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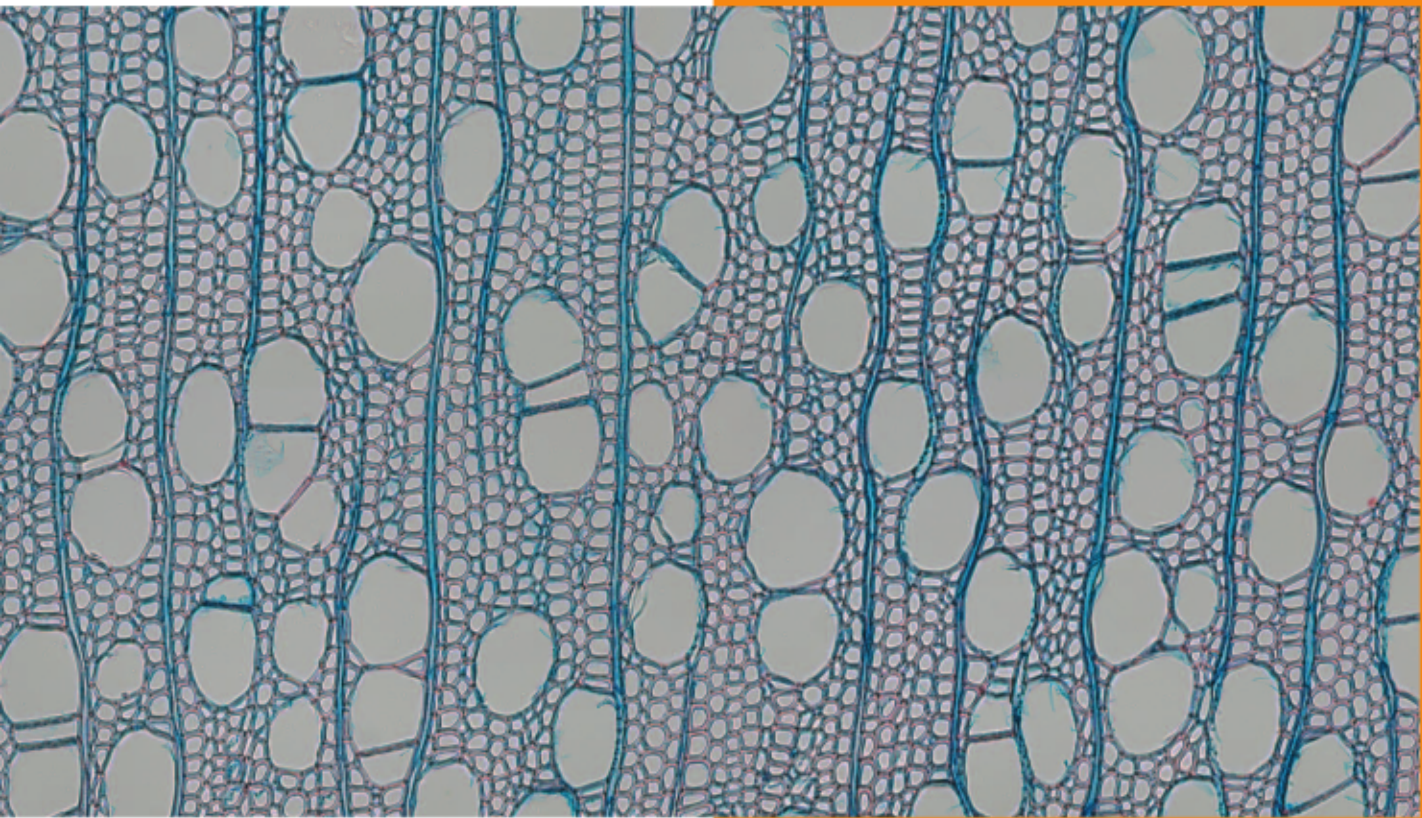
SCIENTIFIC JOURNAL
OF WOOD TECHNOLOGY



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Abdi Atilgan[1], Sait Dundar Sofuoglu[2]

The Effect of Hot and Cold Check Tests on Surface Roughness and Glossiness in Varnished Wood Material

Utjecaj testa vruće i hladne provjere na hrapavost i sjaj površine lakiranog drva

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ABSTRACT • *In this study, specimens from Scots pine (Pinus sylvestris L.), Anatolian chestnut (Castanea sativa Mill.) and Eastern beech (Fagus orientalis Lipsky) tree species, prepared according to ISO 3129, were conditioned according to TS ISO 13061-1. Cellulosic varnish, water-based varnish, and glass polish varnish were applied to their surfaces in accordance with ASTM-D 3023 principles. In the study, roughness (TS 2495 EN ISO 3274 and TS EN ISO 21920-3) and gloss (according to TS EN ISO 2813) values of the samples were calculated after hot and cold check test. Varnished test specimens prepared in 100 mm × 100 mm ×10 mm dimensions were first kept in drying oven at (50±5) °C for 1 hour, then were kept in conditioning room for 1 hour, and then at (-20±2) °C for 1 hour, according to ASTM D1211-97. These processes were accepted as one cycle, and tests were continued until 15 cycles. Afterward, glossiness was measured as perpendicular and parallel to fibers at 60° with a gloss measurement device, and surface roughness values of Ra and Rz were determined with a surface roughness measuring device. According to the results obtained, Scots pine (Pinus sylvestris L.), Eastern beech (Fagus Orientalis Lipsky), and Anatolian chestnut (Castanea sativa Mill.) varnish-coated wood material surfaces all showed a decrease in gloss values after hot-cold shock effect. While an increase occurred in Rz values of roughness for all wooden surfaces, Ra values roughness increased for Scots pine and chestnut and decreased for eastern beech. Gloss and roughness values of surface-treated wood materials against changing weather conditions can determine usefulness of the surface material used. The findings obtained in this study can be useful to manufacturers who use wooden products in the design of marine vehicles and those who export furniture to countries in different climatic conditions.*

KEYWORDS: *wood material; varnish; surface roughness; glossiness; hot and cold-check test*

SAŽETAK • *U ovom su istraživanju uzorci drva bora (Pinus sylvestris L.), anatolskog kestena (Castanea sativa Mill.) i bukve (Fagus orientalis Lipsky) pripremljeni prema ISO 3129 i kondicionirani prema TS ISO 13061-1, a na njihovu su površinu, prema ASTM D 3023, naneseni nitrocelulozni lak, vodeni lak i lak visokog sjaja. U istraživanju su izračunane vrijednosti hrapavosti (TS 2495 EN ISO 3274 i TS EN ISO 21920-3) i sjaja (TS EN ISO 2813) uzoraka nakon testa vruće i hladne provjere. Lakirani uzorci dimenzija 100 mm × 100 mm ×10 mm najprije su jedan sat sušeni pri (50±5) °C, zatim su jedan sat kondicionirani pri sobnim uvjetima i jedan sat pri (-20±2) °C, prema ASTM D1211-97. Ti procesi sušenja i kondicioniranja čine jedan ciklus, a ispitivanje se sastojalo od*

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15 ciklusa. Nakon toga izmjeren je sjaj okomito i paralelno s vlakancima drva pod kutom od 60° u odnosu prema mjernom uređaju te su određeni parametri hrapavosti površine Ra i Rz. Prema dobivenim rezultatima, svi su lakirani uzorci drva bora (Pinus sylvestris L.), bukve (Fagus orientalis Lipsky) i anatolskog kestena (Castanea sativa Mill.) nakon testa hladne provjere pokazali smanjenje vrijednosti sjaja. Međutim, na svim površinama uzoraka drva zabilježeno je povećanje Rz vrijednosti hrapavosti, dok su se Ra vrijednosti hrapavosti povećale na borovini i kestenovini, a smanjile na bukovini. Vrijednosti sjaja i hrapavosti površinski obrađenih drvnih materijala mogu odrediti njihovu uporabljivost u promjenjivim okolišnim uvjetima. Rezultati dobiveni u ovom istraživanju bit će korisni proizvođačima koji upotrebljavaju drvo u proizvodnji brodova ili izvoze namještaj u zemlje s drugačijim klimatskim uvjetima.

KLJUČNE RIJEČI: *drvni materijal; lak; hrapavost površine; sjaj; test vruće i hladne provjere*

1 INTRODUCTION
1. UVOD

Wood is a renewable material very important for human beings, who have used it in art and architecture from the past to the present. Today, protecting the properties of wood from external factors and providing hygienic conditions has become one of the most critical problems (Atilgan, 2022). In the use of wood material after it is finished, top surface materials such as paint, varnish and impregnation applied to wood surfaces are required to maintain its strength and aesthetic properties (Goktas *et al*., 2006).

Wood material might be easily deformed by some external factors. Polymer structures such as lignin and cellulose, which make up the wood material, are affected by ultraviolet rays like other organic polymer structures. Negative changes occur in almost all physical and mechanical properties of wood material due to exposure to outdoor conditions without any protective treatment (Pelit and Korkmaz, 2019). Protective treatment is necessary for the long-term and efficient use of wood and wood-based materials for protection. In wooden materials, these processes include impregnation, varnishing and painting (Vardanyan *et al*., 2015). In the protection process, a protective layer is created, using materials with the feature of layering to protect furniture and decoration elements against physical, mechanical, and chemical effects, outdoor conditions, and biological pests. It is applied in the form of covering wood material surfaces (Sonmez, 2000). In order to extend the aesthetic and economic life of the surfaces, paints and varnishes are the most used materials in creating a protective layer with liquid surface treatments (Kurtoglu, 2000). The most inconvenient factors for wood material in outdoor conditions are temperature, humidity, different wavelengths of sunlight, and UV radiation, and changing of these conditions at certain times of the day according to the season cause negative effects on the wood material (Feist and Hon, 1984). Varnish types and varnishing techniques should be chosen appropriately. The tests applied to determine the resistance of paint and varnish layers against external effects were applied to determine the performance of varnish systems or to develop products. Today, many surface treatment materials and many application methods have been developed for surface treatments (Ozdemir, 2003). Surface treatment and selection of a protective layer are very important for a long-term use of wood material (Ulay ve Budakci, 2015). There are many studies in the literature about varnish, which is one of the wood finishing materials used in the woodworking and furniture industry. The highest hardness value on varnished surfaces was obtained with polyester varnish. The glossiest surfaces were obtained with polyurethane varnish (Sonmez, 2000; Cakicier, 1994). When Scots pine and chestnut tree species were used in outdoor conditions after impregnation and varnishing, higher hardness values were obtained with polyurethane varnish than with synthetic varnish in both tree species (Peker, 1997). When examining the effect of layer thickness on wood varnishes, it was seen that the third layer varnish applications caused an increase in glossiness (Budakci, 1997). It was observed that the glossiness of water-based varnishes was lower than that of solvent-based varnishes (Yakin, 2001). Wood species were found to be insignificant for the glossiness of different varnish layers, while the varnish effect was significant (Budakci, 2003). The layer increase in varnish applications negatively affects the flexibility properties of the varnish layer. The layer increases the surface tension and causes exfoliation (Cakicier, 2007). Gurleyen *et al*. (2017) analyzed glossiness values on parquets with UV varnish applied according to different varnish layers produced from limba, sapele, chestnut, and iroko woods (Gurleyen *et al*., 2017). Dongel *et al*. (2008) investigated the glossiness effect of dry heat on solid wood and wood-based flooring materials. Eastern beech solid parquet covered with polyurethane parquet varnish, laminated parquet covered with UV-curable polyurethane varnish, and laminate parquet with high-density fiberboard (HDF) in the middle layer were used as test samples (Dongel *et al*., 2008). In addition, Gurleyen *et al*. (2017) determined the glossiness values in single and double UV system parquet varnish layers applied to rowan (*Sorbus* L.) wood (Gurleyen *et al*., 2017).

In the work of Sonmez and Kesik (1999), test samples were prepared by using beech (*Fagus orientalis* L.), pine (*Pinus sylvestris* L.) and oak (*Quercus petreae* L.) wood, and cellulosic, polyurethane, and acrylic varnishes were applied to the surface of sample panels. Samples were first kept at -18 °C for one hour and then removed and kept at 50 °C for one hour. These processes were accepted for one cycle and tests continued for 20 cycles. Tests were carried out according to ASTM D-1211. There was no visible failure on the sample panels. However, all the sample panels lost some hardness. On the other hand, in the case of glossiness measurement, the glossiness of oak panels coated with cellulosic varnish was increased, but the glossiness of other panels was decreased. (Sonmez and Kesik, 1999). It was also reported that, when Gubas solid wood material was subjected to the hot-cold check test applied to its surface varnish (acidic hardener and cellulosic), the surface with cellulosic varnish exhibited fracture, cracking, and color change (Yolanda, 1998).

In his study, Altiparmak (2017) observed the deformation of the surfaces after the hot-cold shock effect of yacht varnish, polyurethane varnish, and epoxy varnish applied on limba, chestnut, and sapele woods. No cracking or surface deformation was observed on the panel surfaces in the 20-cycle cold-check test (Altiparmak, 2017). In the study of Budakci *et al*. (2010), cellulosic, polyurethane and acrylic varnishes were applied on oriental beech, yellow pine and sessile oak, and color changes were determined after the effects of hot and cold shocks after accelerated aging effects (Budakci *et al*., 2010). It was stated that, in the hot-cold aging tests of the varnish layers, the samples should be kept at temperatures of -20 and 50 °C for 1 h each, and this process should be considered as 1 period; the performance of the layers with no degradation in 10 periods should be considered as sufficiently good, while layers with no degradation in 25 periods should be considered as high-performance (Payne, 1965).

In some areas where wood materials with wood finishing are used, sudden temperature changes occur, which can cause the painted and varnished surface to lose its properties. It is important to determine the effect of these changes on the wood species and varnishes used in places with sudden climate changes. However, when the literature is examined, few studies can be found.

Based on the data obtained from the surface roughness and glossiness change on varnished surfaces that may be exposed to the effects of sudden climatic changes (hot and cold), it will be possible to select the appropriate wood type and varnish for the place of use.

Wood species of Scots pine (*Pinus sylvestris* L.), Anatolian chestnut (*Castanea sativa* Mill.), and Eastern beech (*Fagus orientalis* L.) and Cellulosic, water-based, and glass polish varnishes, which are used in the woodworking industry and manufacture of various marine vehicles, were used in the present study. These tree species and varnishes, which are frequently used, can be deformed in environments where they are exposed to sudden temperature changes (especially marine vehicles, yachts, etc.). The aim of this study was to determine the effect of the Hot and Cold-Check Test on the roughness and gloss changes on the surfaces and to specify the most suitable wood type and varnish.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

In this study, Scots pine (*Pinus sylvestris* L.), Anatolian chestnut (*Castanea sativa* Mill.), and Eastern beech (*Fagus orientalis* L.) tree species, which are widely used in the woodworking industry and yacht decoration applications, were preferred.

Cellulosic varnish, water-based varnish and glass polish varnish were used in the experiments. The varnishes used in the experiments were obtained from Çaglayan Wood Products and Hardware Industry Afyonkarahisar/Turkey (Table 1).

Timber used in the preparation of the samples were obtained by random selection from companies in Afyonkarahisar/Turkey. Experiment samples were prepared from sapwood not damaged by insects and fungi, free of knots, cracks, and inclusions, with no discoloration and a smooth fiber structure, according to ISO 3129 (ISO 3129, 2019).

According to ISO 3129, 3 wood species, three varnish types, and five replications for each parameter, i.e. 45 test samples were prepared. Measurements were made from 3 different points on each test sample, and 135 data were obtained for each type of test. The test samples were prepared in the dimensions of 100 mm × 100 mm × 10 mm (longitudinal direction × radial direction × tangential direction) in Afyon Kocatepe Uni-

Table 1 Characteristics of varnishes used
Tablica 1. Svojstva primijenjenih lakova

Type of varnish *Vrsta laka*	**Solid ratio, %** *Udio suhe tvari, %*	**Density at 20 °C, g/cm³** *Gustoća pri* 20 °C, g/cm³	**Viscosity at 20 °C, s** *Viskoznost pri* 20 °C, s
Cellulosic / *nitrocelulozni lak*	34-40	0.92-0.99	100-110 (DIN6 mm)
Water-based / *vodeni lak*	32-38	1.02-1.04	30-40 (DIN4 mm)
Glass polish varnish / *lak visokog sjaja*	47-51	0.92-0.94	90-140 (DIN6 mm)

Table 2 Assignment of levels to factors (parameters used)
Tablica 2. Dodjeljivanje razina faktorima (korišteni parametri)

Parameter / *Parametar*	**Coded levels** / *Odabrane razine*		
	Level 1	**Level 2**	**Level 3**
Wood species *vrsta drva*	Scots pine *borovina*	Eastern beech *bukovina*	Anatolian chestnut *drvo anatolskog kestena*
Type of varnish *vrsta laka*	Cellulosic *nitrocelulozni lak*	Water-based *vodeni lak*	Glass polish varnish *lak visokog sjaja*
Hot and cold shock test *test vruće i hladne provjere*	Before *prije*	After *poslije*	

versity Afyon Vocational School Furniture and Decoration Workshop. They were conditioned at temperatures of (20±2) °C and (65±5) °C, with a moisture content (*MC*) of about 12 %. (TS ISO 13061-1, 2021). The surfaces of the samples were sanded with 80 and 120 grit sandpapers, respectively. The varnishing process of the test samples was carried out by the principles specified in ASTM-D 3023 (ASTM-D3023, 2017). The dust on the surface was cleaned with compressed air. The varnishes were applied in two layers at 2 (27.5 bar) atm pressure (DYO, 1990) with an air gun with a tip opening of 1.8 mm, perpendicular and parallel to the fibers, at 125 g/m^2. After 1 coat of application, the samples were sanded with 400-grit sandpaper. After sanding, the varnish was applied again. Table 2 gives the parameters and levels used in the application.

The experimental process of the study is given in Figure 1.

This test is applied to wood material that has been painted and varnished according to ASTM-D 1211 by exposing it to shock, heat and cold. Its purpose is to determine the varnish layer performance in sudden temperature changes that occur in natural climatic conditions. Hot-cold tests indicate the flexibility of transparent varnish layers on wooden surfaces or their resistance to sudden temperature changes (Sonmez and Kesik,1999). In this context, surface-treated test specimens prepared in dimensions of 100 mm × 100 mm ×10 mm were exposed to cold check test for periods of 1 h (50±5) °C, 1 h laboratory conditions, and 1 h (-20±2) °C, for 15 cycles according to ASTM D 1211-97 (2001) standard.

Surface Roughness Tester Time TR220 (Time Group Inc., China) type surface roughness measurement equipment was used for the determination of surface roughness values via a contact stylus trace method. The sampling length was taken as 0.8 mm. The stylus probe speed was chosen as 10 mm/min, the diameter of the measurement needle was 5 µm, and the needle tip was 90°. Care was taken to have a measurement environment around (18–22) °C and without vibrations. Perpendicular and parallel glossiness measurement of the fibers was made. The tool was calibrated before measurement, and calibration was checked at established intervals. The roughness of the wood material after the effect of hot-cold shock was determined according to TS 2495 EN ISO 3274 and TS 6212 EN ISO 4288.

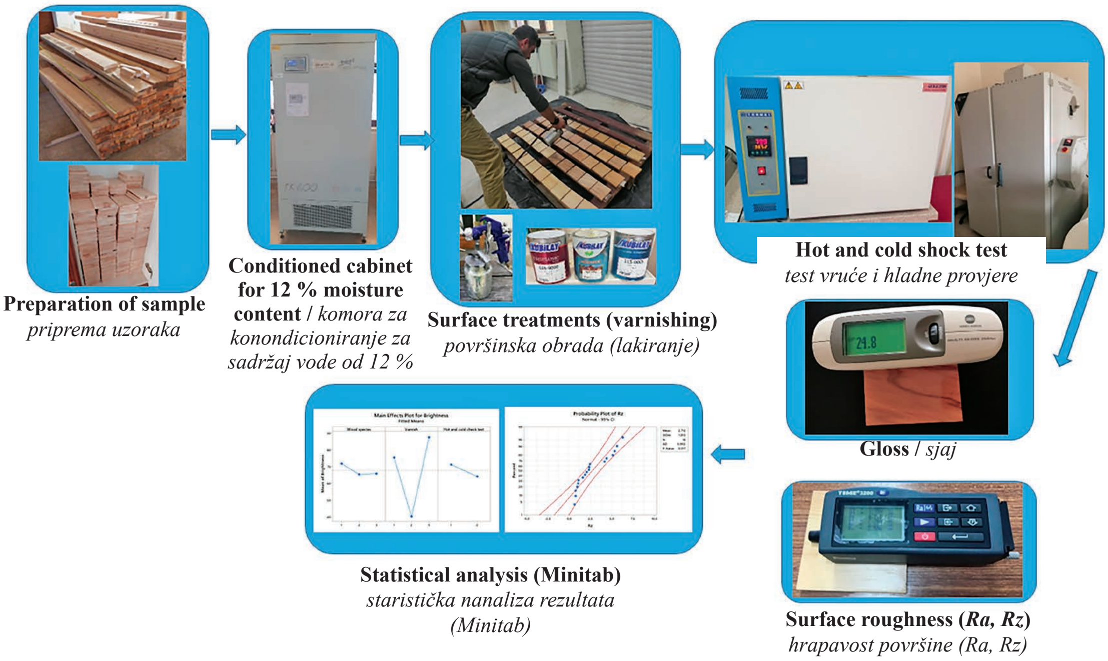

Figure 1 Schematic representation of experimental design
Slika 1. Shematski prikaz eksperimenta

In the study, glossiness measurements were carried out using a Glossmeter at 60°. Not many errors were observed when measuring at 60° on matte and glossy surfaces (Ozen and Sonmez, 1990; Ordu and Sofuoglu, 2016). Glossiness measurements were carried out using Konica Minolta Multi Gloss 268 Plus (Glossmeter) gloss meter according to the principles specified in TS 4318 EN ISO 2813.

For the evaluation of the data, the Minitab 19 statistical software program was used. The values of the factor effects of wood species, varnish types, and hot-cold shock test were determined using the analysis of one-way variance (ANOVA) and the Tukey procedure. The differences in the means were accepted at a significance of $P<0.05$.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

Experiments were carried out to determine the effect of wood species, varnish types, and hot-cold shock test on roughness parameters (*Ra*, *Rz*) and surface glossiness of the samples. Roughness parameters and glossiness values measured on surfaces are given in Table 3.

Since the *P* value is greater than 0.05 (P=0.102) according to Figure 2, it is seen that the values of the *Ra* measurement show a normal distribution at the 95 % confidence level. Analysis of variance results for *Ra* is given in Table 4.

According to the results of a one-way analysis of variance for *Ra* (at 95% confidence level, Table 4),

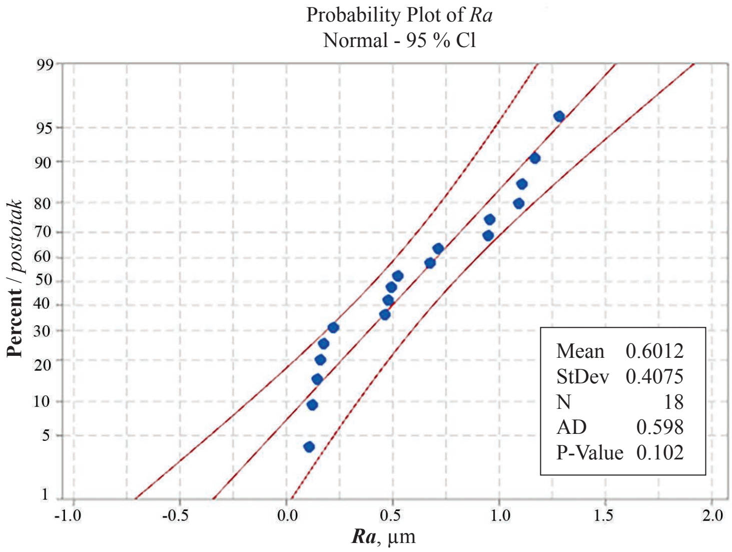

Figure 2 Normality graphs for *Ra*
Slika 2. Grafovi normalne distribucije za *Ra*

Table 3 Roughness parameters (*Ra* and *Rz*) and gloss values measured on surfaces
Tablica 3. Parametri hrapavosti (*Ra* i *Rz*) i vrijednosti sjaja izmjerene na površinama

Wood species *Vrsta drva*	**Varnish** / *Lak*	**Hot and cold shock test** *Test vruće i hladne provjere*	***Ra*, µm**	***Rz*, µm**	**Glossiness** *Sjaj*
Scots pine *borovina*	Cellulosic *nitrocelulozni lak*	Before / *prije*	0.475	1.595	87.753
		After / *poslije*	0.489	1.854	77.007
	Water-based *vodeni lak*	Before / *prije*	0.959	4.147	44.827
		After / *poslije*	1.169	4.408	43.507
	Glass polish varnish *lak visokog sjaja*	Before / *prije*	0.118	0.742	91.520
		After / *poslije*	0.175	0.771	85.927
Eastern beech *bukovina*	Cellulosic *nitrocelulozni lak*	Before / *prije*	0.526	2.047	81.513
		After / *poslije*	0.465	2.337	63.593
	Water-based *vodeni lak*	Before / *prije*	1.092	5.347	36.713
		After / *poslije*	0.948	5.600	36.447
	Glass polish varnish *lak visokog sjaja*	Before / *prije*	0.217	1.049	89.953
		After / *poslije*	0.159	1.090	84.233
Anatolian chestnut *drvo anatolskog kestena*	Cellulosic *nitrocelulozni lak*	Before / *prije*	0.676	2.337	72.053
		After / *poslije*	0.716	2.433	70.40
	Water-based *vodeni lak*	Before / *prije*	1.105	5.131	45.047
		After / *poslije*	1.287	6.309	34.540
	Glass polish varnish *lak visokog sjaja*	Before / *prije*	0.106	0.626	92.160
		After / *poslije*	0.140	0.962	81.253

Table 4 Results of one-way analysis of variance for *Ra*
Tablica 4. Rezultati jednosmjerne analize varijance za *Ra*

Source / *Izvor*	*DF*	*Adj SS*	*Adj MS*	*F* Value	*P* Value
Wood species / *vrsta drva*	2	0.04470	0.02235	0.12	0.887
Error	15	2.77871	0.18525		
Total	17	2.82341			
Varnish / *lak*	2	0.6724	1.33622	132.77	0.000
Error	15	0.1510	0.01006		
Total	17	2.8234			
Hot and cold check test *test vruće i hladne provjere*	1	0.00417	0.004171	0.02	0.880
Error	16	2.81924	0.176203		
Total	17	2.82341			

there is a significant difference since *P*=0.000 for the varnish type. It is seen that tree species (*P*=0.887>0.05) and hot-cold test (*P*=0.880>0.05) did not make any significant difference. Tukey test was applied for the varnish type, and it was seen that each varnish type formed a separate group (Table 5).

Table 5 Results of Tukey test for *Ra*
Tablica 5. Rezultati Tukey testa za *Ra*

Varnish *Lak*	*N*	Mean *Srednja vrijednost*	Grouping *Grupiranje*
2	6	1.0933	A
1	6	0.5578	B
3	6	0.1525	C

Figure 3 presents the main effects plot in terms of *Ra* of wood species, varnish types and hot-cold check test.

