** has shown how different colors and textures affect creativity in people by simulating different environments in virtual reality. The study was conducted using BCD applications, and measurements were performed using an EEG helmet and a monitor that measured heart rate and respiratory rate. The results showed that the environment in which both warm and cool colors were present and in which some of the natural materials such as wood and textiles were present positively affected the participants. Apart from the fact that such materials affect relaxation, they also have a great impact on successful problem solving and clarity of thinking. On the other hand, environments in which very bright colors and artificial materials dominated have led to creating stress in participants. People were tested by looking at and touching three walls of different textures. The wooden wall had the most positive effect because it was proven that looking at and touching that wall increased the brain activity of the subjects. Particleboard and white laminate walls did not have such a positive effect. The brownish wooden materials implemented in the room proved to be an ideal choice for a relaxing environment.

Furthermore, the implementation of wood in the interior for wall coverings has a positive effect on users, and especially on the reduction of stress levels as shown by measurable parameters (Rice *et al*., 2006).

3 DISCUSSION AND CONCLUSIONS
3. RASPRAVA I ZAKLJUČCI

The purpose of this paper was to review the results of previous research on the use of solid wood in the interior environment and its effects on the health of humans, as well as to define the main similar parameters by which wood in the interior affects people's behavior and the feeling of comfort and wellbeing.

A systematic review of knowledge about wood used indoors and its impact on health, and thus on the feeling of comfort, is given in Table 1.

Based on the studied literature, solid wood has a positive effect on the cognitive abilities and psychophysical state of humans, and thus on the feeling of comfort and wellbeing in the indoor environment. When there is wood in the environment, people are less exposed to stress and more productive. The balance of moisture in the air created by wood in interiors results in a sense of comfort and air quality. The surface of the wood has much better antibacterial properties than other materials, which is equally important to note considering the current situation in the world and the time we live in. All these qualities are generated through tactile stimulation and visual perception of solid wood. Considering all the above, solid wood in the interior is a desirable material for various wood products.

However, although it is a well-known fact that wood is a natural material that has a positive effect on humans, this review suggests that thirty-one separate studies might not be enough for a general conclusion about the positive aspects of a certain type of wood, i.e. for understanding which aspects of a particular type of solid wood have a stronger or weaker effect on human perception and wellbeing.

By applying the selected keywords, a greater number of papers investigating the subjective aspects of the use of wood in the interior than the objective ones can be seen. This result indicates that the subjective experience of the space and the feeling of comfort in the space ("I feel good") is more related to the psychological subjective perception than to the objective perception of the physical state of the user's body. It can therefore be concluded that it is necessary to combine subjective and objective methods for assessing the feeling of comfort in the environment. Further research should go in the direction of developing subjective questionnaires, models and methodologies that would give more precise results in unique measurements of the feeling of comfort in wooden buildings and interiors. This would provide more unambiguous instructions to designers, architects and manufacturers in the construction of healthy sustainable buildings and interior design.

Further in-depth research on this topic should go in the direction of deepening the understanding of whether solid wood and solid wood products (veneers, plywood, solid boards) increase the user's sense of comfort when installing wooden products made of certain types of wood in interiors of wooden sustainable buildings.

We hope that this work will encourage researchers to do further research on the visual and tactile perception of wood through various wood products in the

Table 1 Findings on wood used in indoor environment and its impact on wellbeing
Tablica 1. Zaključci o drvu koje se upotrebljava u interijeru i njegov utjecaj na dobrobit korisnika

Objective Results *Objektivni rezultati*	**Findings** / *Zaključci*	**Authors** / *Autori*
Air Quality – Humidity *kvaliteta zraka – vlažnost*	wood improves indoor air quality *drvo poboljšava kvalitetu zraka u zatvorenom prostoru*	Hameury and Lundström, 2004; Simonson *et al.,* 2002; Osanyintola and Simonson, 2006
	wood balances relative humidity *drvo uravnotežuje relativnu vlažnost*	Hameury and Lundström, 2004; Li *et al.*, 2012
	reduces heating and cooling, energy consumption *drvo smanjuje potrošnju energije za grijanje i hlađenje*	Osanyintola and Simonson, 2006
Antibacterial effects *antibakterijski učinci*	wood shows antibacterial properties *drvo ima antibakterijska svojstva*	Vainio-Kaila *et al.*, 2017; Laireiter *et al.,* 2013
	wood shows higher antibacterial properties than plastic *drvo ima bolja antibakterijska svojstva od plastike*	Schönwälder *et al.*, 2002; Milling *et al.*, 2005
	untreated wood shows higher antimicrobial activity *netretirano drvo pokazalo je veću antimikrobnu aktivnost*	Kotradyova, *et al.,* 2019; Hedge, 2015
Subjective results *subjektivni rezultati*	**Findings** / *Zaključci*	**Authors** / *Autori*
Visual perception *vizualna percepcija*	medium wood use in space *umjerena upotreba drva u prostoru*	Nyrud *et al.*, 2014; Tsunetsugu *et al.*, 2007
	knot free wood preferred *poželjno drvo bez kvrga*	Nakamura and Kondo, 2008
	vertically arranged wooden grain preferred *poželjniji vertikalni postav smjera teksture drva*	Nakamura *et al.,* 2019
	the color of wood creates warmth *boja drva stvara toplinu*	Masuda, 2004
Tactile stimulation *taktilna stimulacija*	causes less stress than other materials on the living body *uzrokuje manje stresa od drugih materijala na ljudski organizam*	Wang *et al.*, 2000; Morikawa *et al.*, 1998
	uncoated wood shows most positive relaxing effect *nepremazano drvo pokazalo je najpozitivniji opuštajući učinak*	Bhatta *et al.*, 2017; Sakuragawa *et al.*, 2008; Ikei *et al.,* 2017a; Berger *et al.*, 2006
Overall wellbeing results *ukupni rezultati dobrobiti*	**Findings** / *Zaključci*	**Authors** / *Autori*
Productivity *produktivnost*	better working conditions in view of lighting, noise and temperature *bolji radni uvjeti s obzirom na osvjetljenje, buku i temperaturu*	Watchman *et al., 2017*
	improves satisfaction in workplace *pospješuje zadovoljstvo u radnom prostoru*	Bergs, 2002; Knox and Parry-Husbands, 2018; Ridoutt *et al.*, 2002; Watchman *et al., 2017*
	improves emotional state of elderly *poboljšava emocionalno stanje starijih osoba*	Anme *et al.*, 2012
Stress relief *oslobađanje od stresa*	visual and tactile stimulation of wooden surfaces increases brain activity and promotes relaxation *vizualna i taktilna stimulacija drvenim površinama povećava aktivnost mozga i potiče opuštanje*	Vavrinsky *et al.*, 2019
	visual stimulation of wooden surfaces reduces blood pressure and stress *vizualna stimulacija drvenim površinama smanjuje krvni tlak i stres*	Ohta *et al.*, 2005; Ohta *et al.*, 2008; Kelz *et al.*, 2011; Grote *et al.*, 2010