According to the main effect plot created for *Ra*, it was observed that the highest roughness value occurred in Anatolian chestnut tree species, while lower and close values were obtained in other tree species. In terms of varnish type, the *Ra* roughness value from the lowest is listed as glass polish, cellulosic and water-based. Although the values were close to each other before and after the test in terms of hot and cold-check test, there was a slight increase in the roughness value after the test.

When the interaction graph is examined in terms of R_a (Figure 4), it is seen that all three tree species have similar results in terms of applied varnish. The lowest roughness value was obtained in glass polish, followed by cellulosic and synthetic varnish, respectively. When the wood type and hot-cold test interaction were examined, a slight increase was observed in the roughness value after the test in Scots pine and Anatolian chestnut tree species, while a decrease occurred in Eastern beech tree species. When the interaction between the varnish type and the hot-cold test was examined, no change was observed in the post-test R_a value in cellulosic and glass lacquer varnish types, while an increase occurred in the water-based varnish type after the cold-hot test.

Since the *P* value is less than 0.05 (*P*=0.017) according to Figure 5, it is seen that the values in the *Rz*

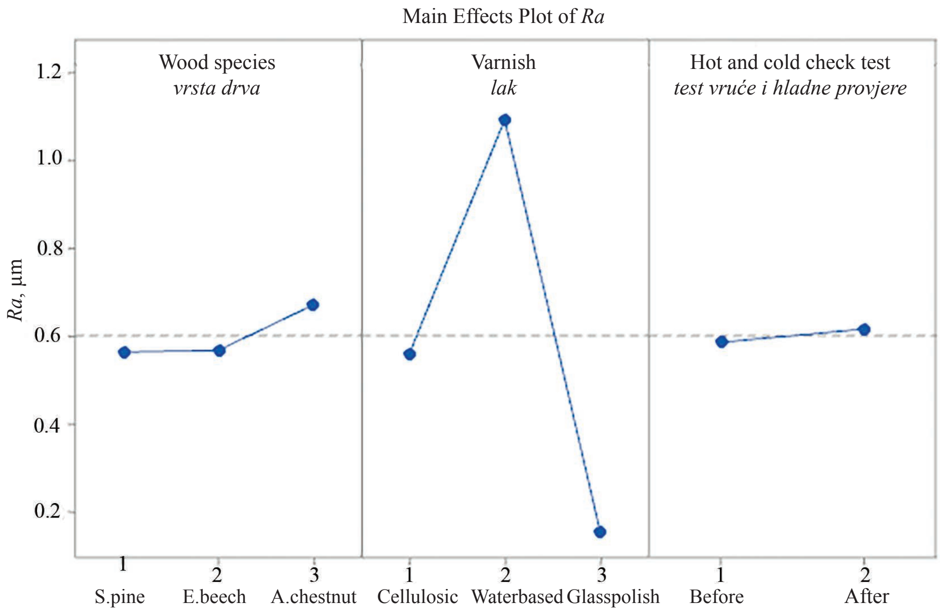

Figure 3 Main effects plot in terms of *Ra* of wood species, varnish types, and hot-cold check test
Slika 3. Dijagram glavnih učinaka na *Ra* s obzirom na vrstu drva, vrstu laka i test vruće i hladne provjere

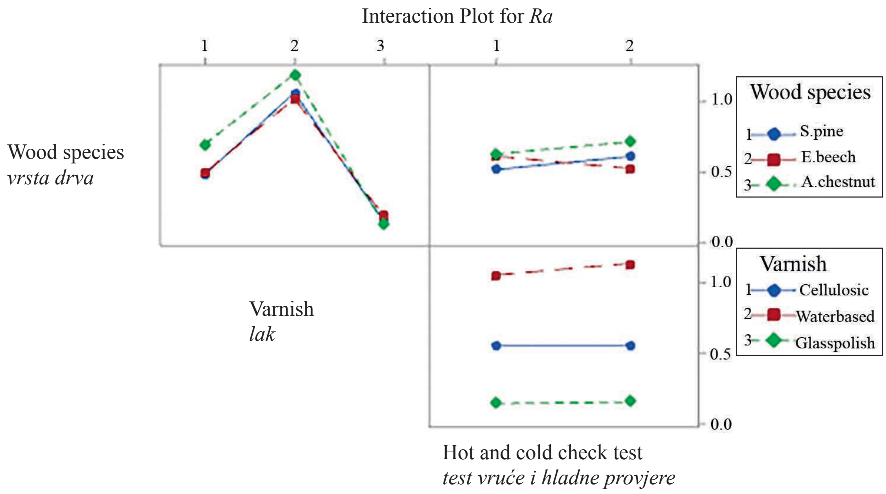

Figure 4 Interactions of wood species, varnish, and hot-cold check test of *Ra*
Slika 4. Interakcije vrsta drva, laka i testa vruće i hladne provjere za *Ra*

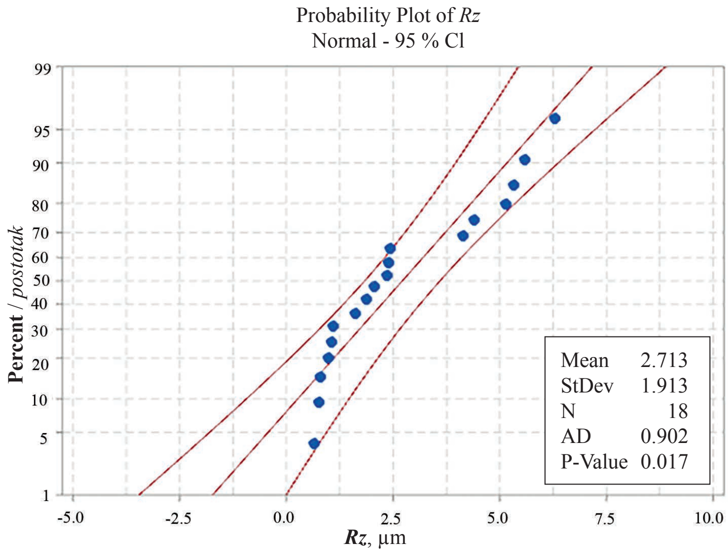

Figure 5 Normality graphs for *Rz*
Slika 5. Grafovi normalne distribucije za *Rz*

measurement did not show normal distribution at the 95 % confidence level. The analysis of variance results for *Rz* is given in Table 6.

According to Table 6, there is a significant difference for *Rz* as *P*=0.000 for the varnish type according to the results of a one-way analysis of variance at a 95% confidence level. It is seen that tree species (*P*=0.792>0.05) and hot-cold test (*P*=0.743>0.05) did not make any significant difference. Tukey test was applied for the varnish type in terms of *Rz*, and it was seen that each varnish type formed a separate group (Table 7).

According to the main effect plot created for *Rz*, it was observed that the lowest roughness value occurred in Scots pine tree species, while higher and

Table 6 Results of one-way analysis of variance for *Rz*
Tablica 6. Rezultati jednosmjerne analize varijance za *Rz*

Source / *Izvor*	*DF*	*Adj SS*	*Adj MS*	*F* Value	*P* Value
Wood species / *vrsta drva*	2	1.909	0.9543	0.24	0.792
Error	15	60.316	4.0211		
Total	17	62.225			
Varnish / *lak*	2	58.347	29.1736	112.85	0.000
Error	15	3.878	0.2585		
Total	17	62.225			
Hot and cold check test *test vruće i hladne provjere*	1	0.4303	0.4303	0.11	0.743
Error	16	61.7946	3.8622		
Total	17	62.2249			

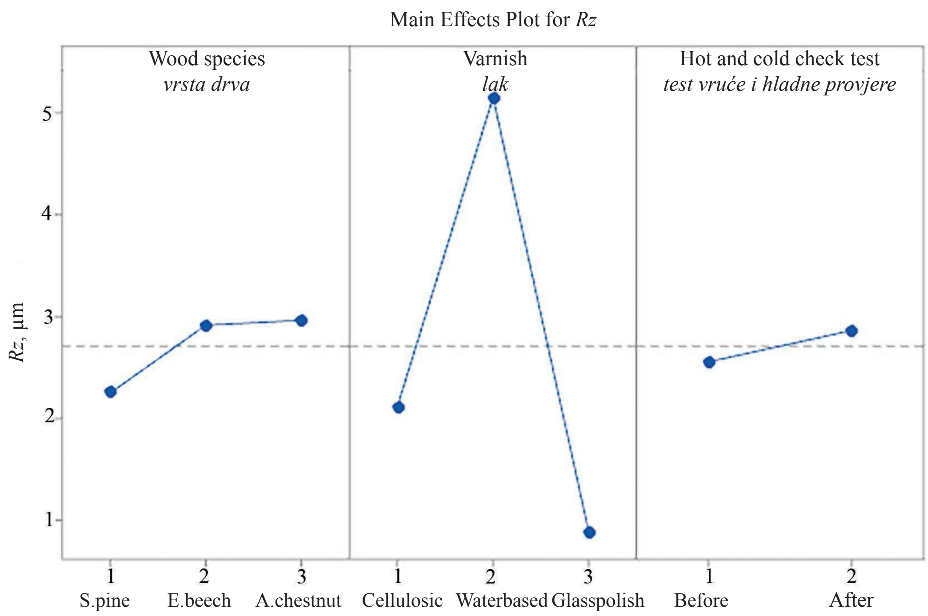

Figure 6 Main effects plot in terms of *Rz* of wood species, varnish types, and hot-cold check test
Slika 6. Dijagram glavnih učinaka na *Rz* s obzirom na vrstu drva, vrstu laka i test vruće i hladne provjere

close values were obtained in other tree species. In terms of varnish type, the *Ra* roughness value from the lowest is listed as glass polish, cellulosic and water-based. Although the values were close to each other before and after the test in terms of the hot and cold-check test, there was a slight increase in the *Ra* roughness value after the test.

When the interaction graph is examined in terms of *Rz*, it is seen that all three tree species have similar results in terms of the applied varnish, as in the *Ra* value. The lowest roughness value was obtained in glass polish, followed by cellulosic and synthetic varnish, respectively. When the interaction between the tree species and the hot-cold test was examined, there was a slight increase in the roughness value after the test in all three tree species. When the interaction between the varnish type and the hot-cold test was examined, it was observed that the *Ra* value of cellulosic and glass varnish, varnish types, increased slightly after the test, while an increase was observed in the water-based varnish type after the cold-hot test. As a result of sudden temperature changes, roughness parameters (*Ra* and *Rz*) increased in Scots pine and Anatolian chestnut tree species for all three varnish types. The literature states that the molecular sizes of water-based varnishes are smaller than those of solvent-based systems; therefore, they penetrate more into the cavities of the wood material, thus giving thin layers (Sonmez *et al*., 2004).

Table 7 Results of Tukey test for *Rz*
Tablica 7. Rezultati Tukey testa za *Rz*

Varnish *Lak*	*N*	**Mean** *Srednja vrijednost*	**Grouping** *Grupiranje*
2	6	5.157	A
1	6	2.107	B
3	6	0.8733	C

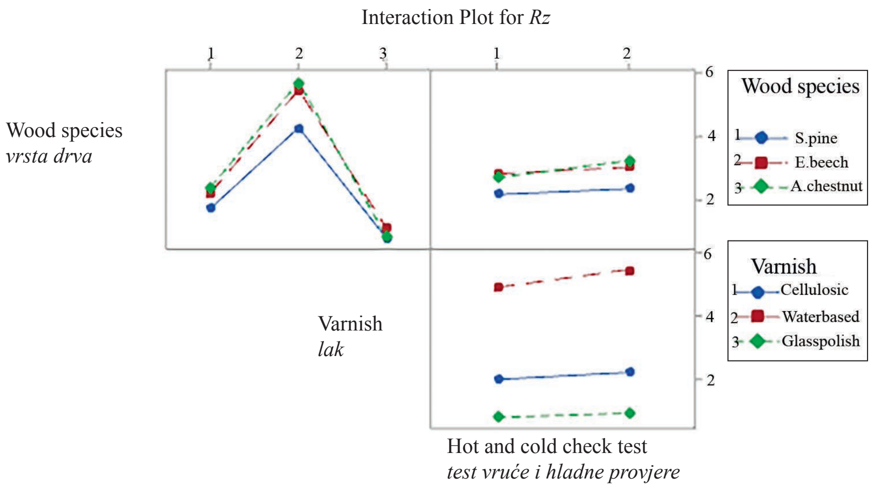

Figure 7 Interactions of wood species, varnish, and hot-cold check test of *Rz*
Slika 7. Interakcije vrste drva, laka i testa vruće i hladne provjere za *Rz*

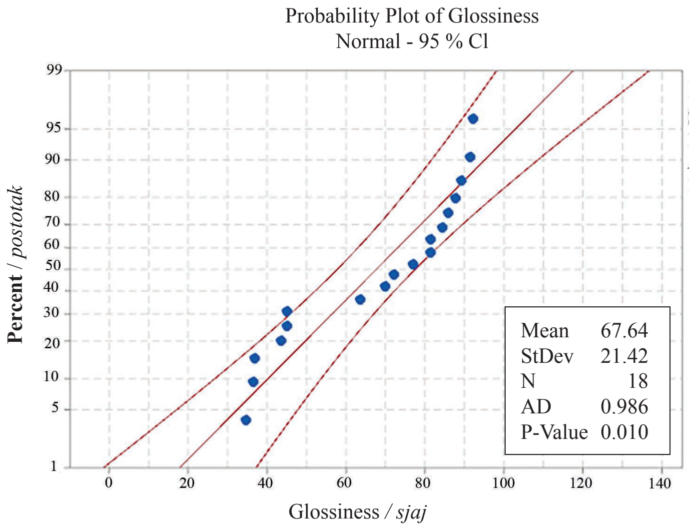

Figure 8 Normality graphs for glossiness
Slika 8. Grafovi normalne distribucije za sjaj

Table 8 Results of one-way analysis of variance for glossiness
Tablica 8. Rezultati jednosmjerne analize varijance za sjaj

Source / *Izvor*	*DF*	*Adj SS*	*Adj MS*	*F* Value	*P* Value
Wood species / *vrsta drva*	2	153.6	76.78	0.15	0.862
Error	15	7649.2	509.94		
Total	17	7802.7			
Varnish / *lak*	2	7223.3	3611.66	93.50	0.000
Error	15	579.4	38.63		
Total	17	7802.7			
Hot and cold check test *test vruće i hladne provjere*	1	230.4	230.4	0.49	0.495
Error	16	75.72.4	473.3		
Total	17	7802.7			

The low layer thickness reveals the effect of the texture of the wood material and may cause higher roughness (Table 3).

The normality graph for glossiness is given in Figure 8.

Since the *P* value is less than 0.05 (*P*=0.010) according to Figure 8, it is seen that the values in the glossiness measurement did not show normal distribution at the 95 % confidence level. Variance analysis results for glossiness are given in Table 8.

Based on Table 8 (according to the results of a one-way analysis of variance at a 95 % confidence level), there is a significant difference in terms of varnish type for glossiness as *P*=0.000. It is seen that tree species (*P*=0.862>0.05) and hot-cold test (*P*=0.495>0.05) did not make any significant difference. Tukey test was applied for the varnish type in terms of glossiness, and it was seen that each varnish type formed a separate group (Table 9).

According to the main effect plot created for glossiness (Figure 9), the highest glossiness value occurred in Scots pine tree species, while lower and close values were obtained in other tree species. Glossiness value in terms of varnish type is listed from the lowest to the highest as water-based, cellulosic, and glass polishes. Although the values were close to each other before and after the test in terms of hot and cold-check test, there was a decrease in the glossiness value after the test.

When the interaction graph is examined in terms of glossiness, it is seen that all three tree species have similar results according to the varnish applied. The lowest glossiness value was obtained in water-based varnish in every tree species, followed by cellulosic and glass varnish, respectively. Considering the wood type and the interaction of the hot-cold test, a slight decrease in the post-test glossiness value occurred in each tree species. When the interaction between the varnish type and the hot-cold test was examined, a decrease in the glossiness value after the test occurred in each type of varnish applied. Glossiness values were listed from the highest to the lowest according to the varnish type applied as glass polish, cellulosic and water-based. This sequence did not change before and after the hot-cold test.

Table 9 Results of Tukey test for glossiness
Tablica 9. Rezultati Tukey testa za sjaj

Varnish *Lak*	N	**Mean** *Srednja vrijednost*	**Grouping** *Grupiranje*		
3	6	87.41	A		
1	6	75.33		B	
2	6	40.18			C

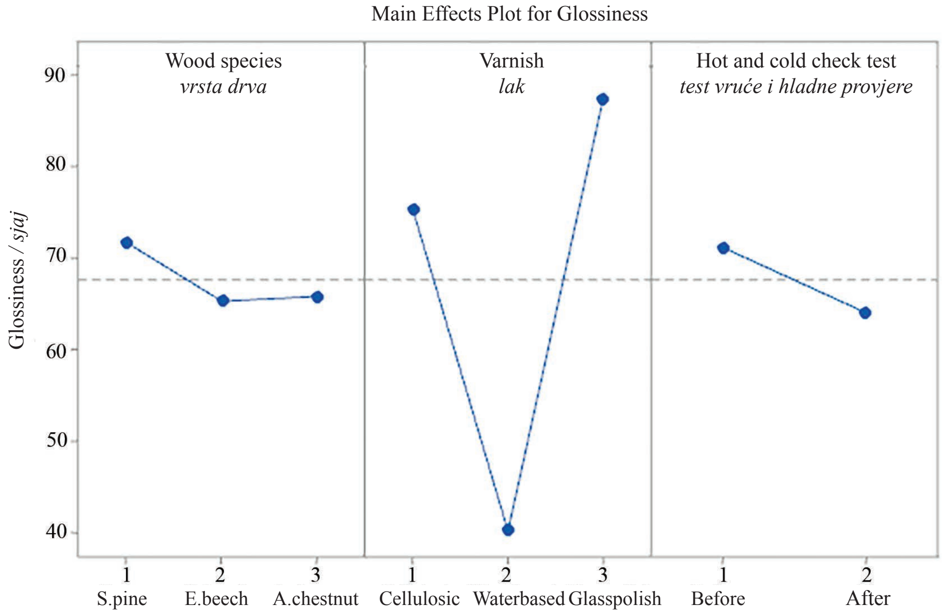

Figure 9 Main effects plot in terms of glossiness of wood species, varnish types, and hot-cold check test
Slika 9. Dijagram glavnih učinaka na sjaj s obzirom na vrstu drva, vrstu laka i test vruće i hladne provjere

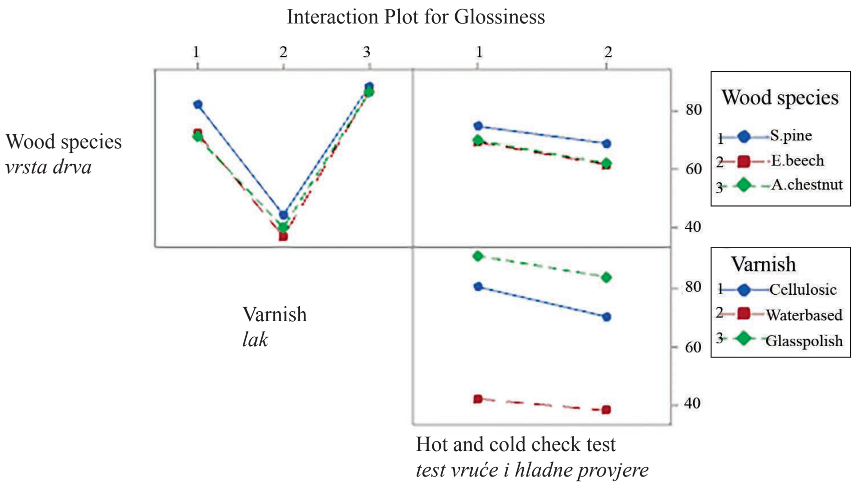

Figure 10 Interactions of wood species, varnish, and hot-cold check test of glossiness
Slika 10. Interakcije vrsta drva, laka i testa vruće i hladne provjere za sjaj

Kesik (1999) determined the effect of the hot-cold shock test on hardness and glossiness in Scots pine (*Pinus sylvestris* L.), Eastern beech (*Fagus orientalis* L.) and oak (*Quercus petreae* L.) tree species with cellulosic, polyurethane and acrylic varnish. According to the results of the experiment, it was determined that after the process, the hardness and glossiness values of all varnish types decreased. Ozalp *et al*. (2009), stated that hardness and glossiness values decreased in solid wood material kept at 100, 150 and 200 °C for 4 and 6 hours. The comparison, made in terms of the aging factor, showed that the highest total color change value was obtained in the samples without the hot-cold test, and the lowest in the samples with the hot-cold test. Similarly, the highest glossiness value was found in the samples without the hot-cold test and the lowest in the samples with the hot-cold test. It was reported in the literature that the varnish layers prepared for wooden surfaces lose their glossiness in outdoor weather conditions (Akpinar, 2012). Glossiness values also differ according to varnish types (Saygin, 2016). It was observed and found in the literature that a decrease in glossiness values occurs when wood is exposed to hot or cold external effects. As a result of sudden temperature changes, the glossiness decreased for each tree species and each varnish type. It was stated that the reasons for the decrease in the value of wood could lie in the formation of hemicelluloses and extractives, decomposition of products resulting from the effect of high heat or lignin polymerization reactions formed as a result of heat (Kamperidou *et al*., 2013; Barcík *et al*., 2015). In their study, they reported that the natural glossiness value of some trees was affected negatively after the increase in temperature and that the decrease in the glossiness value of the wood material depending on the temperature occurred as a result of chemical changes in glucose, hemicellulose and lignin

(Esteves *et al.,* 2008). The glossiness values differed according to the varnish type, and this result was also supported by the literature (Sonmez, 1989). The highest glossiness value was determined in the samples without the hot-cold test and the lowest in the samples with the hot-cold test (source). The literature states that the molecular sizes of water-based varnishes are smaller than those of solvent-based systems. Therefore, they penetrate more into the cavities of the wood material, thus creating thin layers (Sonmez *et al*., 2004). The low layer thickness reveals the effect of the texture of the wood material and the lowest glossiness value is obtained in water-based varnish. Water-based varnishes give the closest glossiness values to unvarnished samples (Sofuoglu and Aykac, 2020).

4 CONCLUSIONS
4. ZAKLJUČAK

Cellulosic varnish, water-based varnish and glass polish parquet varnish were applied to the surfaces of Scots pine, Eastern beech and Anatolian chestnut. Glossiness and roughness measurements were performed to detect the deformation on the surface during the hot-cold test process applied to the surfaces. The results show as follows:

The highest glossiness value was observed in Scots pine trees, while lower and close values were obtained in other tree species.

In terms of varnish type, the glossiness value was listed from the lowest as water-based (40.18 gloss), cellulosic (75.33 gloss) and glass polish (87.41 gloss).

Although the values were close to each other before and after the test in terms of hot and cold-check test, there was a decrease in the glossiness value after the test.

It was observed that the highest average roughness value (*Ra*) occurred in Anatolian chestnut trees, while lower and close values were obtained in other tree species.

In terms of varnish type, *Ra* roughness value was listed from the lowest as glass polish (0.1525 µm), cellulosic (0.5578 µm) and water-based (1.0933 µm).

Although the pre-test and post-test values were close to each other in terms of hot and cold-check test, there was a slight increase in the roughness value after the test.

It can be said that the changes in glossiness and roughness that occur on the surface of the varnished wood material after the hot-cold shock test do not affect the useful functions and surface properties of the products but are only affected in terms of aesthetics.

It can be predicted that such surface changes may occur in the three types of varnish applied to all three tree species, the furniture exported to countries with different climatic conditions, and the surface treatment material used on sea vehicles such as yachts/boats.

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Wood Species Image Classification Using Two-Dimensional Convolutional Neural Network

Klasifikacija vrsta drva prema slikama uz pomoć dvodimenzionalne konvolucijske neuronske mreže

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ABSTRACT • *The woodworking industry's recognition and classification of timber is essential for trade, production and timber science. Traditional methods of identifying wood types are complex, time-consuming, costly and require expertise in wood science. Traditional techniques have been replaced by convolutional neural networks (CNNs), a deep learning tool to better identify wood species. In contrast to earlier studies that used pre-trained models, a novel architecture designed explicitly for the WOOD-AUTH dataset was proposed in this study to develop a new 2D CNN model. The data collection encompasses high-level visual representations of 12 distinct types of timber. It is aimed to create a simpler and faster model as an alternative to time-consuming and heavy wood classification models. Compared to previous studies, this research worked with a newly structured 2D CNN network based on 12 wood species. High accuracy and fast computation time were achieved using fewer numbers (three layers) of the convolutional neural network. The proposed model achieved 94 % accuracy, 87 % precision, 81 % recall, 80 % F1 score and 112 minutes 27 seconds computation time. The 2D CNN model performed better than the transfer learning models regarding training epochs. The primary benefit of the model is its ability to achieve high accuracy with lower computation time, even at high epochs compared to other models. The introduced 2D CNN model produced satisfactory outcomes for wood species classification.*

KEYWORDS: *2D convolutional neural network; image classification; deep neural network; wood species*

SAŽETAK • *Identifikacija i klasifikacija drva u drvnoj industriji ključna je za trgovinu, proizvodnju i znanost o drvu. Tradicionalne metode identifikacije vrste drva složene su, dugotrajne i skupe te zahtijevaju stručnost s područja znanosti o drvu. Za bolju identifikaciju vrste drva tradicionalne su metode zamijenjene konvolucijskim neuronskim mrežama (CNN), odnosno alatom za duboko učenje. Za razliku od ranijih studija koje su se koristile unaprijed obučenim modelima, u ovoj je studiji predložena nova arhitektura dizajnirana upravo za skup podataka WOOD-AUTH kako bi se razvio novi 2D CNN model. Zbirka podataka obuhvaća vizualne prikaze visoke razluči-*

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vosti 12 različitih vrsta drva. Cilj je bio stvoriti jednostavniji i brži model kao alternativu dugotrajnim i složenim modelima klasifikacije drva. Za razliku od prethodnih istraživanja, u ovom je istraživanju primijenjena nova 2D CNN mreža koja se temelji na 12 vrsta drva. Visoka točnost i brzo vrijeme izračuna postignuti su korištenjem manjeg broja slojeva (tri sloja) konvolucijske neuronske mreže. Predloženim je modelom postignuta točnost od 94 %, preciznost od 87 %, opoziv od 81 %, F1 rezultat od 80 % i vrijeme izračuna od 112 minuta i 27 sekundi. Model 2D CNN pokazao se boljim od modela transfernog učenja u smislu epohe poduke. Primarna prednost modela jest njegova sposobnost postizanja visoke točnosti uz kraće vrijeme izračuna, čak i pri visokim epohama u usporedbi s drugim modelima. Prezentirani 2D CNN model dao je zadovoljavajuće rezultate za klasifikaciju vrste drva.

KLJUČNE RIJEČI: *2D konvolucijska neuronska mreža; klasifikacija slike; duboka neuronska mreža; vrste drva*

1 INTRODUCTION
1. UVOD

Wood has played an important role in human existence over time, fundamentally essential in many respects. With technological advancement, the industrial utilization and application of wood have been further enhanced. In its natural state, wood is an attractive traditional material with numerous advantages that have made it popular compared to other materials (Jones and Brischke, 2017; Nguyen *et al*., 2017; Popescu *et al*., 2011). It is important to possess a comprehensive understanding of its remarkable physical attributes, anatomical structure, mechanical properties, and chemical composition to ensure efficient use of wood. Furthermore, it is equally important to recognize its significant role as an essential prerequisite. (Brauns and Rocens, 2007; Kasal, 2004; Winandy and Rowell, 1984; Zabel and Morrell, 2020). Moreover, it should be noted that some wood species are protected nationally and globally. Therefore, identifying the wood species was important (Kırbaş and Çifci, 2022). The exact identification of wood species forms a pressing issue in different domains, including ecology, construction, furniture manufacturing and restoration, and determining the mechanical and economic properties of wood and wood-based materials. Wood species such as teak, ebony, and mahogany possess unique anatomical, physical, aesthetic, chemical, and mechanical properties (Tou *et al*., 2007; Vacha and Haindl, 2013). Differences in composition and structure vary between wood species and are used in their differentiation (Huang *et al.*, 2020).

Indeed, the traditional method of wood species identification based on macroscopic and microscopic characteristics has limitations, such as being time-consuming, impractical, and expensive. Moreover, the accuracy of this method may depend on the expertise of the professionals involved, which may vary. Hence, using machine learning and computer vision methodologies to automate processes holds immense potential for delivering expedited, efficient, and cost-effective solutions of wood species identification (Mohan *et al*., 2014; Rajagopal, 2019). The wood industry faces a significant hurdle in swiftly identifying a substantial timber volume. To address this issue, leveraging machine learning techniques and approaches can substantially enhance the speed and precision of wood species identification methods (Kırbaş and Çifci, 2022). Researchers have been striving to improve efficiency in recent years by shifting towards computer algorithms for wood image recognition methods instead of relying solely on human labour. One commonly utilized technique for recognizing wood species and their classification is to employ surface recognition models based on texture features. Most computer-based identification methods consist of two significant stages: feature extraction and classification. Boundary detection algorithms play an essential role in feature extraction methods. These methods include the Gray Level Co-occurrence Matrix (GLCM) (Manik *et al*., 2020; Santosa, 2019) for extracting grey colour matrices, the colour history statistical method (Zhao, 2013), and classification methods such as Support Vector Machine (SVM) (Sun, 2015; Souza *et al*., 2020), k-nearest neighbour algorithm (KNN) (Gani and Limam, 2013; Kobayashi *et al*., 2015; Fuentealba *et al*., 2014), and neural networks (Ravindran *et al*., 2018; Yinglai *et al*., 2020; Yang *et al*., 2019). Huayu *et al*. (2017) employed the structural covariance networks and mean squared error (SCN-MSE) technique to enhance the characteristics of digital images depicting Mongolian timber. Zhao (2013) devised a reliable approach for classifying wood species based on coloured wood surface images that effectively differentiate between intra-species and inter-species colour variations. Wang *et al*. (2013) successfully detected eight wood species in their experiments, achieving an accuracy rate of 86 %. Using the Fisher-Tree method, they extracted features from Mask Matching Images (MMI) and employed support vector machines (SVM) for species classification. Hafemann *et al.* (2014) conducted a comparative analysis, evaluating the performance of traditional classifiers against convolutional neural networks (CNNs). The first dataset comprised macroscopic images of 41 species, while the second dataset contained microscopic images of 112 species. The study found that the accuracy of CNN for the dataset of microscopic images was 97.32 %, while for the dataset of macroscopic images, it was

95.77 %. Kwon *et al*. (2017) introduced a CNN-based automatic identification system for wood species, while Huang *et al.* (2020) evaluated the performance of various CNN models for wood species recognition. The study reported that the LeNet3 model achieved the highest accuracy of 99.3 %. In addition, combining deep learning and machine learning techniques resulted in a recognition accuracy of 93.07 %. Tang *et al*. (2018) utilized the SqueezeNet architecture to develop a wood identification system for rapidly and reliably identifying wood species. Their research was focused on 100 frequently traded wood species in Malaysia, and their CNN-based model achieved top 1 and top 2 accuracies of 77.52 % and 87.29 %, respectively. Geus *et al*. (2020) conducted a study to evaluate the performance of four newly proposed CNN architectures for the classification of microscope images of wood. They compared the results with a pre-existing attribute method. Among the newly proposed architectures, the DenseNet model was found to achieve the highest accuracy of 98.75 %, indicating its effectiveness in wood species recognition. Lopes *et al*. (2020) assessed the performance of InceptionV4 and ResNetV2 architectures on North American hardwoods and obtained an accuracy of 92.60 %. Sun *et al*. (2021) used transfer learning with ResNet50 for wood species identification and achieved a high accuracy of 99.60 %. Fabijańska *et al*. (2021) employed a residual CNN architecture to recognize 14 European coniferous and flowering wood types in Europe. Their method achieved 93 % and 98.7 % accuracy rates for wood image patches and core images, respectively.

The literature review summarizes the drawbacks of previous research as listed below in bullet points:

- Conventional wood recognition is laborious, expensive, unfeasible, intricate, and arduous.
- The conventional techniques necessitate significant data processing, labour, and proficient expertise in selecting and extracting features.
- While deep learning methods have shown promise in many areas of image classification, the disadvantage is that the architectures employed are resource-intensive and time-consuming.
- It can be seen that the transfer learning models developed by (Haefmann *et al*., 2014), (Kwon *et al*., 2017), (Lopes *et al*., 2020) and (Su *et al*., 2021) provide accuracy higher than the proposed 2D CNN model. However, these applications require higher hardware systems for the model training, such as Tesla C2050 GPU with high numbers of epochs and XEON CPU with 64 GB RAM capacity computer specifications. In contrast to the high transfer learning models, the proposed 2D CNN model provided 94 % accuracy with NVIDIA GeForce RTX 2070 with 16 GB RAM capacity with shorter computation time. The previous transfer learning applications are not only expensive for the hardware system but also expensive for the computation time.

As an alternative version, this paper aims to address the limitations of existing studies and develop an efficient 2-dimensional convolutional neural network (2D CNN) model that can quickly and accurately identify wood species. The proposed model is designed specifically for the WOOD-AUTH (Barmpoutis, 2017; Barmpoutis *et al*., 2018) dataset and aims to improve classification accuracy compared to previous work that used popular pre-trained models. Therefore, it is desired to develop a more straightforward and faster model as an alternative to time-consuming and heavy wood classification models.

This paper is organized as follows: Section 2 details the dataset employed in the study. Section 3 outlines the methodology adopted in the research. Section 4 introduces the proposed 2D CNN model and discusses its implementation. Section 5 presents the findings and corresponding discussions. Lastly, Section 6 concludes the paper with a summary of the conclusions.

2 DATA SET
2. SKUP PODATAKA

The proposed model was developed using the WOOD-AUTH Laboratory of Wood Technology of Forestry and Natural Environment School of the Aristotle University of Thessaloniki, Greece (Barmpoutis, 2017; Barmpoutis *et al*., 2018) dataset. The dataset consists of 12 wood species; the total data contain 8160 images. Table 1 illustrates each wood species class with a number of image datasets. The different number of datasets for Walnut wood is less than for other wood types because of pre-defined data. The dataset can be called imbalanced due to the wood sample size.

The images were cropped to 200 × 200 pixels with 96 dots per inch (dpi). The 200 × 200 is low for some deep-learning applications but still efficient for wood classification. The WOOD-AUTH was prepared with 400 × 400 pixels (Barmpoutis, 2017; Barmpoutis *et al*., 2018) in the original data. However, the image resolution is decided based on the specific task, hardware, software and artificial intelligence model computation time. The 200 × 200 images were arranged for the proposed deep learning model to avoid the high computational time, memory requirement and processing power.

Figure 1 demonstrates the image samples from each class in the WOOD-AUTH. These sections indicate distinct features of the wood to provide detailed information for identifying the wood species. Considering various sections, the WOOD-AUTH dataset has different characteristics and variations.

Table 1 WOOD-AUTH dataset details the Latin name, type of species, category and number of images in each class
Tablica 1. Skup podataka WOOD-AUTH detaljno opisuje latinski naziv, vrstu drva, kategoriju i broj podataka u svakoj klasi

Class index *Indeks klase*	**Latin name** *Latinsko ime*	**Species** / *Vrste drva*	**Category** *Kategorija*	**Number of images** *Broj slika*
Wood-1.	*Fagus sylvatica*	European beech *drvo bukve*	Diffuse-porous hardwood *difuzno porozna listača*	1223
Wood-2.	*Juglans regia*	Walnut *drvo oraha*	Semi-diffuse-porous hardwood *semi-difuzno porozna listača*	289
Wood-3.	*Castanea sativa*	Sweet chestnut *drvo pitomog kestena*	Ring-porous hardwood *prstenasto porozna listača*	1532
Wood-4.	*Quercus cerris*	Turkey oak *drvo turskog oraha*	Ring-porous hardwood *prstenasto porozna listača*	600
Wood-5.	*Alnusglutinosa*	Alder *drvo johe*	Diffuse-porous hardwood *difuzno porozna listača*	696
Wood-6.	*Fraxinusornus*	Manna ash *drvo crnog jasena*	Ring-porous hardwood *prstenasto porozna listača*	648
Wood-7.	*Picea abies*	Norway spruce *drvo obične smreke*	Softwood *četinjača*	460
Wood-8.	*Pinus sylvestris*	Scots pine *drvo bijelog bora*	Softwood *četinjača*	332
Wood-9.	*Ailanthus altissima*	Tree-of-heaven *nebesko drvo*	Ring-porous hardwood *prstenasto porozna listača*	332
Wood-10.	*Robiniapseudoacacia*	Black locust *drvo bagrema*	Ring-porous hardwood *prstenasto porozna listača*	440
Wood-11.	*Cupressus sempervirens*	Mediterranean cypress *drvo sredozemnog čempresa*	Softwood *četinjača*	552
Wood-12.	*Platanus orientalis*	Old world sycamore *drvo azijske platane*	Diffuse-porous hardwood *difuzno porozna listača*	1056
	Total / *Ukupno*			8160

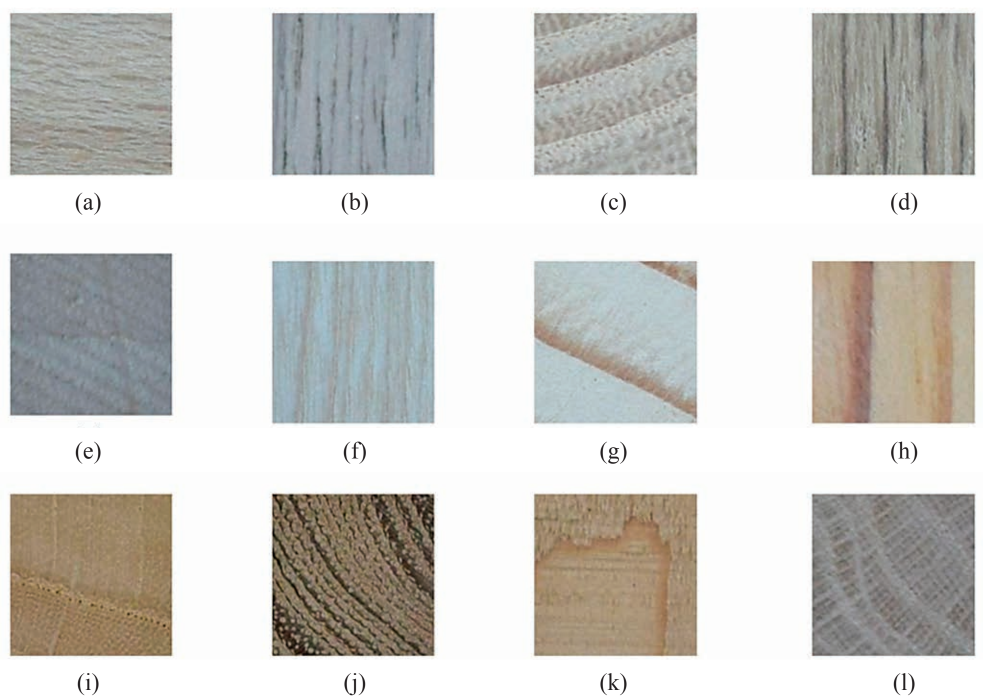

Figure 1 Images display samples of twelve wood species. (a) European Beech, (b) Common Walnut, (c) Sweet Chestnut, (d) Turkey Oak, (e) Common Alder, (f) Manna Ash, (g) Norway Spruce, (h) Scots Pine, (i) Tree of Heaven, (j) Black Locust, (k) Mediterranean Cypress, (l) Oriental Plane
Slika 1. Slike prikazuju uzorke dvanaest vrsta drva: (a) drvo bukve, (b) drvo običnog oraha, (c) drvo pitomog kestena, (d) drvo turskog hrasta, (e) drvo obične johe, (f) drvo crnog jasena, (g) drvo obične smreke, (h) drvo bijelog bora, (i) nebesko drvo, (j) drvo bagrema, (k) drvo sredozemnog čempresa, (l) drvo azijske platane

The 8160-image data was split into the train (80 %) and test (20 %). 6528 images were used for the model training, and 1632 were used for the model test evaluation. The two-dimensional (2D) convolution neural network (CNN), model evaluation metrics and the proposed model are explained in detail in Section 3 and Section 4, respectively.

3 METHODOLOGY
3. METODOLOGIJA

Employing the MATLAB software neural network toolbox, the proposed 2D CNN was configurated from scratch based on the different number of epochs. First, minor epochs were implemented to check model progress with low accuracy. The number of epochs has been increased to 30 and 50; it should be noted that those numbers were decided by trial and error. The best results were obtained with 50 epochs. The 2D CNN classifier consists of the ReLU activation and softmax output function with Adam optimizer and cross-entropy loss. The hyperparameters are elaborated on in Section 4. The model was interpreted by performing accuracy, precision, recall and F1 score indicators and compared to some transfer learning models in TensorFlow Hub.

3.1 Explanation of CNN
3.1. Objašnjenje CNN-a

O'Shea and R. Nash (2015) explained the introduction of the CNN algorithm. (CNNs) resemble traditional Artificial Neural Networks (ANNs) because they consist of neurons that undergo self-optimization through learning. Each neuron receives input and performs operations, such as a scalar product followed by a non-linear function, similar to many other ANNs. The entire network, from the input raw image vectors to the final output of the class score, can still be expressed as a single perceptive score function (the weight). The network last layer contains loss functions associated with the classes, and the usual tips and tricks developed for traditional ANNs are still applicable. CNNs differ from traditional ANNs in their primary use for image pattern recognition. This distinction allows the encoding of image-specific features into the architecture, which is essential when establishing CNN models. The convolutional layer is the first layer of CNN and works for feature extraction from the input image. The pooling layer reduces the number of parameters but does not have any function. The fully connected layer (last layer) is flattening the previous output layer in a vector. Kilic *et al.* (2022) explained the 1D CNN application for a regression analysis to predict machine-based datasets. Dhillon and Verma (2020) indicated that CNN consists of neurons with a learnable weight and bias. Sinaice *et al.* (2022) presented deep neural network image mapping and automatic recognition. Jannat *et al.* (2021) showed that CNN architecture has convolutional, pooling, and fully connected layers. In summary, CNN-based architectures are widely used in the literature for image classification; thus, 2D CNN was preferred to classify the WOOD-AUTH dataset. Section 3 demonstrates the model evaluation metrics, and Section 4 elaborates on the proposed model with hyperparameters.

3.2 Evaluation metrics
3.2. Mjerila evaluacije

3.2.1 Accuracy
3.2.1. Točnost

According to Powers (2020), the accuracy measure refers to the ratio of correctly predicted data points to all data points in the dataset. Eq. 1 expresses the formula of accuracy metrics.

$$accuracy = \frac{TP - TN}{n^{+} + n^{-}} = \frac{TP + TN}{TP + TN + FP + FN} \quad (1)$$

TP as a correctly identified positive instance, *TN* as a correctly identified negative instance, *FP* as a mistakenly identified positive instance, and *FN* as a mistakenly identified negative instance.

3.2.2 Precision
3.2.2. Preciznost

The precision score is a measure of the accuracy between the total number of true positive samples and the total number of predicted samples defined as positive (Power, 2020). Eq. 2 illustrates the calculation for the precision score.

$$precision = \frac{TP}{TP + FN} \quad (2)$$

3.2.3 Recall
3.2.3. Opoziv

The recall score was defined as a metric that compares true positive samples to the total number of true positive samples (Power, 2020).

$$precision = \frac{TP}{TP + FN} \quad (3)$$

3.2.4 F1 Score
3.2.4. F1 rezultat

The F1 score is a harmonic mean of the precision and recall shown in Eq. 4

$$F1score = \frac{2 \cdot TP}{2 \cdot TP + FP + FN} = 2 \cdot \frac{precision \cdot recall}{precision + recall} \quad (4)$$

The worst value is 0 for all evaluation metrics, whereas the best is 1.

4 PROPOSED 2D CNN CLASSIFICATION MODEL FOR WOOD SPECIES
4. PREDLOŽENI 2D CNN MODEL ZA KLASIFIKACIJU VRSTA DRVA

The 2D CNN models are preferred for image recognition and classification for different tasks, such as the wood industry, medical science, and interdisciplinary engineering fields. Therefore, 2D CNN was selected to classify the wood species based on their texture. The image dataset was normalized using zero-centre normalization. Afterwards, the proposed model was designed based on the explanation of (Simonyan and Zisserman, 2015) using 3 and 5 kernels with ReLU activation function and 400 fully connected layers to reduce training time. The filter size was selected. The filter size range between 32 and 1024 as the power of two, and a large filter size can provide a powerful model. However, the large filter size was not selected to create the proposed model in case of overfitting. Therefore, the proposed model worked with 20, 40 and 50 filter sizes. Due to the multiple classes, the output function was decided to work with the softmax function with cross-entropy (Zhang and Sabuncu, 2018). The Adam optimizer was implemented in the convolutional network for gradient descent optimization. Compared to the previous studies, the proposed model included three convolutional layers. Figure 2 illustrates the proposed model structure with the values of each parameter.

4.1 ReLU activation function
4.1. ReLU aktivacijska funkcija

According to Albawi *et al*. (2017) and Ajit *et al*. (2020), the ReLU function is implemented as an activation function. It converts negative values to zero and does not influence volume and hyperparameters. Compared to the sigmoid and tanh activation functions, the ReLU function is unaffected by vanishing gradient descent related to the deeper neural networks. Therefore, the ReLU has been preferred over the sigmoid and tanh activation functions. Eq. 5 shows the ReLU function equation.

$$ReLU(x) = \max(0, x)$$
$$\frac{d}{dx}(x) = \{1 if\ x > 0; 0\ otherwise\} \tag{5}$$

4.2 Softmax output layer
4.2. Softmax izlazni sloj

According to Maharjan *et al*. (2020) and Bridle, (1989), the softmax output activation function effectively classifies multiple classes apart from the binary classification. The WOOD-AUTH dataset has 12 classes and is called various classes. Therefore, the softmax activation function has been selected as the output function. Abd-Ellah *et al*. (2018) presented the softmax function formula in Eq. 6.

$$softmax = \frac{\exp(a_r)}{\sum_{j=1}^{k} \exp(a_j)} \tag{6}$$

Where a is the input vector with k dimensions, and y is the output vector with k dimensions.

4.3 Cross-entropy
4.3. Unakrsna entropija

The cross-entropy was used as a loss function of the model for the multi-classification. (Zhang and Sabuncu, 2018) The cross-entropy loss function was defined as a loss training process in the final layer. The multi-classification of the loss (error) function is presented in Eq. 7.

$$cross - entropy = -\sum_{i=1}^{n}\sum_{j=1}^{k} t_{ij} ln y_j (x_i, \theta) \tag{7}$$

Where θ is the parameter vector, t_{ij} is the index that *i* sample related to *j* class, and $y_j(x_i, \theta)$ is the output.

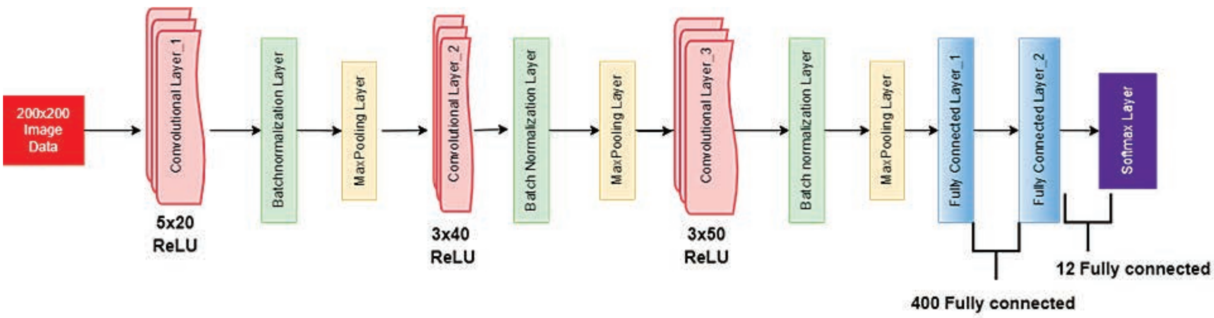

Figure 2 Proposed 2D CNN model structure. Red colour rectangular shape refers to input dataset, pink layered rectangular shape corresponds to convolutional layers, green rectangular shape refers to batch normalization layers, yellow rectangular shape presents max-pooling layers, and the last blue rectangular shape is fully connected layer. The last layer is the output softmax layer. 2D CNN model hyperparameters are detailed in Sections 4.1 – 4.4.
Slika 2. Predložena struktura 2D CNN modela. Crveni pravokutni oblik odnosi se na ulazni skup podataka, ružičasti slojeviti pravokutni oblik odgovara konvolucijskim slojevima, zeleni pravokutnik odnosi se na seriju normalizacijskih slojeva, žuti pravokutni oblik predočuje slojeve maksimalnog udruživanja, a posljednji plavi pravokutni oblik odnosi se na potpuno povezani sloj. Zadnji sloj je izlazni softmax sloj. Hiperparametri 2D CNN modela detaljno su opisani u poglavljima 4.1. – 4.4.

4.4 Adam optimizer
4.4. Adamov optimizator

According to Bock *et al*. (2018), the Adam (adaptive moment estimation) optimizer is a well-known algorithm in gradient descent optimization. The Adam optimizer is distinguished in terms of faster optimization in the deep neural network. The optimizer can converge much faster for deeper networks and convolutional neural networks.

5 RESULTS AND DISCUSSION
5. REZULTATI I RASPRAVA

The model was created based on the WOOD-AUTH to classify the 12 wood species. This model accomplished the task successfully. Apart from the proposed model, some popular transfer learning models have been applied to the dataset to show the strength of the proposed model. The 2D CNN model was outperformed in terms of accuracy and computation time compared to the Efficient B3 and Mobile Net transfer learning models. On the other hand, the outcomes of the proposed model can be compared to the previous applications in the literature. The proposed model performance was evaluated using accuracy metric, precision, recall, F1 score and computation time. Figure 3 demonstrates the confusion matrix of the 12 wood species distribution regarding the predicted and real classes. The 2D CNN model had an accuracy of 94 %, precision of 87 %, recall of 81 % and F1 score of 80 %. The metrics have been explained in Section 3.2. Table 2 demonstrates that the model outcomes are based on the metrics. Figure 3 illustrates the model training and loss relationship. It can be seen that the model worked properly with 50 epochs.

The proposed model training and cross-entropy loss function can be seen in Figure 4. Figure 4 illustrates the model behaviour with the loss function.

It can be noticed that the model was not exposed to overfitting, and the number of datasets was enough to train the model. In addition to Figure 4, Table 2 presents and compares the proposed model outcomes to other popular transfer learning models.

5.1 Model comparison
5.1. Usporedba modela

The comparison indicates that the proposed model provided fast computation (112 min 27 sec) while obtaining high accuracy (94 %). The transfer learning model Xception accuracy is slightly higher than that of the proposed model; however, its computation time is higher than that of the offered model (the proposed model is 41.07 % faster than the Xception). The 2D CNN model is stronger than the VGG19 transfer learning in evaluation metrics and computation. The proposed model provided a reasonable result for the classification of wood species. This research created a new 2D CNN to classify the WOOD-AUTH dataset. In addition to the proposed model, some popular transfer

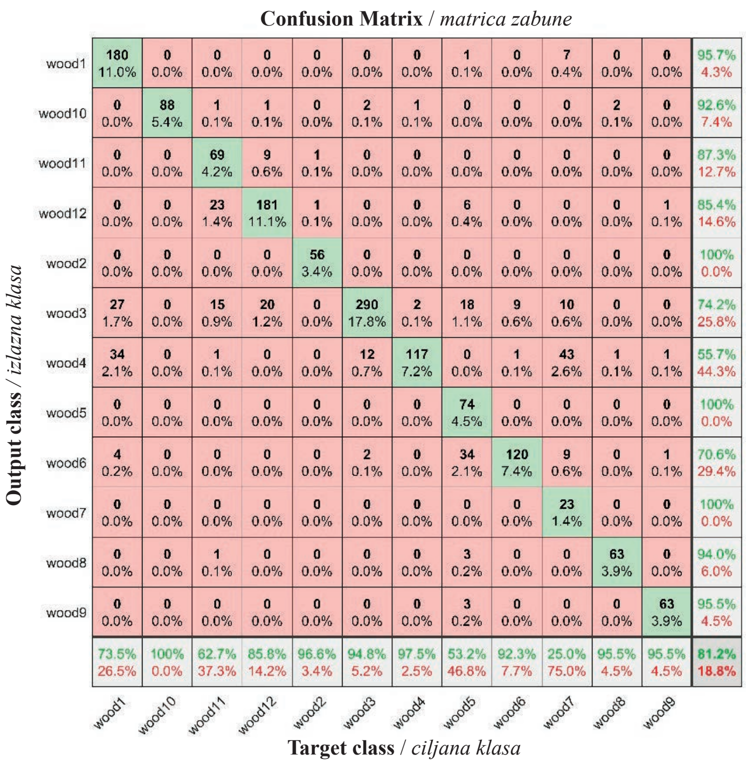

Figure 3 Confusion matrix of 12 wood species with accuracy metrics
Slika 3. Matrica zabune za 12 vrsta drva s metrikom točnosti

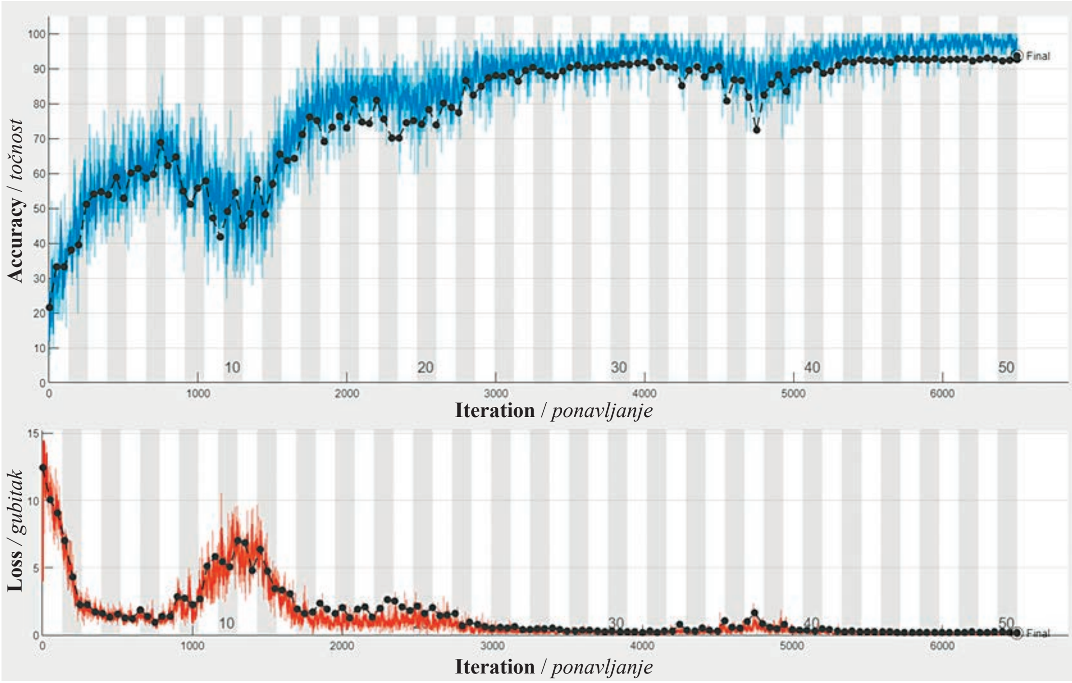

Figure 4 Model training and loss relationship in 50 epochs. Blue line refers to training accuracy, and black line corresponds to test accuracy. Orange bottom line refers to training loss, while black line refers to test loss
Slika 4. Model treninga i odnosa gubitka u 50 epoha. Plava se linija odnosi na točnost treninga, a crna odgovara točnosti testa. Narančasta donja crta odnosi se na gubitak treninga, a donja crna crta predočuje gubitak testa

learning models (Kumar, 2023) have been implemented to show the performance of the newly configurated 2D CNN model. In that case, Efficient B3 and Mobile Net transfer learning models were applied using Tensorflow Hub information.