interiors of sustainable buildings. All these findings can be a potential guide for greater use of wood in the interior design of sustainable timber buildings by wood processors, manufacturers, architects, and interior designers as well as for more vital branding of wood products in the market with the aim of increasing wellbeing in the indoor environments with an emphasis on furnishing sustainable public facilities.

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HRVATSKA KOMORA INŽENJERA ŠUMARSTVA I DRVNE TEHNOLOGIJE

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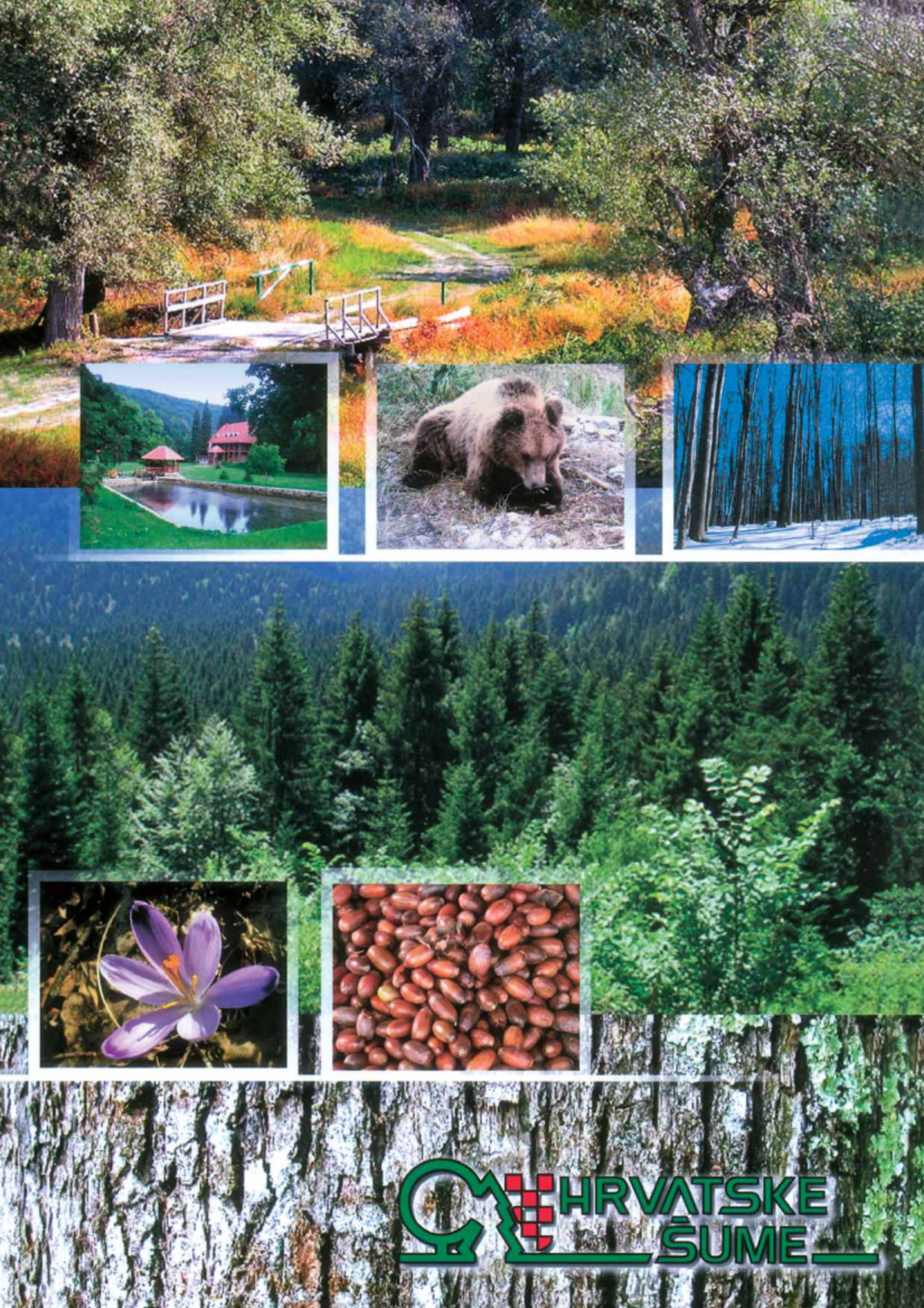
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