On the one hand, Xception, Inception V3, ResNet50 and VGG19 (Kırbaş and Çiftçi, 2022) published in the literature were compared to the proposed model. The model's strength is its faster computation time, higher evaluation metrics and fewer convolutional layers. In contrast to the proposed model, the major shortcomings of the transfer learning models is the need for high memory size and slow training; their network architecture weights are exceptionally high.

6 LIMITATIONS
6. OGRANIČENJA

Even though the proposed model is superior to the previous studies, it has some limitations; trial and error hyperparameter tuning and demanding training required to obtain the proper results, compared to the pre-trained models. The data augmentation can be applied to see the model performance on the balanced dataset. In the future, the model will be saved and applied to different wood species datasets. The model can be converted to an application using deep learning deployment techniques used by non-technical, model accomplished, users.

Table 2 Model comparison
Tablica 2. Usporedba modela

Model name *Naziv modela*	**Accuracy, %** *Točnost, %*	**Precision, %** *Preciznost, %*	**Recall, %** *Opoziv, %*	**F1 Score, %** *F1 rezultat, %*	**Computation time** *Vrijeme izračuna*
The proposed 2D CNN model *predloženi 2D CNN model*	94	87	81	80	112 min 27 sec
Efficient B3	82	87	77	81	3 h 24 min
Mobile Net	72	79	66	72	5 h 35 min
Transfer learning models in literature (Kırbaş and Çiftçi, 2022) *Transferni modeli učenja u literaturi* (Kırbaş i Çiftçi, 2022.)					
Xception	95	95	95	95	2 h 38 min 6 s
Inception V3	88	87	87	86	2 h 23 min 34s
ResNet50	37	58	56	57	2 h 6 m 13s
VGG19	61	20	31	23	7 h 30 m 45s

7 CONCLUSIONS
7. ZAKLJUČAK

This research used the new 2D CNN model to employ the WOOD-AUTH dataset with multiple classes (12 wood species classes). The new 2D CNN model required less computation time with 8160 image datasets than the heavy pre-trained transfer learning models. Therefore, the study aims to release a new alternative to deep learning for future classification tasks.

The main conclusions of this research are:

Compared to previous studies, this research worked with the newly configurated 2D CNN network based on 12 wood species. High accuracy and fast computation time were acquired using fewer numbers (three layers) of the convolutional neural network.

The proposed model achieved 94 % accuracy, 87 % precision, 81 % recall, 80 % F1 score and 112 min 27 sec computation time.

Compared to the transfer learning models, the 2D CNN model worked with fewer epochs in training.

The model does not require a high memory size due to the lightweight network structure. Otherwise, transfer learning models need a high memory size, e.g. VGG19 requires 549 megabytes (M.B.) (Keras, 2022).

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Cebrail Açık[1]

Modeling of Color Design on Furniture Surfaces with CNC Laser Modification

Modeliranje dizajna boje površine namještaja CNC laserskom modifikacijom

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ABSTRACT • *Today, handcrafted furniture surface treatment techniques are less used due to application difficulties and high costs. Recently, the use of CNC router processing machines has resulted in a revival of these techniques. However, since this method is insufficient in micro-processes and color modifications that require precision, these processes have started to be performed with lasers. In this research, the beech wood surface was processed using a CNC laser processing machine with a carbon dioxide gas tube by applying different engraving power and engraving speeds. Product design and manufacturing parameters were determined in the CIE L*a*b* color system using the regression modeling method. A sample application was made by applying laser engraving to a furniture surface designed according to the obtained parameters. As a result of the study, it has been explained that using L* color group regression modeling method and CAD/CAM supported laser technology in furniture top surface color design processes is suitable for industrial engineering approach. It has been determined that many surface color design techniques can be applied with laser to furniture designed for CNC laser production.*

KEYWORDS: *laser pyrography; furniture surfaces; color design*

SAŽETAK • *Primjena ručnih tehnika površinske obrade namještaja smanjila se zbog otežane izvedbe i visokih troškova. Ručne su se tehnike nedavno pokušale oživjeti obradom CNC glodalima. Međutim, CNC glodanje nije odgovarajuća tehnika za mikroprocese i modifikacije boja koje zahtijevaju preciznost, pa su se ručne tehnike počele provoditi laserima. U ovom je istraživanju površina bukovine obrađena CNC laserom s plinskom cijevi od ugljikova dioksida, uz primjenu različite snage i brzine graviranja. Dizajn proizvoda i proizvodni parametri određeni su u sustavu boja CIE L*a*b* primjenom metode regresijskog modeliranja. Na površini namještaja laserskim je graviranjem izrađen uzorak dizajniran prema dobivenim parametrima. Rezultatima istraživanja utvrđeno je da je primjena L* komponente boje u regresijskom modeliranju, potpomognuta CAD/CAM laserskom tehnologijom, prikladna u procesima dizajniranja boje površine namještaja unutar inženjerskog pristupa u industriji. Utvrđeno je da se na namještaju dizajniranom za CNC lasersku proizvodnju laserom mogu provoditi mnoge tehnike dizajniranja boje površine.*

KLJUČNE RIJEČI: *laserska pirografija; površine namještaja; dizajn boje*

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1 INTRODUCTION
1. UVOD

One of the most important issues in laser wood surface treatment is to be able to explain the reaction methodology of wood surfaces against laser beams. Laser engraving on wooden surfaces is generally done to increase the commercial and aesthetic value of furniture or wooden products. Furniture surface decoration is the evaluation of furniture with color, shape, painting and motifs by using various techniques in order to add beauty without disturbing its function. Furniture, which has been in continuous development since ancient times, continued to change in terms of technology, concept and function at the beginning of the 20th century. With the 20th century, industrial society was formed and manual labor lost its previous validity. In the 20th century, it is seen that furniture design movements and architectural movements overlap and affect each other (Çifçi and Demirarslan, 2021). Technology offers the opportunity to reach the development of art techniques, which have become a part of culture and innovations, in a practical, fast and easy way. While art is affected by the development of technology, industry and industrial products also benefit from art and design (Aytepe, 2013). In order to produce the parts of the furniture designed with CAD systems in the computer environment, the designs of these parts can be directed to the CAM systems. In the laser cutting and engraving processes of wood and wood-based materials, the probability of faulty production is less than in the machining made with traditional techniques and using cutters in CNC (router) machines. In addition, in laser processing of wood materials, very precise and micro-processes that cannot be performed on CNC machines with cutters can be easily performed (Karabıyık, 2016).

Studies on color design by laser modification of wood surfaces are not new. Jurek and Wagnerova (2021) investigated the transfer of non-vector photographic images in different color tones to the wooden surface by laser engraving. Kubovsky and Babiak (2009) investigated the changes in the color system of maple (*Acer pseudoplatanus, L.*) and beech (*Fagus sylvatica, L.*) wood surface according to different irradiation doses in the CIE *L*a*b** color system. The changes of scanning speed, scanning power and nozzle height parameters in the CIE *L*a*b** color system in laser engraving of poplar wood were investigated using the mathematical modeling method according to the response surface method (RSM) (Li *et al*., 2018). Reinprecht and Vidholdov (2021) analyzed the effects of CO_2 laser etched surfaces on adhesion resistance after coating with a polymer layer of polyvinyl acetate (PVAc) and polyurethane (PUR) at different irradiation doses for the modification of European beech (*Fagus sylvatica L.)* and Norway spruce (*Picea abies L.)* wood surfaces. CIE *L*a*b** color measurements were performed on samples obtained from beech, ash, linden and spruce tree species to evaluate aesthetic changes due to different laser beam intensity and the number of laser points (points/inch) on the surface (Petutschnigg *et al*., 2013). In order to examine the photodegradation of wood, two different lasers (KrF- and XeCl-laser) were etched and the changes in wood color resulting from heat treatment after laser irradiation were investigated by DRIFT (Diffuse Reflectance Infrared Fourier Transform) spectroscopy and color measurement of the changes on the wood surface (Mitsui *et al*., 2005). Color changes on aspen, birch, spruce, pine, red beech, oak, alder, birch, plywood and MDF surfaces using different power and speed laser combinations were visually examined (Teivonen, 2016). The color changes of the effect of CO_2 laser engraving speed at different rates on sycamore maple (*Acer pseudoplatanus L.*) were investigated by making CIE *L*a*b** color measurements (Petru and Lunguleasa, 2017). Gurau *et al*. (2017) investigated the effects of CO_2 laser beam power output and scanning speed on the surface roughness and color changes in samples obtained from beech (*Fagus sylvatica*) wood. The effects of CO_2 laser modification of beech wood surfaces modified with Aspergillus niger and Penicillium brevicompactum molds using different (J/8) radiation dose and power were investigated in the CIE *L*a*b** system (Vidholdova *et al*., 2017). The color changes of the effect of CO_2 laser engraving speed and irradiation dose (Joule) at different rates on beech wood were investigated by making CIE *L*a*b** color measurements (Kacik and Kubovsky, 2011). Kudela *et al*. (2020) investigated the variation of color, roughness and waviness parameters of the beech wood surface depending on the CO_2 laser engraving power and raster density. Kubovsky and Kacik (2014) investigated the changes in color and wood chemical components caused by irradiation dose of lime (*Tilia vulgaris L.*) tree with CO_2 laser beam at different rates.

Until today, many studies on color design on wood surface treatment with laser have been made. Studies on the effect of laser surface modification of wood on color changes have generally focused on color change rate parameters. These studies have generally remained at the experimental level and their application in furniture design and production process has not been explained. In this study, experimental studies were carried out on a medium-sized CNC laser machine, which is widely used, by using beech wood, which is frequently used in industrial production. In line with the findings, furniture surface decoration color design and application were made with the regression modeling method, which is important in industrial production. An original study was carried out by presenting the material, machine, production method, design and manufacturing processes required by the industrial production sector as a whole.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

In the study, beech wood with a density of 721 kg/m³ in air-dried condition at 12 % humidity was used. The samples were prepared from randomly selected 1st class wood material, with smooth fibers, no knots, no cracks, no difference in color and density, and sapwood parts. Experimental specimens measuring 10 mm × 100 mm × 100 mm were cut and sanded with 120 grit sandpaper. In material processing, engraving processes were carried out on the experimental samples in a CNC laser machine with a maximum power output of 130 watts, a carbon dioxide gas tube, 2 inch (50.8 mm) focal length lens, water-cooled, 1.5 mm nozzle diameter and 10.6 µm wavelength.

2.2 Methods
2.2. Metode

The factorial experimental design method was preferred in the study. With this method, it was possible to evaluate each of the variable factors together with each other and to determine the extent of the effect of each variable on the event. In addition, as a result of the interaction of the variables with each other, different behaviors that the factors may show can be determined. There are many factors that affect laser processing performance. According to the information obtained from the experimental studies carried out so far, it is aimed to determine the effect of the factors that the user is allowed to access, by keeping the factors belonging to the system constant. In this study, 10 %, 40 % and 70 % laser power and engraving speeds of 200, 350 and 500 mm/s were applied to wood samples in a CNC laser machine, taking into account the limitations of machine processing functions. There are 2 variable parameters in the experimental design and each of them takes 3 different values. According to the specified levels, Taguchi orthogonal design is the full factorial design, with 27 data for each type of laser engraving and can be called L27(23). This notation indicates that the number of experiments is 27, with 2 factors having 3 different levels.

In the tests performed, the experimental data were evaluated using SPSS statistics version 22. The interaction of the laser engraving speed and laser engraving power dependent variables and the color independent variable were analyzed. For each analysis performed, multiple linear regression analysis was preferred to determine whether there was a significant difference. Preliminary estimates made to achieve optimum yield quality in production are calculated according to the regression Eq. 1.

$$\hat{Y} = \beta_0 + \beta_1 \cdot X_1 + \beta_2 \cdot X_2 \quad (1)$$

Where;

$\hat{Y}$ – dependent variable (color value),

β_0 – donstant beta value,

β_1 – 1st independent variable (engraving power) beta value,

X_1 – value of 1st independent variable (Engraving Power Watt%),

β_2 – 2nd independent variable (engraving speed) beta value,

X_2 – value of 2nd independent variable (engraving speed mm/s) defined.

2.3 Color measurement
2.3. Mjerenje boje

For color measurements, CIELAB color range was defined by the International Commission on Illumination (CIE) in 1976 according to the principles specified in ASTM D 2244. In this system, color is represented as a point in 3 dimensions on the *x*, *y* and *z*

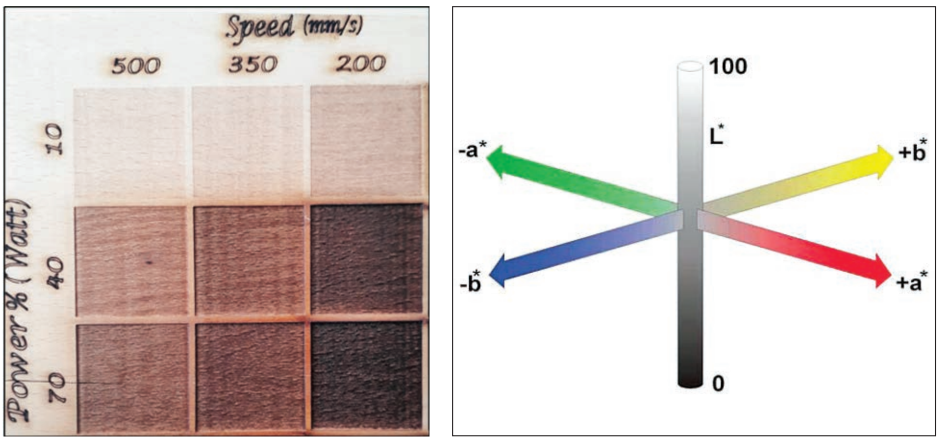

Figure 1 (a) Example of laser engraving experiment, (b) structure of axes *L**, *a**, *b** in the coordinate system CIELAB (Kubovsky and Babiak, 2009)
Slika 1. (a) Primjer eksperimenta laserskoga graviranja, (b) struktura osi *L**, *a**, *b** u koordinatnom sustavu CIELAB (Kubovsky i Babiak, 2009.)

axis. In the CIE *L*a*b** color system, the differences in colors and their locations are determined according to the *L**, *a**, *b** color coordinates. Here, *L** is on the black-white (*L**=0 for black, *L**=100 for white) axis, *a** is on the red-green (red is positive, green is negative) axis, *b** is on yellow-blue (yellow is positive, blue is negative) axis. In addition, in the *bi**, *br**, *ai**, *ar*, *Li**, *Lr** notations, *i* shows the treated experimental measurement value and *r* shows the untreated reference value. Figure 1.a shows the sample of the experiment which was measured. In the color area shown in Figure 1.b, the *L** coordinate constitutes the vertical axis (*y*), the *a** coordinate constitutes the horizontal (*x*) axis, and the *b** coordinate constitutes the (*z*) axis.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

The laser engraving colors obtained according to the speed (*S*) and laser processing power (*P*) test factors in laser engraving applied to the parallel directions of the wood laser engraving samples are shown in Table 1.

In the untreated natural state of the beech massif, the white value (*L**) on the black and white axis was measured as 76.21, the yellow value (*a**) on the blue-yellow axis was 7.27 and the red value (*b**) on the green-red axis was measured as 19.94. In laser engraving of wood surface, power increments in all parameters decreased the white value and increased the black value. This situation changed inversely with the engraving speed increments. In other words, the engraving speed increments increased the whiteness value in all parameters. These results have been confirmed in many studies. For example, it has been stated that the scanning speed negatively affects the *L** change in laser engraving of poplar wood (Li *et al*., 2018). In the laser engraving of wooden surfaces, the engraving speed increments did not have any effect on the *a** and *b** color values in engravings with a power of 10 %. This situation has also been explained by the changes in the wood surface in engraving with XeCl-lacelet to examine the photodegradation of wood (Mitsui *et al.*, 2005). On the other hand, engraving speed increments increased both the *a** value and the *b** value in engravings with 40 % and 70 % power. In other words, while the engraving speed at high powers increased the yellow and red color values, it decreased the green and blue color values. This was confirmed in a study made to evaluate the aesthetic changes due to different laser beam intensity and the number of laser points (points/inch) on the surface of samples obtained from beech trees (Petutschnigg *et al*., 2013). In wood surface engraving, the color changes in the green-red axis showed the same behavior as the changes in the blue-yellow axis in all parameters. In another study, it was stated that the trends in the blue-yellow and green-red axis of beech wood surfaces treated with laser engraving were the same (Jurek and Wagnerova, 2021). However, in another study, it was stated that the yellow and red tendency of the beech wood surface increased with laser engraving power increments (Kudela *et al*., 2020). This may be due to operating under 10 % power. As the wood cell structure has not yet been degraded by full laser irradiation, it has been explained that complete carbonization cannot occur (Kubovsky and Kacik, 2014). In laser engraving of the wood surface, *b** yellow color values were lower than an untreated reference value, below the engraving speed of about 300 mm/s when processing at 40 % engraving power. The same situation occurred in the color changes under the effect of laser engraving speed at different rates on Sycamore maple (*Acer pseudoplatanus L.*) (Petru and Lunguleasa, 2017). In this study, the engraving power increased both the blue and green color ratio of the

Table 1 Wood surface laser engraving colors
Tablica 1. Boje za lasersko graviranje površine drva

N	*P*, W	*S*, mm/s	*L**	*a**	*b**	*N*	*P*, W	*S*, mm/s	*L**	*a**	*b**
1	Control		76.67	7.31	19.72	16	40	350	33.79	13.71	24.71
2	10	500	68.54	9.92	29.42	17	40	200	22.66	9.16	14.07
3	10	350	65.87	10.65	30.42	18	70	500	34.97	14.75	25.61
4	10	200	59.36	11.66	30.09	19	70	350	29.02	12.44	20.4
5	40	500	43.77	15.38	31.71	20	70	200	20.91	7.87	11.79
6	40	350	34	13.85	25.14	21	Control		75.72	7.32	19.41
7	40	200	24.64	9.29	14.11	22	10	500	66.7	10.38	26.82
8	70	500	35.38	14.43	26.07	23	10	350	62.18	11.00	26.26
9	70	350	28.63	12.41	19.94	24	10	200	52.98	11.97	26.18
10	70	200	21.36	7.61	11.35	25	40	500	36.08	14.53	26.06
11	Control		76.77	7.18	20.71	26	40	350	25.85	10.94	17.31
12	10	500	68.97	9.81	27.93	27	40	200	15.38	5.36	7.71
13	10	350	66.14	10.38	28.54	28	70	500	30.65	12.63	20.55
14	10	200	58.2	12.13	29.95	29	70	350	24.26	9.71	14.79
15	40	500	41.99	15.47	30.99	30	70	200	15.79	4.15	6.34

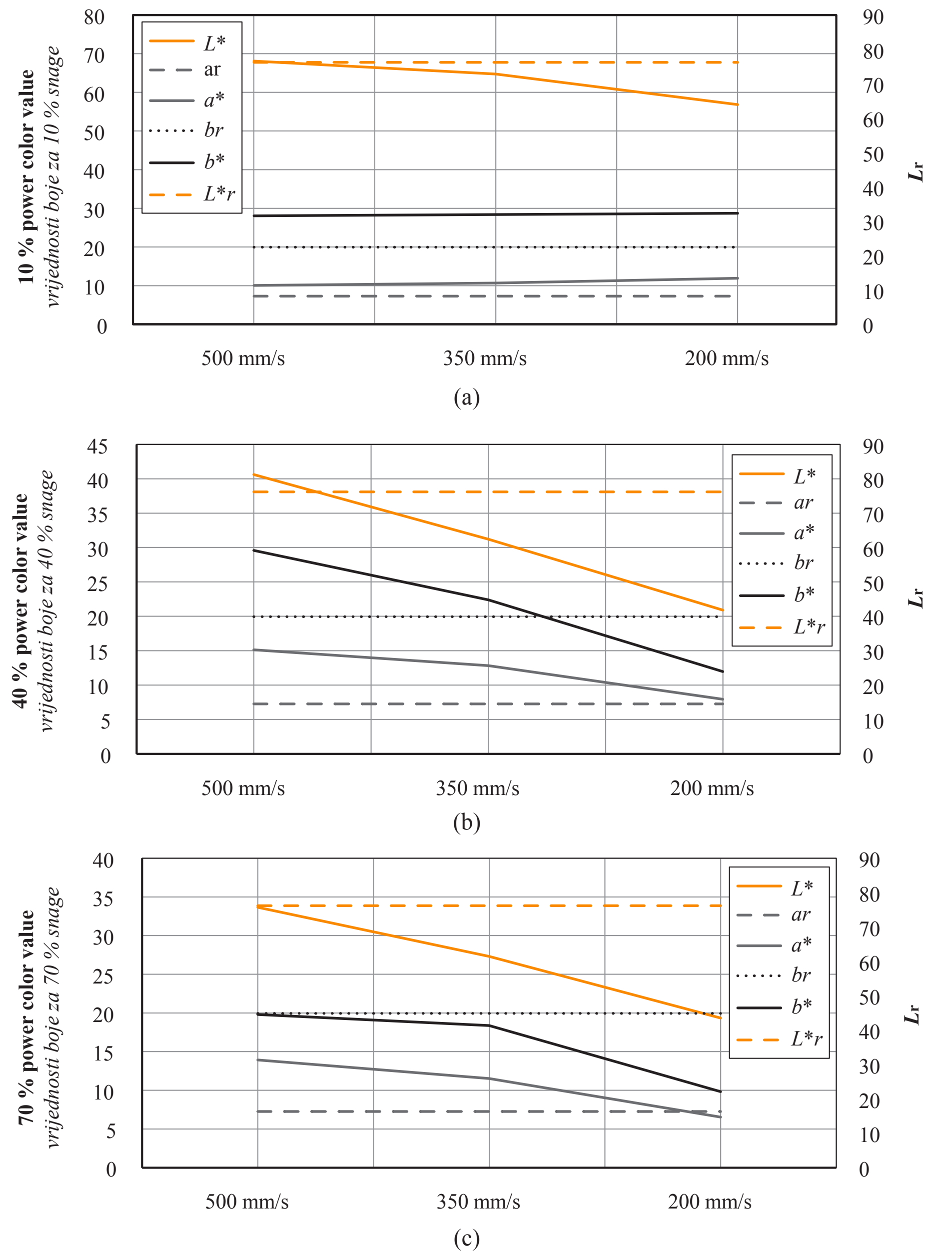

Figure 2 Surface engraving color changes at (a) 10 % power, (b) 40 % power, (c) 70 % power
Slika 2. Promjene boje gravirane površine pri (a) 10 % snage, (b) 40 % snage, (c) 70 % snage

wooden surface. The same results were obtained in the modification of beech wood surfaces with laser using different (J/8) radiation doses and power (Vidholdova *et al*., 2017). In this study, the color changes of the laser-treated surfaces of the beech massif at different engraving speed parameters at 10 % engraving power are shown in Figure 2a, color changes at different engraving speed parameters at 40 % engraving power are shown in Figure 2b, and at 70 % in Figure 2c. The color changes are shown at different engraving speed parameters and at a specific engraving power.

Multivariate linear regression analysis was performed to determine the effect of engraving power and engraving speed variables on color changes in wood surface laser engraving. The analysis results of the laser engraving process are shown in Table 2 below.

According to the analysis results in Table 2 above, laser engraving power negatively and significantly affected the color of whiteness (L^*) in the black-white axis on the laser engraving surface with an effect size of 80 % (pr^2 = 0.802). In other studies, it has been reported that the color changes on the wood surface of beech (*Fagus sylvatica, L.*) are the mostly L^* color changes according to different ($J \cdot cm^{-2}$) irradiation doses (Kubovsky and Babiak, 2009). The laser engraving speed affected the white color on the laser engraving surface positively and significantly with an effect size of 41 % (pr^2 = 0.412). The regression analysis model was 95 % reliable (p<0.05), and 68 % of the whiteness color change (R^2=0.723) was performed by the scratch power and engraving speed variables. Based on the data in Table 2 above, the mathematical modeling re-

Table 2 Regression analysis results
Tablica 2. Rezultati regresijske analize

Dependent *Zavisne varijable*	*L**			*a**			*b**		
	β	*pr*	*p*	*β*	*pr*	*p*	*β*	*pr*	*p*
Constant / *konstanta*	46.993	-	0.000	6.372	-	0.000	17.567	-	0.000
Engraving power, W % *snaga graviranja,* W %	-0.607	-0.896	0.000	-0.004	-0.039	0.850	-0.183	-0.731	0.000
Engraving speed, mm/s *brzina graviranja,* mm/s	0.050	0.642	0.000	0.014	0.616	0.001	0.035	0.712	0.000

Β – Beta, *pr* – Partial correlations, $p>0.005$ / *β – beta, pr – djelomične korelacije, p>0,005.*

sult of the beech massif was used to theoretically provide optimum laser engraving quality and predict the whiteness color, generating the following regression equation:

$L^{*}=46{,}993+(\textit{Engraving power}^{*}{-0.607})+$
$+(\textit{Engraving speed}^{*}0.050)$

In wood surface engraving, laser engraving power did not affect the redness (a^*) color on the green-red axis on the laser engraving surface at the 95 % confidence level. Laser engraving speed affected the red color on the laser engraving surface positively and significantly with an effect size of 38 % ($pr^2 = 0.379$). The regression analysis model was 95 % reliable ($p<0.05$), and 11 % of the whiteness color change ($R^2=0.107$) was achieved by the engraving power and engraving speed variables. Since the effect of engraving power is meaningless and the impact value of engraving speed is low, the regression equation was not produced to predict the red color of the beech massif as a result of mathematical modeling.

In wood surface engraving, laser engraving power affected the yellowness (b^*) color of the blue-yellow axis on the laser engraving surface negatively and significantly with an effect size of 53 % ($pr^2 = 0.534$). The laser engraving speed affected the yellowing color on the laser engraving surface positively and significantly with an effect size of 51 % ($pr^2 = 0.506$). The regression analysis model was 95 % reliable ($p<0.05$), and 47 % of the yellowness color change ($R^2=0.469$) was performed by the engraving power and engraving speed variables. Based on the data in Table 2 above, the mathematical modeling result of the beech massif was used to theoretically ensure optimum laser engraving quality and predict the turban color, generating the following regression equation:

$b^{*}=17.657+(\textit{Engraving power}^{*}{-0.183})+$
$+(\textit{Engraving speed}^{*}0.035)$

3.1. Design and implementation
3.1. Dizajn i implementacija

CNC laser machine works with a computer hardware loaded with CAD (Computer Aided Design) program and CAM (Computer Aided Manufacturing) programs that convert CAD drawings into machine codes. In this study, a case study was conducted to determine the applicability of CNC laser wood surface decoration with regression modeling method. The steps of design and production process of CNC laser operations on the wooden box made of beech massif used in the experiment are explained below.

The engraving paths to be made with the ornament motif forming the wooden box surfaces were drawn in a CAD program. The motif has been transferred to the CAM program in the software of the CNC laser machine as vector. The production design was completed by determining the motif design, and production parameter values were measured according to the box. Since the effect level of the white color (L^*) value in the above regression analysis in the motif design is much higher than the yellow (a^*) and red (b^*) color values, calculations were made over the white color analysis values in the power parameter calculations. As the L^* color is more effective in color designs in other studies, a color design study was carried out based on the L^* value (Jurek and Wagnerova, 2021). For the motif on the box surface, it is aimed to obtain three-stage colors that will create contrast to each other. The white value (L^*) on the black and white axis of the untreated natural state of the beech massif was measured as 76.21. The change of this value in the black direction was obtained as 19.35 at the maximum 200 mm/s engraving speed and 70 % engraving power parameter, as seen in Figure 2.a. It was determined between the maximum (76.21) and minimum (19.35) values according to the mean values in the three targeted white color test findings. These values were determined as 20, 40 and 60 according to the homogeneous distribution. In order to complete the engraving power process with maximum efficiency and in the shortest time, the engraving speed was kept constant at the highest value of 500 mm/s. The speed-power parameter to be applied to the box surface was calculated from the Scratch color regression formula obtained from the above findings.

$L^{*}_{1}=46.993+(\textit{Engraving power}^{*}{-0.607})+(\textit{Engraving speed}^{*}0.050)$

$20=46.993+(\textit{Engraving power}^{*}{-0.607})+(\textit{Max speed}^{*}0.050)$

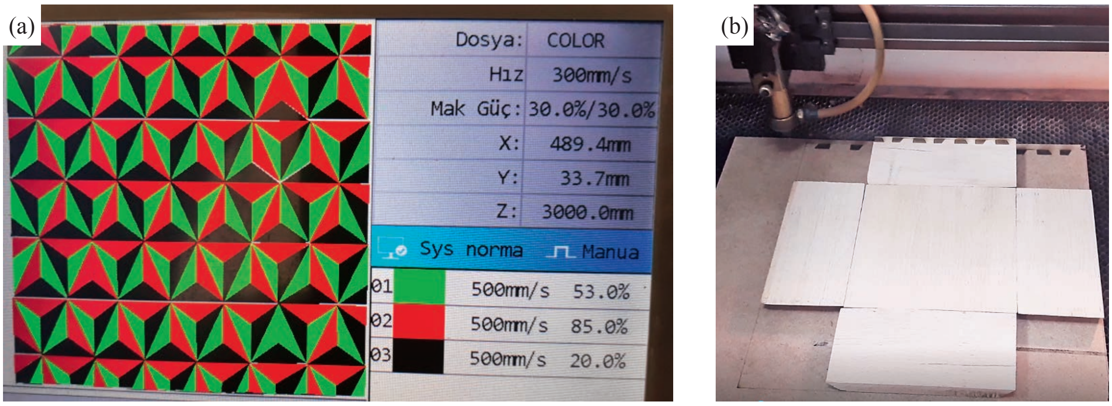

Figure 3 (a) CNC laser product manufacturing design, (b) CAM manufacturing initial stage
Slika 3. (a) Dizajn proizvodnje CNC laserskog proizvoda, (b) početna faza CAM proizvodnje

*Engraving power**-0.607= 46.993+(500*0.050)-20 (Projected white color)

Engraving power= 46.993+5/0.607=85.65

L^*_2=46.993+(*Engraving power**-0.607)+(*Engraving speed**0.050)

40 =46.993+(*Engraving power**-0.607)+(*Max speed**0.050)

*Engraving power**-0.607= 46.993+(500*0.050)-40 (Projected white color)

Engraving power= 46.993-15/0.607=52.70

L^*_3=46.993+(*Engraving power**-0.607)+(*Engraving speed**0.050)

60 =46.993+(*Engraving power**-0.607)+(*Max speed**0.050)

*Engraving power**-0.607= 46.993+(500*0.050)-60 (Projected white color)

Engraving power= 46.993-35/0.607=19.75

The required engraving power ratios were determined as 85 %, 53 % and 20 % in order to perform the prescribed laser engraving processes with a whiteness value of 20.40 and 60 at 500 mm/s engraving speed. In Figure 3a, the design is ready for production in the CAM program and in Figure 3b, the initial stage of surface processing is shown.

The CAM design, developed with the obtained parameters, was completed and the production phase was started. The completed surface treatment is shown in Figure 4a, the local view of the motif processed at 20 %, 55 % and 85 % speed rates is shown in Figure 4b, and the perspective of the finished wooden box in Figure 4c.

4 CONCLUSIONS
4. ZAKLJUČAK

In this study, the applicability of CNC laser assisted regression modeling method in furniture surface treatments in CIE *L*a*b** color system was investigated. The highest values of the effect of laser wood surface treatment parameters on *L**, *a** and *b** color changes were obtained in the *L** black and white axis. While the laser engraving power increased the black color level, the engraving speed decreased the black color. It has been determined that the regression modeling method can be successfully applied over *L** values in furniture surface decoration with CNC laser, provided that material, machine and processing parameters are taken into consideration. The same research can be done for color types with a higher effect level in studies to be carried out on other tree species. However, in this study, both the effect level of the *a** and *b**

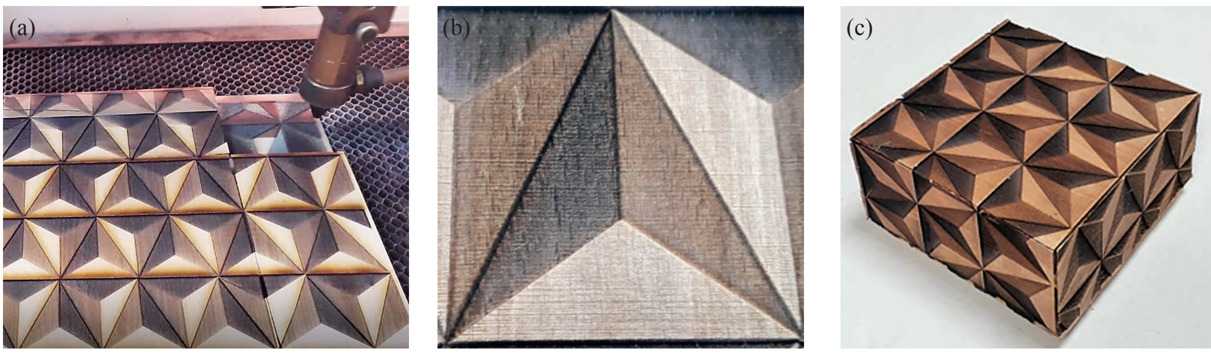

Figure 4 (a) CNC laser engraved surface, (b) Local view of the engraving motif, (c) Assembled wooden box
Slika 4. (a) CNC laserski gravirana površina, (b) lokalni prikaz motiva za graviranje, (c) sastavljena drvena kutija

color groups was low and the regression relationships were low. When a good design is made, the color tones from natural wood color to black on the engraving surfaces add value to the decoration, depending on the processing power of the laser.

If the motif design, color design and production parameter process in laser wood decoration are not well managed, poor images may be encountered in color contrasts on the decoration surface. While it is desired to obtain darker colors, layer differences on the surfaces may be a disadvantage due to the engraving depth. As a result, this study has shown that the regression modeling method of the CNC laser engraving technique can be applied in furniture top surface color design in mass production in accordance with the industrial engineering approach, although it has some limitations.

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Timber Strength Grading as Necessary Basis for Structural Design in Ex-YU Region: Part 1

Ocjenjivanje drva prema čvrstoći kao nužna osnova za projektiranje konstrukcija na području bivše Jugoslavije: dio 1.

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ABSTRACT • *Classification of timber for various commercial purposes is essential for its proper application in order to ensure the reliability and economic use. Visual grading of structural timber is commonly used in a number of EU countries, with different grading national standards optimized for locally available wood. Countries in the ex-YU region are traditional partners in wood trade and had the same standards for visual grading, but in most of the regions these standards are not completely compliant with EN requirements. Consequently, that leads to the fact that the most of regionally available structural timber is not assigned into strength classes, which is the starting point for the limit-state concept in design of timber structures. The aim of this paper is to emphasize the lack of strength classification of structural timber in the ex-YU region, which is a prerequisite for the design of timber structures made by civil engineers. Based on an overview of visual classification types with regional experience in grading, relevant EN standards, and differences in design concepts with possible consequences of grading approach, it can be concluded that "quality" grades and "strength" classes are not easily comparable.*

KEYWORDS: *structural coniferous timber; visual grading; strength classes; safety factors; European standards*

SAŽETAK • *Klasifikacija drvne građe za različite komercijalne namjene ključna je za njezinu pravilnu upotrebu kako bi se zajamčila sigurna i ekonomična uporaba. Vizualno ocjenjivanje konstrukcijskog drva obično se provodi u mnogim zemljama EU-a uz pomoć različitih nacionalnih standarda ocjenjivanja optimiziranih za lokalno dostupno drvo. Zemlje bivše Jugoslavije tradicionalni su partneri u međusobnoj trgovini drvom i imale su zajedničke standarde za vizualno ocjenjivanje drvne građe, ali u većini njih ti standardi nisu u potpunosti usklađeni s EN zahtjevima. To posljedično rezultira činjenicom da većina regionalno dostupnoga konstrukcijskog drva nije*

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razvrstana u klase čvrstoće, što je polazište za koncept graničnog stanja u projektiranju drvnih konstrukcija. Cilj rada jest upozoriti na nepostojanje klasifikacije čvrstoće konstrukcijskog drva na području bivše Jugoslavije, a to je preduvjet za projektiranje drvnih konstrukcija, što je posao građevinskih inženjera. Na temelju tipova vizualne klasifikacije i regionalnih iskustava u ocjenjivanju, relevantnih EN normi te razlika u konceptima projektiranja s mogućim posljedicama pristupa ocjenjivanju, zaključeno je da ocjene razreda kvalitete i klase čvrstoće u promatranim zemljama jednostavno nisu međusobno usporedive.

KLJUČNE RIJEČI: *konstrukcijsko crnogorično drvo; vizualno ocjenjivanje; klase čvrstoće; faktori sigurnosti; europske norme*

1 INTRODUCTION
1. UVOD

Wood as a building material has been used for centuries, but during the past few decades, it has been recognized as one of the favourite materials in the construction sector. Renewable sources, availability in the immediate surroundings, relatively low energy consumption to process in addition to continuous construction technology innovation have all contributed to the wider affirmation of wood-based products in modern architecture, providing extraordinary possibilities in the shaping and design of structures, Figure 1.

The built environment generates 47 % of annual global CO_2 emissions: building operations are responsible for 27 %, while building materials and construction (typically referred to as embodied carbon) are responsible for an additional 20 % (Agopian, 2022). Growing awareness leads to wood becoming the trending material of the 21st century, in the form of solid and engineered wood products (EWPs) for structural and indoor/outdoor use. Modern architectural trends in prefabricated residential buildings and large-scale timber construction lead to eco-friendly urbanization, where timber grows in popularity compared to steel, concrete and other mineral-based building materials. According to Hetemaki *et al.,* 2017, the indirect share estimation of timber construction was 15 % in turnover and 19 % in employment of the total EU building construction sector. On the basis of recent decade production of wood fibre insulation boards, glulam, cross-laminated timber and laminated veneer products, the estimation of growth rates is in a range from 2.5 % to 15 % (Hildebrandt *et al*., 2017), which could create an added value by a lower environmental impact throughout the entire life cycle. Previous low economic competitiveness of multi-storey timber buildings has been overcome by technological progress in EWPs production, so the share of timber construction in total building market increases in the EU and world. The share of "wooden houses" depends on the region, forest resources and on tradition of building with timber, as well as on technological opportunities (North and Western Europe N/WE) and/or political and economic opportunities (South Europe SE). It was found that "social and political barriers most typically limit the development of wood construction" (Leszczyszyn *et al.,* 2022), which means that it is necessary to provide the basic knowledge and social conditions for increasing the share of wood in countries where the use of timber is still under the average EU level.

South-Eastern Europe (SEE region) has a long tradition in building with timber, which was first reflected in the craftsmanship and later in industrial approach to construction. Nowadays, structural engineers and architects in the ex-YU region are faced with two challenges: the imperative to increase the use of timber in the construction sector and the harmonization / adjustment of regulations with the recently introduced safety concepts in design. The transition from the concept of global safety factors, which refers to common ex-YU design codes (JUS), to the concept of partial safety factors, which re-

a) b) c)

Figure 1 a) Pancho Aréna Hungary (2014), b) Mjøstårnet tower Norway (2019), c) Mistissini Bridge Canada (2014)
Slika 1. a) stadion Pancho Aréna, Mađarska (2014.), b) neboder Mjøstårnet, Norveška (2019.), c) most Mistisini, Kanada (2014.)

fers to the set of Eurocodes (ENs) in design of timber structures, requires more precise classification of structural timber and products, where cooperation between wood processing industry and civil engineers is necessary, as well as between countries that traditionally trade in timber in the region.

SEE region is considered as the smallest forest area in total (30446·10^3 ha) and in forest share (23.5%) compared to the rest of Europe, e.g. the highest forest area share is 53.2 % in NE (Alexandrov and Iliev, 2019). Data on the forestry potentials in the ex-YU countries (Bosnia and Herzegovina, Montenegro, North Macedonia, Serbia as well as Croatia and Slovenia as EU members), as part of the SEE region, are given in Table 1, where the data on coniferous sawn wood as the basic raw material for construction is particularly highlighted. Data is given according to FAO database (FAO, 2022).

Table 2 presents the data about wood product trade i.e. coniferous sawn wood export/import in six ex-YU countries, together with leading trade partners. The data is given according FAOSTAT and INDEXBOX platforms (FAOSTAT, 2021; INDEXBOX, 2022). Strong trade connections between the analyzed countries are evident, besides traditional partner countries (Austria, Germany, Italy) and some new ones (Albania, Turkey, Bulgaria, Slovakia and Czech Republic).

Based on natural recourses, Bosnia and Herzegovina and Slovenia are recognized as the main suppliers of sawn coniferous wood in the ex-YU region, which dates back to the past. The existing trade and interconnection of the countries based on tradition and common regulations for the visual classification of sawn wood in the ex-YU area indicates the necessity to harmonize the quality assessment of structural coniferous timber in the sense of EU requirements and construction industry needs.

This paper is limited to the SEE ex-YU countries because of previous common legislation in construction and forestry domain standards and a number of existing timber structures from earlier period (XX century). Besides, sawn-wood mutual export/import is still

Table 1 Forest area, growing stock and sawn wood production in ex-YU countries (2020/21)
Tablica 1. Površina šuma, drvne zalihe i piljeno drvo u zemljama bivše Jugoslavije (2020./21.)

Country *Zemlja*	**Forest area** *Površina pod šumom*		**Growing stock** *Drvna zaliha*		**Sawn wood** *Piljeno drvo*	
Ex-YU	**Total, 10^3 ha** *Ukupno, 10^3 ha*	**Of land area, %** *Zemljišne površine, %*	**Total, 10^6 m^3** *Ukupno, 10^6 m^3*	**Coniferous, 10^6 m^3 / %** *Četinjače, 10^6 m^3 / %*	**Total, 10^3 m^3** *Ukupno, 10^3 m^3*	**Coniferous 10^3 m^3** *Četinjače, 10^3 m^3*
Bosnia & Herzegovina / *Bosna i Hercegovina*	2188	42.7	404.7	n/a	1650	743
Croatia / *Hrvatska*	1939	34.7	427.2	50.6 / 11.8	1298	257
Montenegro / *Crna Gora*	827	61.5	121.4	42.8 / 35.3	109	108
North Macedonia *Sjeverna Makedonija*	1001	39.7	76.4	n/a	7	2
Serbia / *Srbija*	2723	31.1	420.9	27.0 / 6.4	452	99
Slovenia / *Slovenija*	1238	61.5	414.1	172.0 / 41.5	1029	904

Table 2 Wood product trade in ex-YU: coniferous sawn wood (10^3 m^3) (2021)
Tablica 2. Promet proizvoda od drva na području bivše Jugoslavije: piljena građa četinjača (10^3 m^3) (2021.)

Country *Zemlja*	**Consumption** *Potrošnja*	**Production** *Proizvodnja*	**Export** *Izvoz*	**Import** *Uvoz*	**Leading partners** *Vodeći partneri*	
					Export *Izvoz*	**Import** *Uvoz*
Bosnia & Herzegovina *Bosna i Hercegovina*	32.3	743.0	717.9	7.2	Serbia, Croatia, Albania, Italy, Austria	Austria, Croatia, Serbia
Croatia *Hrvatska*	343.3	256.5	265.0	351.8	Slovenia, Italy, Austria	Austria, B&H, Slovenia
Montenegro *Crna Gora*	17.4	107.8	93.6	3.2	n/a	B&H
North Macedonia *Sjeverna Makedonija*	45.1	2.0	0.5	43.6	Turkey, Italy, Serbia	B&H, Montenegro, Bulgaria
Serbia *Srbija*	379.0	99.0	15.0	295.0	Italy, B&H, Germany	B&H, Montenegro, Austria
Slovenia *Slovenija*	693.1	904.0	851.6	640.7	Italy, Austria, Croatia	Austria, Czech Rep., B&H, Slovakia

very frequent in the region although two countries are the members of the EU. In order to understand the importance of the transition problem from quality (stress) grades to the system of strength classes of structural timber, and to obtain the relevant conclusions, an overview of visual classification types with regional experience in grading, differences in design concepts, relevant standards, as well as possible consequences, is given in the next chapter. The final goal of the present paper (Part 1) is to emphasize the necessity to assign visual grades and species to strength classes of structural timber in the region. In Part 2 of the paper, the integral procedure for the conversion of the II grade coniferous timber into strength classes is presented and its application is illustrated through the analysis of archive data sample obtained from regional sources.

2 VISUAL GRADING AND TIMBER STRUCTURAL DESIGN
2. VIZUALNO OCJENJIVANJE I PROJEKTIRANJE DRVNIH KONSTRUKCIJA

2.1 Importance and types of timber visual grading
2.1. Značenje i načini vizualnog ocjenjivanja drva

The quality assessment of wood as a material is crucial for engineering design, and therefore for the structure reliability. Without knowledge of the theory and practice of classification, there may be misunderstandings that threaten the structural safety, while the grading system is expected to be as uniform and unified as possible within the European market. For the correct use of timber in constructions, it is necessary to carry out adequate classification, because today civil engineers face two problems: assessing the quality of timber built in existing buildings due to the need for reconstruction, as well as assessing the quality of locally available wood for use in newly designed buildings.

Wood as a material could be classified at different stages of harvesting and/or production using different procedures, which are initially based on visual inspection and appropriate measurement. Thus, after a rough dimensional classification (EN 1315) of timber, when the final use is still unknown, the roundwood classification is made according to quality (classes A, B, C, D) in accordance with EN standard (e.g. EN 1927 or EN 1316) depending on the type/species of wood and the presence, size and distribution of factors that affect quality (EN 1309). These EN standards are officially in force in ex-YU countries, although not-binding, because they are intended for the log trade where different conditions can be agreed in trade contracts (e.g. traditional quality according to previous JUS). Generally, from the forestry aspect, the main difference in the classification of roundwood lies in the fact that EN standards classify according to quality without prejudicing its future purpose, while JUS classified according to its final use. According to ex-YU regulations, in addition to being classified into dimensional grades, logs and round timber had to be classified into quality classes for intended use, where, for structural purposes, the classification of sawmill logs into three categories (I, II, III) is of importance for designers.

Sawn timber could be used for non-structural and structural purposes, so different grading procedures could be applied due to the final use of the material. Generally, the grading process is always based on visual sorting, but opposite to grading of non-structural elements that is exclusively based on (surface) appearance, grading of structural elements also considers determination of relevant strength & stiffness properties, i.e. visual grading is followed by strength classification and testing. Appearance grading is a process of assessing the prescribed number and size of parameters by visual sorting and it is not designed to take into account the final use of timber (e.g.: linings, joinery, packaging or construction). For example, acceptable timber for a structural engineer in terms of bearing strength could be completely unacceptable for an architect in appearance (Swedish wood, 2023).

In the ex-YU region, the structural grading meant assessing the timber grades for construction purposes by adequate appearance requirements (particularly related to size and position of knots and their perceived effects on bending strength and stiffness) as useful indication, performed by experienced operators. Such assessment was often followed by additional testing of load-bearing properties on relevant samples by destructive, non-destructive or semi-destructive methods (Nowak *et al.*, 2021). Machine strength grading is not present in the region, while evaluation by non (semi) destructive methods are not widely used, except for research purposes or for *in-situ* evaluation of existing buildings with high importance (Stepinac *et al.,* 2017).

In order to preserve traditional national grading systems, and to provide the necessary unification level as well, EU introduced the standard EN 14081-1 for structural timber of rectangular cross-section with prescribed general requirements. The standard gives general limitations in 3 aspects of strength reducing characteristics that have to be taken into account during visual grading process as basic principles: Limitations for strength and stiffness reducing characteristics (knots, slope to the grain, density and rate of growth, fissures); Limitations for geometrical characteristics (wane, warp); Limitations for biological characteristics (insect and fungal damage).

The existing regional standards for visual grading of timber (ex JUS U.D0.001/1983) are based on

DIN 4074:1958. The changes introduced in DIN during the harmonization with EN 14081-1 were incorporated in national legislation of Slovenia and Croatia (2009), while in the rest of the region the grading standard remained unchanged. Due to new demands in timber industry (use of smaller/slender timber pieces in construction and laminated products), the crucial novelties introduced in DIN 4074-1:2012 are the four kinds of cross-section with different specifications and possibilities of flatwise and edgewise orientation of planks and boards. That imposes that different orientation of the same slender timber element could lead to different "quality" grade because of different criteria due to final position of timber element in the structure. Although this looks very similar compared to previous versions of the standard, the amendment about possibility of edgewise orientation of boards and planks may have significant implications for the timber classification and its use in construction, particularly when designed according to EC5 (EN 1995-1-1, 2004).

2.2 Design concepts of timber structures: global vs. partial safety factors
2.2. Koncepti projektiranja drvnih konstrukcija: globalni nasuprot parcijalnim činiteljima sigurnosti

The safe use of timber in construction is a question of design and quality of chosen timber (products). All engineering design methods can be reduced to the basic concept of safety, according to which the design resistance of structural elements should be greater or equal to the effects of design loads (actions), with adequate safety factors. The way of providing a safety factor determines the concept of design: deterministic ("working" - allowable stress design (ASD) with global safety factor) or reliability-based (semi-probabilistic) design concept (limit states design (LSD), with partial safety factors). In the basic form of ASD, the factor that provides safety is applied only to the resistance of structural material, while loading is generally taken as nominal, with some modifications due to load-duration effects and load combinations. LSD takes into account the uncertainty of inaccurate models and unfavourable deviations associated with strength properties, but also with uncertainties in the assessment of the effects of actions.

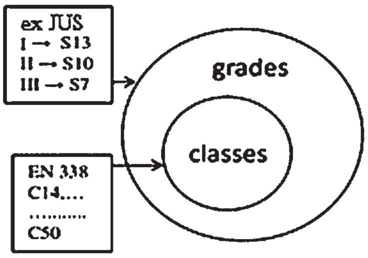
ASD has served for a long time to provide a simple approach to design procedure, with uncertainty in reliability of structures designed with such a procedure. The analysis performed in the ex-YU region showed that: "the previous (JUS) regulations have large deviations in reliability depending on the location of the structure. The reliability of constructions in areas with higher snow load is insufficient..." (Čizmar *et al.,* 2018). That reflects disadvantages in aspects of selection of different factors of safety by "intuition" regarding the variety of materials and by using the same factor of safety for all types of load. Global safety factors for different stress states in the ASD concept are in the range of $n = 2 - 4$, where permissible stress is based on extensive research of properties by testing small clear wood samples. The absence of strength-reducing characteristics and favourable orientation on small clear specimens provide an indication of the upper limit of the expected performance of realistic timber pieces (Crews and Ritter, 1996), so "the critical design criterion is not how strong a piece of timber is, but rather how weak it could be". ASD codes have been focused on providing adequate strength and achieving a proposed level of safety, while LSD ones, besides strength limit state, also recognize other limit states, such as serviceability, stability, fire and fatigue. In LSD, the partial safety factors for strength limit state and timber materials are in the range of γ_m = 1.25-1.30 that indicate lower safety factor and require higher reliability in assessment of relevant timber properties.

The conclusion of rough comparison of ASD and LSD is that global and partial safety factors are different in "composition" and in values. LSD, as advanced and more reliable concept, requires different experimental procedures and statistical distribution models of testing data, followed by improved visual classification of timber.

3 STRENGTH CLASS SYSTEM IN EUROPEAN STRUCTURAL CODES
3. SUSTAV KLASA ČVRSTOĆE U EUROPSKIM KODOVIMA ZA PROJEKTIRANJE

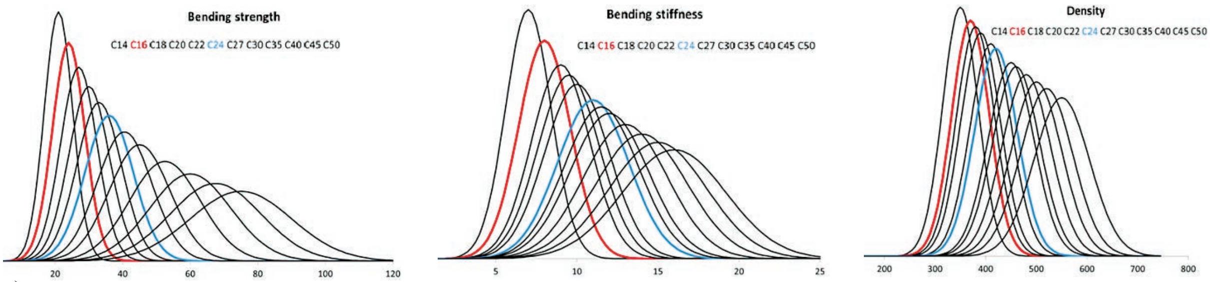
3.1 Visual ("quality" or "stress") grades vs. strength class system
3.1. Vizualne ocjene („kvaliteta" ili „naprezanje") u odnosu prema sustavu klase čvrstoće

In a new era of timber structures design, the first challenge for the engineers is handling the classification of timber as a structural material in order to obtain the input data for calculations. Namely, according to ASD concept, the structural coniferous timber in the region was visually graded into quality "stress" grades (I, II, III) in accordance with DIN 4074 standard (S13, S10, S7). In contrast to that, LSD concept introduced in Eurocode 5, considers the so called "strength class system" (EN 338) of softwood (12 classes - coniferous and poplar), where structural timber is graded based on a set of rules given in EN 14081-1 and supporting ENs. It is obvious and important to emphasise that quality grades are wider terms than strength classes (SC), Figure 2, as well as that "strength classes" are not based only on strength but also on stiffness and density of timber from a particular source and region. "Strength



EU countries	Germany / Austria	Slovenia	Balkan countries	Great Britain	Italy	Nordic
National standards	*DIN / ON 4074-1*	*SIST DIN 4074-1*	*Ex JUS*	*BS 4987*	*UNI 11035-2*	*INSTA 142*
Visual grades	S13 S10 S7	S13 S10 S7	I II III	SS GS	S1 S2 S3	T3 T2 T1 T0

b)



c)

Figure 2 a) Grades vs. strength classes, b) Visual grades according to national standards, c) Strength classes EN338 - class determining properties (Ridley-Ellis *et al*., 2016)
Slika 2. a) Ocjene u odnosu prema klasi čvrstoće, b) vizualne ocjene prema nacionalnim standardima, c) klase čvrstoće EN338 – svojstva koja određuju klasu (Ridley-Ellis *et al*., 2016.)

class system" groups together grades and species with similar strength properties making them interchangeable. Additional species/grades can be incorporated into the system at any time, and engineers could "specify a chosen strength class in their calculations without awareness of costs or availability of alternative species or grades" (EN 338). Due to the number of classes and grading precision, "SC system" is primarily optimized for machine grading (EN 14081:1-5) and large sawmills with huge industrial capacity.

Despite machine grading, visual grading is still in common use in a number of European countries (including ex-YU region), with different national grading standards optimized for locally available wood. Due to diversity of locally spread wood, it is impossible to lay down a single standard for all EU Member States, so the prescribed general requirements for structural timber (within EN 14081-1) have to be met by national standards. It is necessary to respect EN 14081 recommendations in order to avoid large inconsistency in classification criteria and further application of timber (Prka *et al.,* 2001; Ištvanić *et al*., 2008). Due to evident overlapping of SCs, Figure 2c, and economic reasons, it is useful to adopt a small number of SCs in design practice (Bather, 2021).

3.2 Relevant EN standards for testing and quality control of structural timber
3.2. Relevantni EN standardi za ispitivanje i kontrolu kvalitete konstrukcijskog drva

In order to provide an adequate and consistent basis for the introduction of "SC system" in design of timber structures and on the EU market, a set of standards for testing and statistical procedures are established, accompanied with a standard that allows the assignment of visual grades and species of locally spread wood into strength classes. Harmonized normative standards that follow general requirements in EN 14081-1 are:

- EN 338: Structural timber - Strength classes (provides characteristic values of strength, stiffness and density for softwood and hardwood, where the dominant classification is made by designation due to characteristic value of bending strength).
- EN 408: Timber structures - Determination of some physical and mechanical properties (specifies test methods for determining the structural properties).
- EN 384: Structural timber - Determination of characteristic values of mechanical properties and density (provides a procedure to derive characteristic values that are comparable in terms of population. The standard permits the use of as much existing test data as possible from various sampling and testing techniques).
- EN 14358: Timber structures - Calculation and verification of characteristic values (gives statistical methods for the determination of characteristic values from test results on a sample drawn from a clearly defined reference population. In case of solid timber, it combines with specific adjustment factors given in EN 384).
- EN 1912: Structural timber - Strength classes - Assignment of visual grades and species (national document that lists visual strength grades, species and sources of timber specifying the strength classes to

which they are assigned according to tradition and adequate quality control).

Evaluation of structural timber of the ex-YU region from visually established 3 quality grades (ASD) into numerous strength classes (LSD) is a demanding but necessary task for structural designers in the region that implies knowledge about standardized testing methods and statistical procedures for converting the archive data and proper analysis of newly obtained data. In ASD, the leading parameter of classification was the mean value of bending strength, tested by 3-point test on small clear specimens, while stiffness parameter (bending modulus of elasticity II to the grains) together with density were given as general mean values that slightly differ for solid and glulam timber. In LSD concept, a detailed procedure of quality assessment is based on 3 reference material properties (5 % characteristic bending strength, mean stiffness and 5% characteristic density), where the minimum value is relevant for strength class estimation (EN 338). The reliability basis for strength class system is given in JCSS Probabilistic model code: preferred theoretical distributions and desirable coefficients of variations (CoV) of referent properties, while the established relationships with other relevant material properties are given in Table 2 (EN 384). Test methods (EN 408) for "key" strength and stiffness properties in bending (overall span 18 times the specimen depth, including full size specimens) are based on 4-point test. Modulus of elasticity (*MoE*), considered as "the best single indicator of timber quality" (Bostrom, 1999), could be measured as local and global, which gives the opportunity to use the archive data from previous tests where the *MoE* is measured as global. Determination of wood density is provided on small defect free specimens (EN 408), which is consistent with the previous JUS standards.

Once properties are determined through EN 408, standards EN 384 and EN 14358 give the necessary statistical basis and methods for determining the characteristic values of mechanical properties and density for defined populations of visual grades and/or strength classes of machine graded structural timber. These standards provide the possibility of assessment by "calculation" of archive data results obtained from previously conducted test under different load arrangements from defect-free specimens or from products of structural size.

Finally, by applying to CEN/TS 124 with a documented report, EN 1912 will publish the list of assignments of local wood to strength classes according to EN 14081-1 and EN 338. This list is not exhaustive, but in every moment gives good guidelines for the quality of trade market and input data for architectural and civil engineers' design projects. With an insight into prEN 1912:2022, it can be concluded that only Slovenia, of all the ex-YU countries, has implemented this procedure and has classified its national timber resources into strength classes guided by national SIST standards (mixed spruce/fir of grade S10 is assigned to C24, of grade S7 to C18, while only fir of grade S7 is assigned to C16).The official assignment is very useful for structural engineers because some provisional explanations could be found in the regional documents (e.g. JUS grade I is sometimes assigned to series of SCs from C30 to C50, II grade as C24-C27, while III as C22). That kind of "assignment" overestimates the structural timber from regional recourses and could mislead the designers.

Regardless of the fact that the classification of timber into the strength class is not a prerequisite for trade, it is a condition for the CE marking and for placing timber on the EU market (Negro *et al.,* 2013). In the ex-YU region, apart from large glulam factories (e.g. "Voćin", Croatia) that closely cooperated with EU companies, strength classified timber is not offered in the regional trade. That requires a mutual effort in which structural engineers and engineers from forest-based industries should participate in order to ensure proven quality for the design and additional trade value.

4 CONCLUSIONS
4. ZAKLJUČAK

The consideration of EN standards, JUS visual grading rules and practice, limit state design approach with sensitivity on consistent grading, leads to the following conclusions:

The substantial improvement of the regional visual grading rules must take into account the future position of the element in the structure i.e. visual assessment has to be conducted for edgewise and/or flatwise orientations of the boards. In addition, in a lack of machine grading, the regional visual grading of structural timber must be consistent and stricter in application of EN requirements because of smaller safety material factors in structural design compared with the previous global ones.

The proclaimed SC system with classes from C14 to C50 (although the highest recognised timber class in EU is C35 with limited application) is established by statistical tools with overlaps in relevant parameters and it is optimized for machine grading. In visual grading it is practical and effective to have only a few SC with consistent grading rules defined by final purpose in structure (e.g. Sweden established classes with structural description of use: C14, C18, C24 for normal structural use and C30, C35 for extra load-bearing purposes but not for elements of large dimensions).

It is important to notice that SC classification of coniferous solid timber boards directly influences the production and classification of glued laminated products (glulam and cross-laminated timber), the prefabricated products that are the essence of modern building with timber.

Although the strength classification of timber is not necessary for regional trade, for structural engineers and designers it is of high importance to have a framework of available construction timber in the ex-YU region (Part 2). Otherwise, the overestimation or underestimation by random selection of SC could lead to inadequate or low quality projects, as well as to high prices and conflicts with investors and suppliers.

The joint efforts between civil and wood processing engineers are necessary in order to classify regional structural timber according EN requirements. One of the first tasks is to assign visual grades and regional species of structural timber to strength classes according to EN 1912.

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Comparison of Reaction Wood and Normal Wood of Some Commercial Tree Species

Usporedba reakcijskoga i normalnog drva nekih komercijalnih vrsta drva

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ABSTRACT • *This study aims to analyze the reaction wood samples for some industrial tree species naturally grown in Kastamonu province in Turkey and compare them with the relevant standards. Some anatomical, chemical, fiber morphological, optical properties, and color changes before and after drying were analyzed for the reaction wood (RW) samples. While the holocellulose content of fir and pine compression wood (CW) was found to be lesser (~3-4 %), the lignin content was higher than those of the opposite wood (OW) (~34 % for pine and 12 % for fir). On the contrary, the amount of holocellulose was found to be higher (~1-4 %), and the lignin was lower (at about 6-15 %) in the tension wood (TW) samples. It was observed that average lengths are more extended in TW (~50-54 %) and shorter in CW (~13-17 %) than those of OW. Significant differences were observed between the anatomical structures of the coniferous and deciduous species studied. Although, the greatest color differences in wet and oven-dried samples of coniferous trees were measured in CW (~15-17 %), it has been found as about 0.7-3 % in TW for deciduous species. Some differences were observed in the anatomical, optical, fiber morphological, and chemical properties of the RW for the studied wood species. Due to its higher lignin content and better physical properties, CW can be used for producing small households and hand tools, ornaments, toys, etc. It will also be appropriate for use in milling and turning work. It is recommended that, because of the lower lignin content and higher polysaccharide ratio, TW should be primarily used for the cellulose, pulp, and paper industries, where high mechanical resistance values are required. Consequently, RW formation causes some physical, chemical, mechanical, anatomical, and optical differences compared to OW in deciduous and coniferous species.*

KEYWORDS: *pine; fir; oak; beech; anatomical and chemical structure*

SAŽETAK • *Cilj ovog istraživanja bio je analizirati uzorke reakcijskog drva nekih industrijskih vrsta drva koje prirodno rastu u pokrajini Kastamonu, u Turskoj, i usporediti ih s relevantnim standardima. Na uzorcima reakcijskog drva (RW) analizirana su njihova anatomska i kemijska svojstva, morfološka svojstva vlakana te optička svojstva i promjena boje prije i nakon sušenja. Sadržaj holoceluloze u kompresijskom drvu (CW) jelovine i borovine bio je manji nego u opozitnom drvu (OW) (~3 – 4 %), dok je sadržaj lignina u kompresijskom drvu bio veći nego u opozitnome (~34 % u borovini i 12 % u jelovini). Naprotiv, utvrđeno je da je u uzorcima tenzijskog drva (TW) količina holoceluloze veća (~1 – 4 %), a lignina manja (~6 – 15 %). Uočeno je također da su prosječne duljine*

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vlakanaca u tenzijskom drvu veće (~50 – 54 %), a u kompresijskom drvu manje (~13 – 17 %) nego u opozitnome. Nadalje, uočene su značajne razlike u anatomskoj strukturi između proučavanih crnogoričnih i bjelogoričnih vrsta drva. Iako su u crnogoričnih vrsta drva razlike u boji između mokrih i suhih uzoraka bile najveće u kompresijskom drvu (~15 – 17 %), u bjelogoričnih su vrsta drva razlike u boji bile veće u tenzijskom drvu, i to za oko 0,7 – 3 %. U proučavanih vrsta drva uočene su neke razlike u anatomskim, optičkim, morfološkim i kemijskim svojstvima reakcijskog drva. Zbog većeg sadržaja lignina i boljih fizičkih svojstava kompresijsko se drvo može upotrebljavati za proizvodnju malih kućanskih i ručnih alata, ukrasa, igračaka itd., a prikladno je i za glodanje odnosno tokarenje. Preporučuje se da se tenzijsko drvo zbog nižeg sadržaja lignina i većeg omjera polisaharida primarno rabi u industriji celuloze i papira, gdje je potrebna velika mehanička otpornost. Stvaranje reakcijskog drva posljedično uzrokuje neke fizičke, kemijske, mehaničke, anatomske i optičke razlike u usporedbi s opozitnim drvom bjelogoričnih i crnogoričnih vrsta.

KLJUČNE RIJEČI: *borovina; jelovina; hrastovina; bukovina; anatomska i kemijska struktura*

1 INTRODUCTION
1. UVOD

The wood material is important globally because it is both natural and sustainable. Turkey has a total forest area of 22,740,297 hectares, while the productive forest area is 13,830,510 hectares. Oak, pine, beech, juniper, and fir are the first five most important commercial tree species, with the highest distribution in Turkey (Kuzka, 2013; OGM, 2021).

Wood materials are used for various purposes, such as fiber-firewood in the paper industry, wood-based material production, etc. Although Turkey's total annual wood consumption is 32 million m^3, the annual wood production is 26.3 million m^3. The difference between production and consumption is supplied from private plantation forests (5 million m^3) and covered by imports (1.5-2 million m^3) (OGM, 2021).

Reaction wood, formed under the effect of forces that bend wood due to external factors (wind, etc.), affects the end-product properties of the wood (Rowell, 2005). These defects are CW (compression wood) in coniferous trees and TW (tension wood) in hardwoods. In CW, force is exerted on the lower side of the bent stem to make the trunk upright, while in TW, pressure is applied on the upper side of the bent stem. These abnormal formations in wood sourced from RW (reaction wood) are seen as defects in living trees and sawn timber (Donaldson and Singh, 2016). Almost all forests are located on sloping lands in Turkey. Therefore, the reaction wood formation rate is high in industrial woods (OGM, 2022).

There are many studies on reaction wood in the literature. Köksal and Kılıç Pekgözlü (2016) investigated the microscopic structures of *Pinus sylvestris L.*, *Pinus nigra Arnold,* and *Pinus brutia Ten.* compression woods in their study. Ruelle *et al.* (2010) examined the physical and mechanical properties of CW and OW (opposite wood) samples from three different tropical tree species. It has been determined that the compressive strength of TW is generally lower than that of OW. Geffertova *et al.* (2019), in their study with beech TW samples, determined that the samples fiber length and width were higher than those of OW.

In this study, RW samples taken from fir (*Abies nordmanniana* (Stev.) Spach. subsp. *bornmuelleriana*), pine (*Pinus nigra Arn.* subsp. *pallassiana* (Lamb.) Holmboe), oak (*Quercus robur* L.), and beech (*Fagus orientalis* Lipsky) were studied in terms of anatomical, chemical, fiber morphological, and optical properties. Besides, some evaluations and comparisons between RW and OW, coniferous and deciduous woods, and inter-species properties have been reported.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materijal
2.1. Materijal

2.1.1 Study area
2.1.1. Istraživani lokalitet

The wood samples were taken from Bostan Forestry Management Chiefdom, located within the borders of Kastamonu/Turkey. The area of the management directorate is 8,287.7 hectares, the forest area is 6,378.4 hectares, and the open area is 1,910.3 hectares. It is located between 41º02'58''- 41º09'57'' north latitude and 33º40'33''- 33º51'50'' east longitude. The highest point is Büyük Hacet Hill, with a height of 2,587 m, and the lowest point is 1,155 m, where Karasu Stream and Açlık Stream meet. Samples containing RW were selected from log storage areas of the Kastamonu Forest Regional Directorate.

2.1.2 Sample collection and preparation
2.1.2. Prikupljanje i priprema uzoraka

For each tree species, three wood disks were taken from 1.30 m of breast height with a 10 cm thickness. The RW and OW zones of the disk samples were determined according to Figure 1. All RW and OW zones of the wood disks were cut carefully and chipped to the size of a matchstick (0.5-1.0 mm). Air-dried and grounded in a Willey-type mill, wood samples were

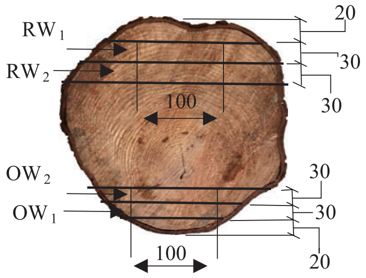

Figure 1 Preparation of RW (reaction wood) and OW (opposite wood) samples for analyses (mm)
Slika 1. Priprema uzoraka RW-a (reakcijskog drva) i OW-a (opozitnog drva) za analizu (dimenzije su u milimetrima)

screened through 40 to 60 mesh sieves. The coarse and thick parts were eliminated by staying above 40 mesh, and the thin pieces were eliminated by going below 60 mesh. The remaining material over 60 mesh was used for experiments. The powdered wood was stored in a dry and clean glass jar with its mouth closed.

2.2 Methods
2.2. Metode

To determine the chemical analyses, lignin (TAPPI T 222 om-88), holocellulose (Wise's Chlorite method), alpha-cellulose (TAPPI T 203 os-71), ethanol solubility, 1 % NaOH solubility, hot water and cold water solubility (TAPPI T 207 om-88) experiments were done according to the above applicable standards.

For microscopic examinations, 1.5 cm × 1.5 cm × 1.5 cm samples were taken from the RW and OW regions of the wood. The samples were boiled with distilled water to soften them for better sectioning. The samples were boiled in distilled water until they softened, and then they were soaked in a solution mixture of glycerine, ethyl alcohol, and distilled water (6:2:2 v/v/v) for 20 days (Köksal and Kılıç Pekgözlü, 2016). For diffuse-porous anatomical structured wood species, vessel cells were sampled from the area near the previous annual ring boundary of earlywood zones and from the area near the next annual ring boundary of latewood zones. For the morphological properties of fibers, the chlorite method was used for maceration (Wise and Karl, 1952), and measurements were taken using Digimiser® for at least 150 fiber samples.

For the determination of the optical properties of the samples, the samples were optically measured after both fresh cutting and oven-dried conditions at (37±3) °C for four days. The color coordinates (L^*, a^*, b^*) were measured according to the D65 standard with the Konica Minolta CM2500d device (Geffertova *et al.,* 2019). The percentage rates can be found by dividing the value by the total value and then multiplying the result by 100. The formula used to calculate the percentage is (value/total value) × 100 %.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Chemical analysis
3.1. Kemijska analiza

The holocellulose content in the pine CW was 3.87 % less than in OW and 4.97 % less in fir CW than in OW. Studies conducted on pine show that the holocellulose content is around 60-70 %, and in some studies, its amount increases up to 75 % (Ateş, Kırcı and Tutuş, 2008). In studies on fir species, 70-80 % holocellulose content was determined (Topaloğlu and Erisir, 2018). The details are given in Figure 2.

It has been determined that there may be differences in both the amount of CW and holocellulose content of the coniferous wood samples according to harvested regions (Arslan *et al.*, 2021; As *et al.*, 2001; Ataç and Eroğlu, 2013; Gülsoy and Öztürk, 2015; Popescu *et al.*, 2011).

Lignin content was 12.6 % and 34.8 % higher in coniferous trees compared to OW. Wood properties vary from species to species and within the same species depending on many factors (age, genetic charac-

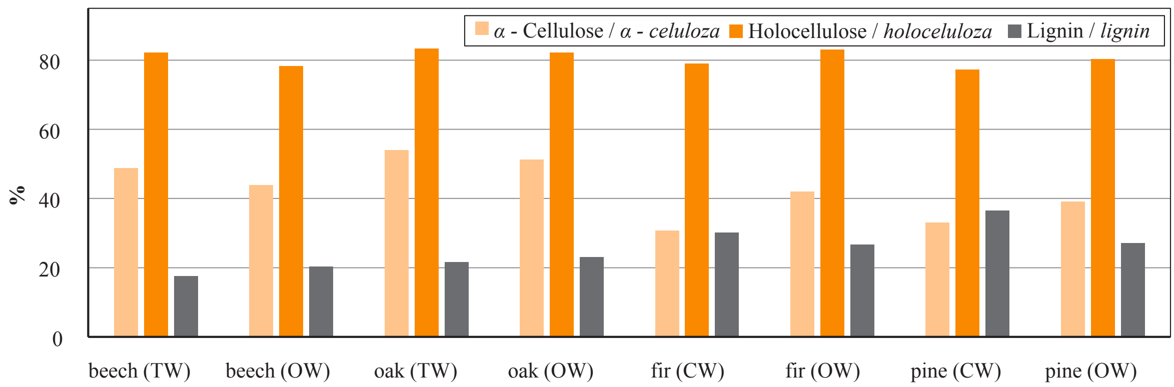

Figure 2 Chemical compositions of reaction and opposite wood of tree species (TW – Tensile wood, CW – Compression wood, OW – Opposite wood)
Slika 2. Kemijski sastav reakcijskoga i opozitnog drva različitih vrsta (TW – tenzijsko drvo, CW – kompresijsko drvo, OW – opozitno drvo)

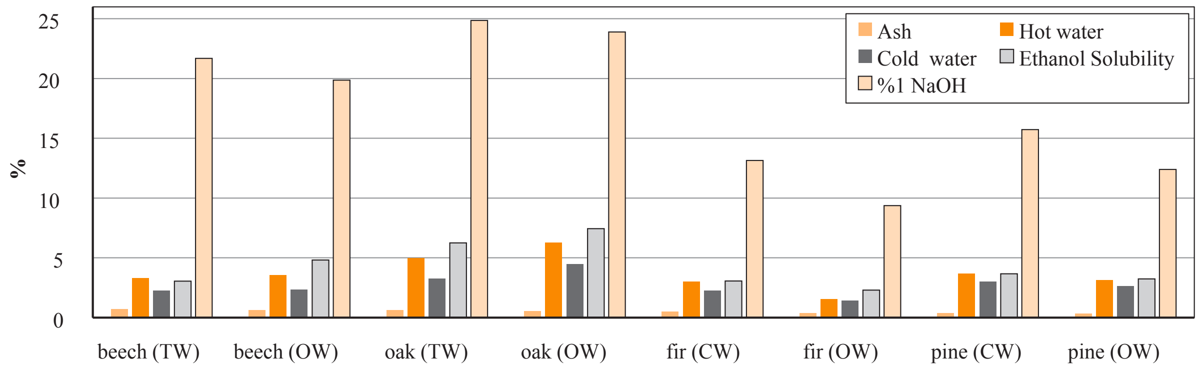

Figure 3 Solubility of reaction and opposite wood of tree species (TW – tensile wood, CW – compression wood, OW – opposite wood)
Slika 3. Topljivost reakcijskoga i opozitnog drva različitih vrsta (TW – tenzijsko drvo, CW – kompresijsko drvo, OW – opozitno drvo)

teristics, environmental characteristics, etc.) (Doğu *et al.,* 2001). The reason why lignin contents are different between pine and fir species is thought to be due to genetic factors. It was observed that the lignin content of TW in the deciduous trees was 15.74 % and 6.68 % in the beech and oak OW, respectively.

Figure 3 shows that ethanol solubility in RW is ~18.25 % greater than that of OW for coniferous wood. The difference is assumed to be caused by changes in the quantity of CW in the tree species (Kılıç *et al*., 2010). While the ethanol solubility of fir species is commonly considered to be around 3 %, pine species have a solubility of 3-4 % (Rowell, 2005). Similar to previous investigations, ethanol solubility was shown to be 11.39 % greater in pine CW compared to OW (Kılıç *et al*., 2010). It demonstrates that fir OW has a lower extractive content than pine samples of about 1.37 %. The extractive content of wood samples for the same species is also affected by variables such as altitude, climate, and so on (Akyıldız and Ateş, 2008). Also, it was determined that the amount of ash in CW of coniferous species was higher (13.15 % in pine and 26 % in fir) than in OW. Compared to the literature findings, alcohol benzene solubility is rising up to ~5.9 % in oak wood (Miranda *et al.,* 2017), and this rate is coming to around 3 % in beech (Malakani *et al.,* 2014). As a result, ethanol solubility was determined to be 5.38 % in coniferous trees (fir, pine) and 3 % in deciduous trees (oak, beech) on average.

The amount of α-cellulose in the samples was 18.48 % and 37.08 % less than OW and CW of pine and fir, respectively. Similarly, α-cellulose content in pine wood was found to be between 35 % and 50 % (Arslan *et al.,* 2021; Kılıç *et al*., 2010). It has been determined that the cellulose content of CW is reduced by up to 30 % compared to normal wood (Bozkurt and Erdin, 2011). The α-cellulose content in fir CW was found to be 58.94 % and 75.82 % lower than beech and oak TW, respectively. It has been observed that the amount of α-cellulose in deciduous trees is generally around 40-50 % (Fišerová *et al*., 2013; Rowell, 2005). The average amount of α-cellulose was found to be 29.82 % lower in CW than in TW.

The hot water solubility of pine CW was found to be 3.69 % and 3.0 % for fir CW. The same solubility was found at 3.16 % and 1.56 % for pine and fir OW, respectively. Compared to deciduous species, hot water solubility is 37.08 % lower in coniferous species. Similar studies were found in the literature (Kılıç *et al*., 2010; Hafızoğlu and Usta, 2005). Cold water solubility was found to be 32.11 % higher in TW of deciduous tree species compared to CW of coniferous species. Also, cold water solubility was found to be 24.67 % higher for deciduous trees than for coniferous species. It was observed that the cold water solubility of TW was 4.54 % higher than that of CW. Tutuş *et al.* (2010) determined the cold water solubility as 3.42 % in their research on pine wood.

1 % NaOH solubility of fir CW and pine CW was found to be 13.15 % and 15.73 %, respectively; it was determined as 9.37 % for fir OW and 12.40 % for pine. 1 % NaOH solubility of RW of deciduous species was found to be greater at about 78.27 % than RW of coniferous species, and TW was also higher at a rate of 61.14 % compared to CW. Similar studies were found in the literature (Benouadah *et al.,* 2018).

3.2 Anatomical properties
3.2. Anatomska svojstva

Earlywood vessel and tracheid diameters on transverse sections were greater than latewood cross sections in all studied species. In Figure 4, the tangential diameter of the earlywood tracheid in OW of coniferous species was found to be larger (~1.4 µm) than that of CW, while the latewood tracheid was found to be larger (~2.94 µm) in CW.

The tangential diameter of the latewood vessel cells in beech TW was found to be 12.44 % and was narrower than that of OW. It has been stated in the literature that the beech has a circular vessel structure,

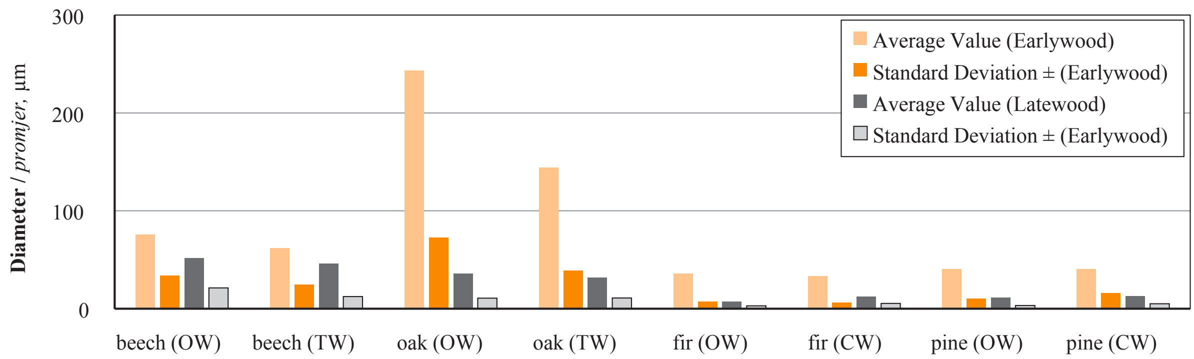

Figure 4 Average tangential diameters of vessel or tracheid (TW – tensile wood, CW – compression wood, OW – opposite wood)
Slika 4. Prosječni tangentni promjeri traheja ili traheida (TW – tenzijsko drvo, CW – kompresijsko drvo, OW – opozitno drvo)

the latewood vessels are smaller in diameter and few in number, and the rays are in the form of longitudinal lines in the transverse section (Safdari *et al.*, 2008).

While the tangential direction of tracheid diameters was found to be 1.69 % wider in the average latewood dimeters of pine CW in cross-section, it was found to be 54.24 % wider in the earlywood than that of OW (Figure 4). The earlywood and latewood widths of fir and pine CW were found to be wider (Fir: ~63.5 % earlywood, 55 % latewood. Pine: 28.3 % earlywood, 34.8 % latewood) than those of OW. The earlywood widths of oak (~60.8 %) and beech (~15.8 %) TW were found to be less than those of OW, and the latewood (~2.7 % beech, ~22.2 % oak) widths were higher than those of TW. Earlywood tracheids had a larger lumen and a thin wall structure (Esteban and Palacıos, 2009). For pine wood, tracheid diameters were averagely narrower in CW (about 0.22 %) than in OW, and they were also 8.59 % wider in latewood. CW transition from earlywood to latewood is gradually compared to OW.

The average number of rays per mm^2 of pine CW was found to be 4.58, and the average number of rays per mm^2 of OW was found to be 4.00. In Figure 5, it was observed that the average number of rays per mm^2 of pine CW in the tangential section was 14.50 % larger than that of OW. The average number of rays 1 mm^2 in fir CW was 6.24 and 5.74 in OW. This rate was found to be 8.70 % for fir in the same direction. On average, about two times more ray numbers were found on 1mm^2 in fir wood. The average number of rays in 1 mm^2 was 9.60 and 7.40 in the beech TW and OW, respectively.

The average number of rays in 1 mm^2 of oak TW was 10.20, and the average number of rays in 1 mm^2 of oak OW was 9.63. For oak and beech TW, 5.91 % and 29.72 % more ray numbers were measured, respectively, in 1mm^2 than for OW. The average ray number was determined to be 16.0 % higher in oak wood than in beech. The average number of rays per 1mm^2 in deciduous species was 78.98 % higher than in coniferous species. Similarly, the average number of rays in 1mm^2 of TW was 82.99 % higher than that of CW. Compared to pine and fir woods, the average number of rays in 1mm^2 was higher in beech and oak. Similar results were obtained for *Quercus aucheri* Jaub. and Spach (11.17), *Quercus cocifera* L. (16.23), and *Quercus ilex* L. (7.03) (Kadem and Fakir, 2017).

3.3 Annual ring widths
3.3. Širina godova

Figure 6 shows that annual ring widths are almost 100 % higher in pine OW than fir OW. The earlywood zone for pine CW was 1.20 % greater than that of fir

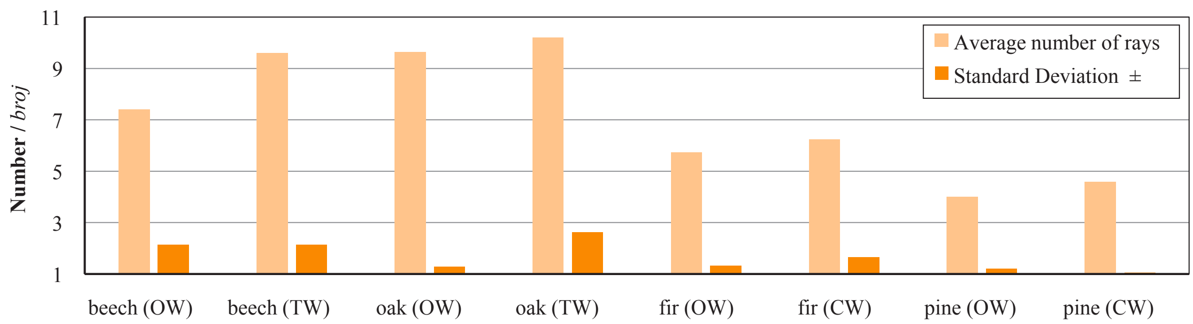

Figure 5 Number of ray cells in 1 mm^2 of tangential section of wood species (TW – tensile wood, CW – compression wood, OW – opposite wood)
Slika 5. Broj stanica trakova na 1 mm^2 tangentnog presjeka različitih vrsta drva (TW – tenzijsko drvo, CW – kompresijsko drvo, OW – opozitno drvo)

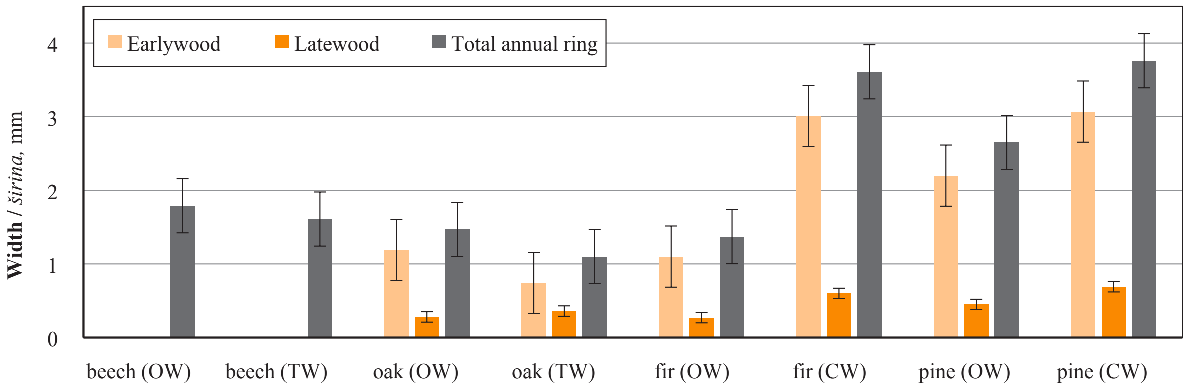

Figure 6 Earlywood and latewood widths of studied wood species (TW – tensile wood, CW – compression wood, OW – opposite wood)
Slika 6. Širine ranoga i kasnog drva ispitivanih vrsta (TW – tenzijsko drvo, CW – kompresijsko drvo, OW – opozitno drvo)

CW. The latewood portion of pine OW was wider (66.67 %) than that of fir OW. Also, the late wood of pine CW was 15 % wider than that of fir TW.

The total annual ring width found for beech OW was 21.77 % wider than that of oak OW, although the annual ring widths of pine OW were on average 93.43 % greater than those of fir OW. However, the annual rings for the beech TW are on average 46.36 % broader than those of oak TW. This ratio for pine TW was 4.15 % higher than for fir TW. Bektaş *et al.,* (2016) have found an annual ring width of 2.11mm in oak. The average width of oak annual rings was between 1.09 and 2.94 mm (Matisons and Brümelis, 2012). Another study stated that climate significantly impacts annual ring width (Pourtahmasi *et al.,* 2011; Roibu *et al.,* 2020; Yaman *et al.*, 2020). In the literature, it has been reported that the anatomical characteristics of oak are affected by environmental conditions (Bozkurt and Erdin, 1995; Gricar *et al.*, 2013).

3.4 Morphological properties of fibers
3.4. Morfološka svojstva vlakana

Morphological measurements of the studied wood fibers are presented in Figures 7 and 8. Although the fiber length of pine OW was 10.61 % longer than that of fir OW, pine CW had 14.04 % longer fiber length than fir CW. Also, the average fiber length of oak OW is 1.79 % longer than that of beech OW, and this rate for oak TW is 9.35 % higher than for beech TW. Figure 8 shows that both fiber and lumen widths are decreasing in CW (~19.75 %) and increasing for TW samples (~7.13 %). However, the average fiber width of beech OW was 24.83 % greater than that of oak OW.

It was determined that the tracheid width of pine OW was 18.11 % wider than that of fir OW. Oak TW fibers were 16.71 % smaller than those of beech TW. Tracheid widths of fir CW were on average 27.79 % narrower than those of pine CW. It was also determined that the lumen width of fir OW cells was 43.15 % less than that of pine OW, and oak OW had 58.53 % less fiber cell lumen than beech OW. While this rate for oak TW was 50.83 % smaller than that of beech TW, fir CW cell lumen widths were 41.95 % less than those of pine CW.

Comparing the cell wall thickness, beech OW was 8.48 % thicker than oak. This rate was 2.95 % higher for pine OW than for fir OW. Also, 0.15 % thicker beech TW was observed than oak TW. This rate for pine CW was 15.70 % higher than for fir CW. Although the difference is not significant, all RW samples have a thicker cell wall than the OW zone. The growth location characteristics and the growth environment af-

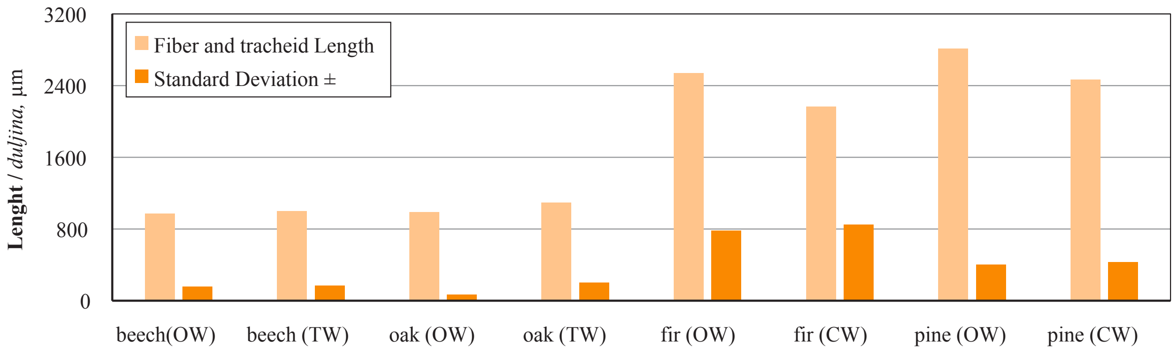

Figure 7 Fiber length of the studied wood samples (TW – tensile wood, CW – compression wood, OW – opposite wood)
Slika 7. Duljina vlakana ispitivanih uzoraka drva (TW – tenzijsko drvo, CW – kompresijsko drvo, OW – opozitno drvo)

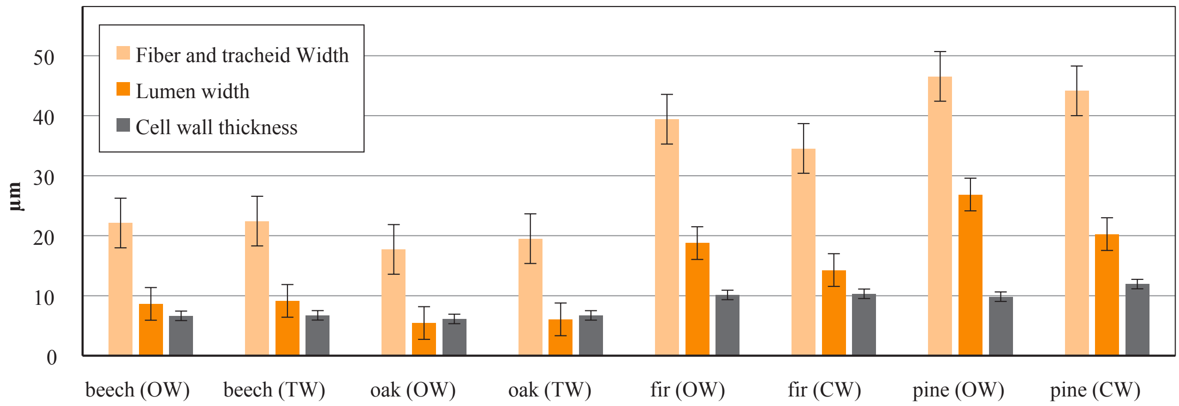

Figure 8 Fiber width, lumen width and cell wall thicknesses (TW – tensile wood, CW – compression wood, OW – opposite wood)
Slika 8. Širina vlakana, širina lumena i debljina stanične stijenke (TW – tenzijsko drvo, CW – kompresijsko drvo, OW – opozitno drvo)

fect the characteristics of the same type of tree (Abuamoud *et al.*, 2018).

3.5 Optical properties
3.5. Optička svojstva

Wood samples were oven-dried. As shown in Figure 9, the color changes (Δ*E**) of green wood and dried wood were 36.86 % higher in coniferous species compared to deciduous species. The color change in CW was higher (42.23 %) than that of TW. The total color change in fir CW was 15.09 % higher than in fir OW, and 17.01 % higher in pine CW than that of OW. It was determined that the total color change of oak and beech TW was higher than that of OW, 2.66 % and 0.72 %, respectively. As a result, it was assumed that the color differences between the different wood species may be due to the different chemical content, especially the extractive contents.

The average color change in fir and pine species was 8.87 % higher than in oak and beech species. The red color parameter (*a**) was 1.92 % lower in fir and pine CW than in OW; it was observed that the TW of oak and beech was 0.09 % higher than that of OW. While the yellow color parameter (*b**) was 1.42 % lower in fir and pine CW than in OW, it was also 1.04 % lower in oak and beech TW than in OW. The brightness parameter (*L**) was 2.65 % higher in fir and pine CW than in OW; it was observed that the TW of oak and beech was 0.35 % lower than OW.

In similar studies, it has been stated that the color of the wood is generally dark, and that the temperature is the main factor. Also, RW caused an increase in the color change in the wood. Due to the high content of extractive substances and lignin in coniferous species, the color difference in wood is found to be high (Aydemir and Gündüz, 2009; Tarmian *et al.,* 2011; Yazıcı and Özlüsoylu, 2020).

4 CONCLUSIONS
4. ZAKLJUČAK

This study investigated the anatomical and chemical structures, fiber, and optical properties of the RW and

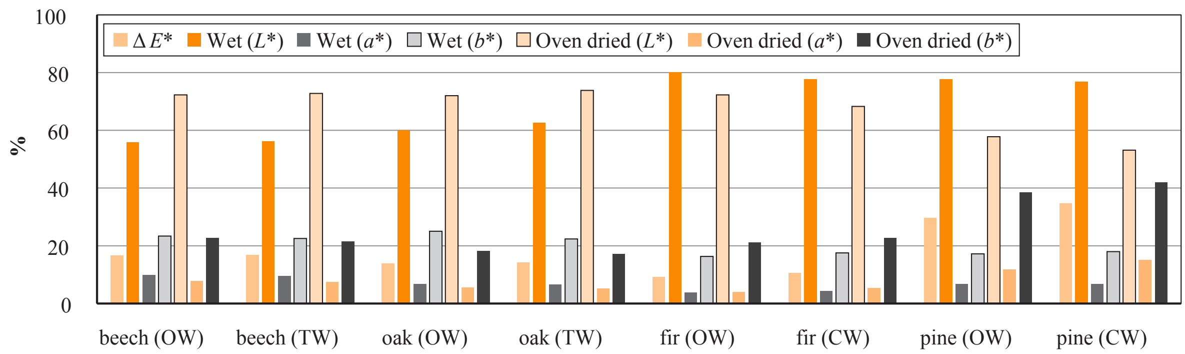

Figure 9 Optical properties of reaction and opposite woods of fir, pine, beech and oak species (Δ*E** – total color change value, *L** – brightness value, *a** – red color value, *b** – yellow color value, TW – tensile wood, CW – compression wood, OW – opposite wood)
Slika 9. Optička svojstva reakcijskoga i opozitnog drva jelovine, borovine, bukovine i hrastovine (Δ*E** – ukupna promjena boje, *L** – svjetlina, *a** – crvenozeleni ton, *b** – žutoplavi ton, TW – tenzijsko drvo, CW – kompresijsko drvo, OW – opozitno drvo)

OW parts of the beech, oak, pine, and fir wood species naturally grown in a specific environment over time.

The amount of ash was found to be higher in deciduous species compared to other wood species. Besides, it was determined to be higher in TW and CW of coniferous species than in OW.

Although the amount of α-cellulose was higher in TW and lower in CW part than in OW, the lignin amount was higher in CW and lower in TW.

Compared to OW, although fiber and lumen widths are decreasing in CW and increasing for TW samples, the color change ratio is higher for CW than for TW.

TW contains more cellulose and less lignin than normal wood in the production of paper and cellulose. However, the strength of the fibers was lower in samples containing high TW. Since CW and TW are more sensitive than normal wood, they cause problems in applications such as structural elements, carriers, surface treatments, cutting, and sawing.

This study determines the anatomical, chemical, fiber, and optical properties of the four wood species commonly used in industry and gives their comparison with each other. The data obtained will likely provide important benefits in practice. In addition, in scientific terms, such comparison studies fill an important gap in the literature in reaching clear results.

During the growing period of trees, necessary environmental, sylvicultural, and genetic interventions should be taken to prevent the formation of reaction wood and to provide more efficient use of wood in the forest product industry.

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Thermal Modification Intensity of Heat-treated Poplar Wood Part 1: Characterization and Predication of Surface Layer

Intenzitet toplinske modifikacije topolovine. Dio 1: Karakterizacija i predikcija površinskog sloja

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ABSTRACT • *Wood heat treatment is an environmentally friendly method, and the heat-treated wood properties are closely related to thermal modification intensity. This study focuses on the 0-3 mm surface layer (SL) of poplar wood heat treated at 160~220 °C. The modification intensity, including surface color, hardness, chemical component and morphological changes of the SL, was evaluated. The findings of this research showed that the color difference of the poplar wood before and after heat treatment (ΔE^*_1) increased; the color difference between up-surface and down-surface of the SL (ΔE^*_2) also increased with the treatment temperature. Consequently, the surface hardness (H_R) decreased with the increase of treatment intensity. When the treatment temperature was higher than 160 °C, the up-surface and down-surface of the SL were statistically different in color. Chemical component analysis revealed that the heat treatment degrades wood components, especially the hemicellulose, and correlation analysis showed a significant correlation between the change rate of hemicellulose and the ΔE^*_1 or H_R value; the prediction functions have been established at a high confidence level of 0.99. Overall, the thermal modification intensity of the heat-treated surface layer (SL) of poplar wood varies, and the H_R and ΔE^*_1 value could be used to characterize and predict the modification intensity and degree of thermal degradation of the surface layer of heat-treated poplar wood.*

KEYWORDS: *heat treatment; modification intensity; hardness; color; poplar wood*

SAŽETAK • *Toplinska obrada drva ekološki je prihvatljiva metoda, a svojstva toplinski modificiranog drva usko su povezana s intenzitetom toplinske modifikacije. Ovo je istraživanje usredotočeno na površinski sloj (SL) od*

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0 – 3 mm toplinski modificirane topolovine pri 160 – 220 °C. Istražen je utjecaj intenziteta modifikacije na boju površine, tvrdoću, kemijski sastav i morfološke promjene površinskog sloja. Rezultati su pokazali da se s povećanjem temperature modifikacije povećala razlika u boji topolovine prije i nakon modifikacije (ΔE^*_1) te razlika u boji između gornje i donje površine površinskog sloja (ΔE^*_2). Posljedično se tvrdoća površine (H_R) smanjila s povećanjem intenziteta modifikacije. Pri temperaturi modifikacije višoj od 160 °C razlika u boji između gornje i donje površine površinskog sloja statistički je značajna. Analiza kemijskog sastava otkrila je da toplinska modifikacija razgrađuje komponente drva, posebice hemicelulozu, a korelacijskom je analizom utvrđena značajna međusobna ovisnost između stupnja promjene hemiceluloze i vrijednosti ΔE^*_1 i H_R. Utvrđene su funkcije predviđanja s visokom razinom pouzdanosti od 0,99. Sve u svemu, intenzitet toplinske modifikacije topolovine u površinskom sloju varira, a vrijednosti H_R i ΔE^*_1 mogu se iskoristiti za karakterizaciju i predviđanje intenziteta modifikacije i stupnja toplinske razgradnje površinskog sloja toplinski modificirane topolovine.

KLJUČNE RIJEČI: *toplinska obrada; intenzitet modifikacije; tvrdoća; boja; topolovina*

1 INTRODUCTION
1. UVOD

Lignocellulosic materials are thought to be one of the most promising alternative resources for fossil fuels. Worldwide attention has been focused on environment-friendly material, and biomass-based functional materials have been applied in various fields (Li *et al*., 2018; Wu *et al*., 2021). Wood is renewable and degradable with a high strength-to-weight ratio, and it could also provide a comfortable indoor environment because of its decorative color and humidity-control properties (Zhang *et al*., 2016).

As one of the main artificial trees in China, poplar wood could provide a large amount of timber for uses such as paper making and wood-based panels (Yang *et al*., 2020). However, poplar wood also has many shortcomings, such as low density, dimensional stability, and durability (Korkut *et al*., 2008), which prevents its use as solid wood material. Many modification methods like acetylation (Mantanis, 2017), resin impregnation (Xu *et al*., 2021), densification (Srivaro *et al*., 2021) and natural, eco-friendly chemicals (Primaningtyas *et al*., 2021), and heat treatment (Chu *et al*., 2020) were applied to provide a better performance and economic benefit to poplar wood and broaden its application field (Dong *et al*., 2020). Heat treatment deepens the color of wood (Liu *et al*., 2021) and reduces wood moisture absorption, enhances dimensional stability (Gu *et al*., 2019), and improves biological durability (Gérardin, 2016). Therefore, demands for heat-treated wood have increased rapidly worldwide (Hill *et al*., 2021), and its application has grown dramatically in various fields, including furniture, architectural decoration, etc. (Gurleyen *et al*., 2017).

Studies show that heat treatment influences the chemical properties of wood through changes in the chemical content and structure of chemical components such as extractives, hemicellulose, cellulose, and lignin (Miklečić *et al*., 2016). The mechanical properties of treated wood are affected by the reaction medium, which mainly includes steam, inert gas (nitrogen), air, hot oil, or water (Rahimi *et al*., 2019). High-temperature heat treatment of wood usually reduces its mechanical properties (Wu *et al*., 2019; Widmann *et al*., 2012), and increases overall brittleness (Hughes *et al*., 2015; Yildiz *et al*., 2006). Guo *et al*. (2016) used the stress-strain curve to divide the elastic and plastic regions and found that the brittleness of wood increased significantly with the increase in thermal modification intensity. Schneid *et al*. (2014) used the same method to calculate the brittleness of the heat-treated wooden chair; the result showed that the brittle was highly correlated with thermal mass loss. Fu *et al*. (2020) found that the change of chromatic value can accurately predict the mechanical properties of heat-treated wood, and that the surface color was closely related to mechanical properties (Nasir *et al*., 2019). Lengowski *et al*. (2021) found that the hardness of the heat-treated teak wood decreased due to the degradation of amorphous polysaccharides during the heat treatment process, and that the hardness was related to thermal mass loss.

In practice, the processing performance of heat-treated wood is closely associated with the properties of the surface layer of a certain thickness, and the chemical or physical characteristics of wood surfaces affect the gluing and coating processes (Gu *et al*., 2019; Hill *et al*., 2021). Thermal modification intensity of the surface layer is an influential factor in determining the processing performance and quality of heat-treated wood, according to the theory of mechanical weak boundary layer (MWB) (Stehr *et al*., 2020; Chu *et al*., 2020). Wang (2011) studied the thermal effect of vacuum heat treatment on *eucalyptus grandis* and found that the sample dimension had a significant impact on unit mass heat absorption, leading to uneven heat transfer in the direction of thickness. Ramdane *et al*. (2015) found a significant difference in temperature with the increase of sample thickness based on transient analysis of the heat and mass transfer process. The thermal degradation intensity of wood surface layer is higher than that of the inner part of wood, because of the low thermal conductivity of wood and the heat and mass diffusion, and the moisture or contacting medium difference.

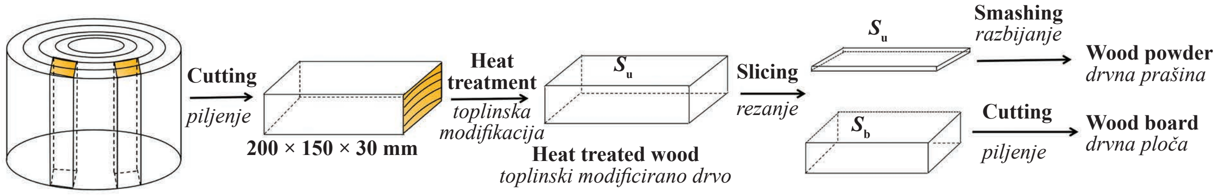

Figure 1 Flow chart of sample preparation
Slika 1. Dijagram toka pripreme uzorka

In our previous studies, it has been reported that the heat treatment process declined the surface abrasion and Shore D hardness, as well as bonding capacity (Chu *et al*., 2020), and that an enhanced surface layer could be generated by an impregnated chemical agent and thermal compression (Chu *et al*., 2019). However, there are few studies on the degree of thermal degradation of the heat-treated wood at 0-3 mm surface layer to the best of the authors knowledge, although there are several studies involving the overall properties of the wood (Schneid *et al*., 2014; Costa *et al*., 2020). This study aims to study the influence of temperature and duration on thermal modification intensity of heat-treated poplar wood at 0-3 mm surface layer and establish a potential fitting model for revealing the variation of degradation degree of the heat-treated wood regarding the surface layer. The results would provide the basis for evaluating the processability of the heat-treated poplar wood.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Sample preparation
2.1. Priprema uzoraka

The 12-year-old Zhonglin 46 poplars (*Populus euramericana 'zhonglin 46'*) were selected, and the straight part of the air-dried lumber was cut into 200 mm × 150 mm × 30 mm sapwood boards. A self-made high-temperature experiment device was used to conduct high-temperature heat treatment on the poplar wood samples, and a cistern was installed as a steam generating device.

The poplar wood samples were put evenly in the experiment box, the temperature of the inside of the box was raised from room temperature to 130 °C at a heating rate of 20 °C·min^{-1}, kept for 30 minutes, and then the box was set at a heating rate of 10 °C·min^{-1}. The internal temperature was raised to the target treatment temperature (160 °C, 190 °C, 220 °C), for 2h and 4h. The experimental group numbers were H_{160-2}, H_{160-4}, H_{190-2}, H_{190-4}, H_{220-2}, H_{220-4}, the control group was the untreated wood. Each group had ten to fifteen duplicate specimens.

The surface layer of the heat-treated poplar wood with a thickness of 3 mm was named *SL*, and its upper-surface and bottom-surface were marked as S_u and S_b, respectively. Then, the *SL* part of the heat-treated wood was smashed into wood powder for further use.

2.2 Measurement of color and hardness of heat-treated poplar
2.2. Mjerenje boje i tvrdoće toplinski modificirane topolovine

According to the CIE1976LAB standard colorimetric system, a precision colorimeter (HP-200, Shenzhen Hampoo) was used to obtain the surface color parameters. There were 10 replicates for each experimental group, and 5 points were randomly selected on the upper-surface (S_u) and bottom-surface (S_b) of the heat-treated wood for color measurement. Color difference value ΔE^* was calculated as follows:

$$\Delta L_1^*=L_h-L \quad (1)$$

$$\Delta a_1^*=a_h-a \quad (2)$$

$$\Delta b_1^*=b_h-b \quad (3)$$

$$\Delta E_1^*=[(\Delta L_1^*)^2+(\Delta a_1^*)^2+(\Delta b_1^*)^2]^{1/2} \quad (4)$$

$$\Delta E_2^*=[(\Delta L_2^*)^2+(\Delta a_2^*)^2+(\Delta b_2^*)^2]^{1/2} \quad (5)$$

Where, L_h, a_h, b_h are the chromatic parameters of the poplar wood after HT, and L, a, b are the parameters of wood before the heat treatment; ΔL_2^*, Δa_2^*, Δb_2^* are the chromatic parameters difference of S_u and S_b on the heat-treated wood, ΔE_2^* is the color difference between S_u and S_b of the heat-treated wood on the surface layer.

Pressure ball hardness test uses a high-precision double-column universal testing machine (AG-X plus, Shimadzu, Japan), using a hemispherical steel indenter mold, in accordance with the national standard GB/T1941-2009 Wood hardness test method. The hemispherical steel indenter was pressed into the specimen surface of 2 mm at a uniform speed of 1 mm/min, and the pressing load at the pressed depth of 0.5 mm, 1.0 mm, 1.5 mm, and 2.0 mm was recorded and marked as *P*. Each experimental group was repeated 15 times. The surface hardness (H_R) was calculated based on the indentation load of the untreated poplar wood (Yu *et al*., 2020).

$$H_R=P_1/P_0 \quad (6)$$

P_1 – pressing load on 1 mm surface of heat-treated wood, unit: N;

P_0 – pressing load on 1 mm surface of untreated wood, unit: N.

2.3 Chemical component and morphological analysis
2.3. Kemijske komponente i morfološka analiza

Triplicates were used to perform quantitative chemical analysis of control and modified wood samples. According to the Chinese standards of wet chemical analysis of wood components shown in Table 1, the hemicellulose content was estimated by the difference between holocellulose and a-cellulose. Each group had three replicates, and the average value was calculated.

FT-IR analysis was performed on the samples (potassium bromide compressed tablets) by Fourier infrared spectrometer. Samples were scanned in the range of 390-4000 cm^{-1} with a 4 cm^{-1} resolution, and each sample was analyzed by 32 scans.

The experimental equipment used an XRD-6 X-ray diffractometer. The X-ray tube was a copper target and the wavelength λ=1.5406 nm. The test temperature was room temperature, the tube voltage was 40 kV, and the tube current was 30 mA. Surface sample wood powder was dried and pressed into thin sheets at room temperature. Then a 2θ diffraction intensity curve was drawn. The scanning angle 2θ ranged from 5° to 35°, the scanning rate was 2 °/min, the step width was 0.01, and the diffraction intensity curve was recorded. It was output by the plotter immediately, and each sample was sampled twice, and the average value was taken.

The surface of untreated wood and heat-treated wood were selected, a *SL*ide *SL*icer (SM2010R, Leica, Germany) was used to cut the surface of heat treated poplar. Scanning electron microscopy (SEM) scans of the tangential section of the wood chips were obtained using a scanning electron microscope FEG-XL30 (FEI, USA). Samples were covered by galvanic gold deposition using an MC1000 ion sputter (FEI, USA) with a current of 5 mA for 45 s. The analyses were performed with an acceleration voltage of 20 kV.

Table 1 Testing standard reference of heat-treated poplar wood
Tablica 1. Standardi za ispitivanje toplinski modificirane topolovine

Measured items *Mjerene sastavnice*	**Testing standard reference** *Ispitni standardi*
Moisture content *sadržaj vode*	GB/T 36,055–2018
Benzene-alcohol extractives *benzen-alkoholni ekstrakti*	GB/T 35,816–2018
Holocellulose *holoceluloza*	GB/T 35,818–2018
Cellulose / *celuloza*	GB/T 744–2004
Lignin / *lignin*	GB/T 35,818–2018

2.4 Data analysis and modeling of degradation intensity at surface layer
2.4. Analiza podataka i modeliranje intenziteta razgradnje površinskog sloja

The correlations between the chemical component content, ΔE_1^*, hardness, and crystallization values of untreated and heat-treated samples were calculated, and the correlation matrix was obtained using Origin software. Then, the prediction models of the wood degradation intensity at surface layer were established based on the hemicellulose change rate and the color changes before and after the heat treatment.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Color and hardness of heat-treated wood on the surface layer
3.1. Boja i tvrdoća površinskog sloja toplinski modificiranog drva

Surface color and hardness are prominent properties of furniture, flooring, and other wooden materials. The heat treatment usually darkens the surface and decreases the hardness of wood, which could reflect the degradation intensity and brittleness degree of the heat-treated wood. Table 2 summarizes the chromatic values and hardness of the surface layers (*SL*) of heat-treated poplar.

As presented in Table 2 and Figure 2, the lightness value L^* of the *SL* of heat-treated poplar is significantly lower than that of untreated poplar. The red-green index a^* and yellow-blue index b^* are much higher. Among the chromatic parameters, the change in L^* value is the most obvious; it decreases with the increase of heat treatment temperature. The L^* of H_{160-2} and H_{220-2} are 84.69 and 34.58, respectively, which is 3.39 % and 60.55 % lower than that of untreated poplar. The a^* and b^* values first increased and then decreased with the increase of the heat treatment temperature; both a^* and b^* reached their maximum at the temperature of 190 °C. Heat treatment duration has a minor effect on the changing of wood surface color; the L^* value of H_{160-4} was 3.64 % lower than that of H_{160-2}. There was no notable difference in the chromatic parameters between H_{220-2} and H_{220-4}. Numerous studies have shown that the surface color of heat-treated wood becomes darker and tends to be red and yellow (Mantanis, 2017), and that the change of surface color is closely related to the heat treatment temperature (Chen *et al.,* 2018; Cao *et al.,* 2018). The ΔE_1^* of H_{220-2} is 10.97 times higher than that of H_{160-2}.

The surface color of S_u and S_b of the *SL* in the same condition of heat-treated poplar is different, and the ΔE_2^* value ranges from 0.89 to 3.42, according to Table 2. The increase of ΔE_2^* value indicated that the

Table 2 Chromatic values and pressing load of surface layer of heat-treated poplar wood
Tablica 2. Kromatske vrijednosti i tlačno ispitivanje površinskog sloja toplinski modificirane topolovine

Groups *Skupine*		**Color change** *Promjena boje*			**Pressing load at different depth, N** *Tlačno opterećenje pri različitoj dubini,* N			
		ΔL^*	ΔE_1^*	ΔE_2^*	0.5 mm	1.0 mm	1.5 mm	2.0 mm
Control		-	-	-	77.52±5.76	166.80±4.85	254.68±11.60	368.81±13.88
H_{160-2}	S_u	2.97	4.46	0.89	52.16±6.57	110.48±10.72	171.69±15.04	234.86±17.37
	S_b	2.32	4.34					
H_{190-2}	S_u	24.37	27.2	2.73	32.27±5.16	83.24±5.32	158.66±9.16	240.66±17.85
	S_b	21.86**	24.52**					
H_{220-2}	S_u	53.08	53.46	3.42	18.97±3.06	58.59±12.57	128.00±18.20	202.03±17.88
	S_b	50.24**	50.87**					
H_{160-4}	S_u	6.05	8.58	2.33	68.53±6.52	155.62±5.67	245.93±9.68	339.83±18.54
	S_b	5.02*	6.55**					
H_{190-4}	S_u	35.36	37.23	2.83	45.86±9.01	118.27±22.43	220.20±29.91	319.01±28.49
	S_b	33.39*	35.76					
H_{220-4}	S_u	53.13	53.62	2.83	37.47±7.70	84.44±14.79	162.82±23.19	260.83±46.77
	S_b	50.52**	51.15**					

* and ** mean significant differences of 0.05 and 0.01 between the S_u and S_b / * *i* ** *znači da su za* S_u *i* S_b *izmjerene značajno različite vrijednosti pri p<0,05 i p<0,01*

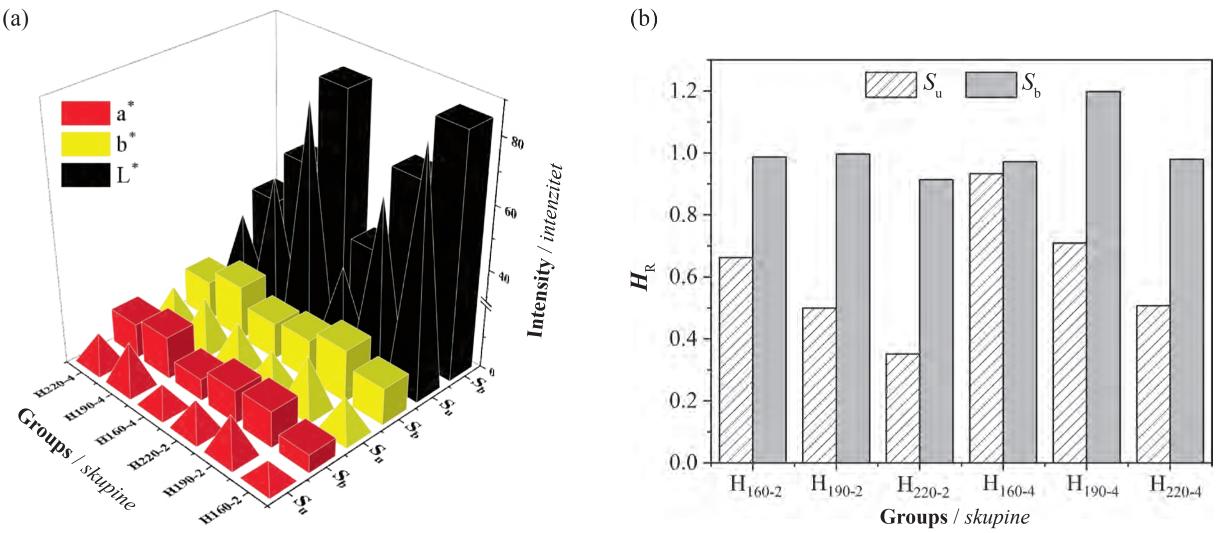

Figure 2 CIELab values (a) and hardness (b) of heat-treated poplar wood at surface layer
Slika 2. CIELab vrijednosti (a) i tvrdoća (b) površinskog sloja toplinski modificirane topolovine

degradation intensity difference between S_b and S_u increased with the increase of heat treatment temperature. Besides, the L^* value of S_u is lower than that of S_u in all heat-treated groups. Variance analysis shows that in all heat-treated groups (except H_{160-2}), the ΔL^* and ΔE_1^* values are significantly different at the 0.05 level, which means the degree of thermal degradation of heat-treated poplar on the *SL* is different. When the heating temperature is higher than 190 °C, the uneven heating on the surface and inside leads to the difference in the degree of thermal degradation. Shi and Bao (2021) found that the extractives migrated from the core layer to the surface during the thermal modification process, which was also one of the reasons for the color difference of the wood surface layer. The possible reason for an inconsiderable difference of H_{160-2} is that the heat treatment is relatively low, and the *SL* is not significantly thermally degraded.

Figure 2 represents the H_R of heat-treated wood at the *SL*, which is the degree of surface plastic deformation to evaluate its ability to resist external force pressing. With the increase of the press-in depth, the ability of the compressed part to resist external force is enhanced due to the densification of the surface material. The H_R of the *SL* is calculated from the load when the press-in depth is 1 mm. After the heat treatment, the H_R of wood was reduced to different degrees, and the H_R value decreased with an increase in the heat treatment temperature in the duration of 2 hours. The H_R values of H_{160-2}, H_{190-2}, and H_{220-2} decreased by 34 %, 50 %, and 65 %, respectively, compared with that of untreated wood. Previous studies stated that the hardness of wood is sensi-

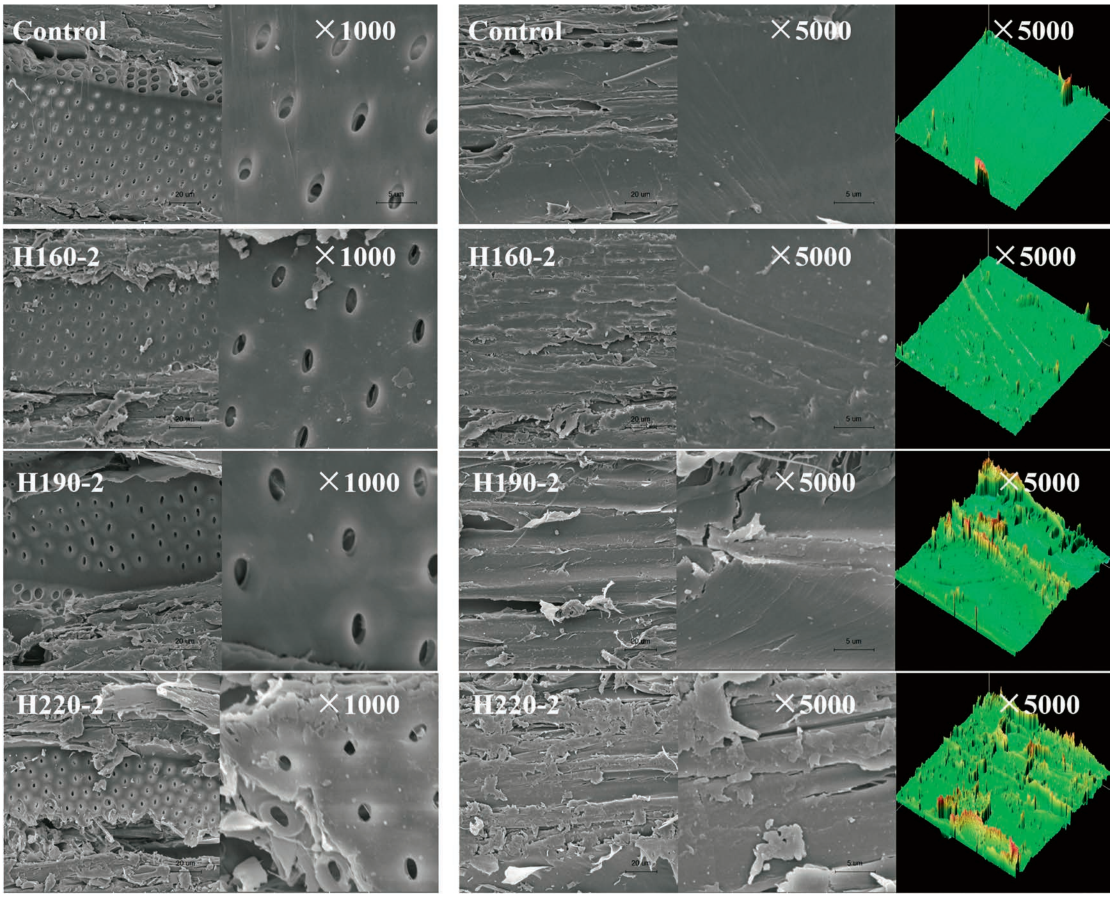

Figure 3 SEM images of heat-treated poplar surface and roughness of cell wall
Slika 3. SEM slike površine toplinski modificirane topolovine i hrapavosti stanične stijenke

tive to the change in hemicellulose content (Konnerth *et al.,* 2010; Borůvka *et al.,* 2018). Hemicellulose acts as a cell wall-filling component and its degree of degradation increases with the increase of the degree of heat treatment. The H_R value of the *SL* decreased with the increase of heat treatment temperature when the duration was 4 hours, as can be seen in Figure 2. However, the H_R value of the *SL* on H_{160-4}, H_{190-4}, and H_{220-4} was higher than that of H_{160-2}, H_{190-2}, and H_{220-2}, respectively. It could be explained by the comprehensive effects of the changing on crystallinity and the cross-linked lignin network during the long duration of the heat treatment process. When poplar wood was treated at 160 °C, the degradation of wood cell wall components was relatively low, and the crystallinity of wood increased with the HT duration, which caused the increase in H_R. When the heating temperature is higher, the crystallinity of wood decreases with the heat treatment duration, and the relative content of lignin increases with the degradation of hemicellulose and cellulose. In addition, the degradation products of hemicellulose and lignin also form cross-linked lignin networks, which leads to an increase in H_R (Altgen *et al.,* 2018). Xi (2018) found that crystallinity and lignin contents also have a great influence on wood hardness.

Existing research also showed that the destruction of the cell wall structure reduces the hardness of the heat-treated wood (Aytekin *et al.,* 2021; Chu *et al.,* 2020). The surface cell wall and the roughness of the heat-treated wood were observed by scanning electron microscope as shown in Figure 3. It can be seen that the untreated wood surface was smooth, and the cell wall structure was undamaged. When the poplar wood was treated at 160 °C, the surface and pit structure did not change significantly. The roughness analysis showed that the sample surface was relatively smooth. So, the low-temperature heat treatment did not have a significant indigenous effect on the cell wall structure. With the increase in heat treatment temperature, the surface of H_{190-2} and H_{220-2} was cracked and gradually expanded, and some cell walls were stratified. The pit membrane deforms, and the pits become hollow and rupture, resulting in cracks and shedding. The increase of burrs on the surface made the heat-treated wood rougher, and the cell wall structure was damaged and collapsed. The cell wall structure of H_{220-2} was significantly changed and severely damaged mainly due to further degradation of hemicellulose and partial pyrolysis of lignin at the high temperature of 220 °C (Budakçı *et al.,* 2016). Wang (2017) found that the changes in the pit structure were due to a pressure gradient generated by the degradation of cell wall components, which resulted in the increase of internal pressure of wood.

3.2 Chemical component changes of surface layer of heat-treated poplar wood
3.2. Promjene kemijskih komponenata u površinskom sloju toplinski modificirane topolovine

The change of the chemical composition is the basic reason for the change of properties of the heat-treated wood. Table 3 documents the results of the wet chemical analysis of the surface layer (*SL*) of heat-treated poplar wood.

Among all wood components, hemicellulose is the most severely degraded by thermal treatment, and it is the main reactive substance during the heat treatment process; this result is consistent with the study of Dubey *et al*. (2012). Hemicellulose showed a downward trend with the increase of heat treatment temperature and duration; the hemicellulose contents of H_{160-4}, H_{190-4}, and H_{220-4} were 31.42 %, 21.75 %, and 15.12 %, respectively. On the contrary, the extractives content increases with the increase of heat treatment temperature and duration. Especially, the content of the extractives of H_{220-4} is 10.13%, which is 3.26 times higher than that of untreated wood. Hemicellulose degrades to produce small molecules (Candelier *et al*., 2020), which probably increases the extractives content. The content of cellulose and lignin are relatively stable, but when the heat treatment temperature is higher than 190 °C, cellulose also degrades to a certain extent (Chien *et al*., 2018), and lignin branched-chain breaks (Yang *et al*., 2021); the relative content of these two components increases because of the decrease of hemicellulose.

The FT-IR spectra of the chemical functional group changes of poplar wood before and after heat treatment are shown in Figure 4. The experiment mainly analyzes the change of characteristic peaks in the range of 1800 cm^{-1}-750 cm^{-1}. There is a subtle change of characteristic peak at 1424 cm^{-1} after the HT process, which is assigned to the shear vibration peak of cellulose. Therefore, this peak could be chosen to normalize the spectrum (Lei *et al*., 2021). Table 4 represents the ratio of each peak.

As shown in Figure 4(a), the absorbance at 1735 cm^{-1} is assigned to the hemicellulose acetyl non-conjugated carbonyl group (Wentzel *et al*., 2019); its peak is reached relatively smoothly with the increase of heat treatment temperature mainly due to the degradation of hemicellulose. The higher the heat treatment temperature, the more severe the degradation of the hemicellulose. The absorption peak at 1163 cm^{-1} is attributed to the C-O-C stretching vibration of cellulose and hemicellulose. The increase in the intensity of this peak is mainly due to the formation of ether bonds by the hydroxyl groups on hemicellulose and cellulose during the heat treatment. However, the peak at 1163 cm^{-1} was reduced for H_{220-2} and H_{220-4}, indicating that the ether bond broke at a high heat treatment temperature of 220 °C. The change of absorption peak near 1058 cm^{-1}, which is attributed to the C-O shrinkage vibration of cellulose and hemicellulose, indicated that aldols products are formed

Table 3 Chemical components and XRD of surface layer of heat-treated poplar wood
Tablica 3. Kemijske komponente i XRD površinskog sloja toplinski modificirane topolovine

Groups *Skupine*	**Chemical composition change, %** *Promjena kemijskog sastava, %*				**Crystallization** *Kristalizacija*		
	Hemicellulose	Cellulose	Lignin	Benzene-alcohol extractives	I_{002} (2θ)	I_{am} (2θ)	CI_{XRD}
Control	34.83	46.63	23.71	2.38	22.22	18.62	42.09
H_{160-2}	32.79	47.22	23.50	2.24	22.20	18.48	44.82
H_{190-2}	25.91	47.69	20.43	4.67	22.26	18.86	51.38
H_{220-2}	17.05	44.19	26.81	8.30	22.24	18.44	56.35
H_{160-4}	31.42	46.35	21.36	2.53	22.16	18.58	45.33
H_{190-4}	21.75	49.60	20.83	5.14	22.26	18.52	51.27
H_{220-4}	15.12	46.86	27.88	10.13	22.46	18.92	53.05

Table 4 Intensity ratio of each peak after normalizaton treatment at 1424 cm^{-1}
Tablica 4. Omjer intenziteta svakog vrha nakon normalizacije na 1424 cm^{-1}

Groups *Skupine*	I1735	I1603	I1512	I1463	I1264	I1163	I1058
Control	0.865	0.801	0.737	0.974	1.840	0.904	2.560
H_{160-2}	0.782	0.824	0.725	0.944	1.620	1.577	2.542
H_{190-2}	0.773	0.864	0.733	0.962	1.553	1.538	2.659
H_{220-2}	0.709	0.847	0.810	0.985	1.518	1.385	2.234
H_{160-4}	0.816	0.810	0.741	0.966	1.425	1.598	2.534
H_{190-4}	0.846	0.787	0.757	0.978	1.375	1.551	2.603
H_{220-4}	0.707	0.900	0.833	1.013	1.360	1.387	2.280

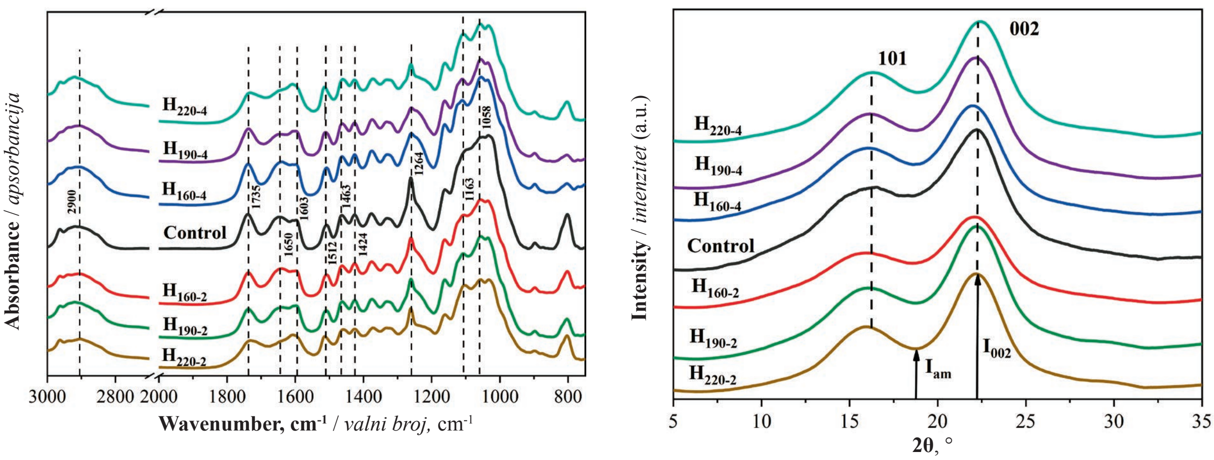

Figure 4 Infrared spectra (a) and X-ray diffraction curves (b) of surface layer of heat-treated poplar
Slika 4. Infracrveni spektar (a) i difrakcijska krivulja rendgenskih zraka (b) površinskog sloja toplinski modificirane topolovine

during the heat treatment process at relatively low temperature, while the aldols undergo condensation reaction at the high heat treatment temperature of 220 °C.

The stretching vibration peak at 1650 cm^{-1} is assigned to the C-O group in lignin, mostly located on the propyl branch of lignin. Besides, absorbance at wave numbers 1603 cm^{-1} and 1264 cm^{-1} is assigned to the conjugate stretching vibration of the C=O and the benzene ring skeleton of lignin and the stretching vibration of the G ring and the acyl-oxygen bond C=O in the lignin (Gao, 2019). With the increase of heat treatment temperature, the peak intensity at 1650 cm^{-1} and 1264 cm^{-1} decreased, while the peak intensity at 1603 cm^{-1} increased, which could also support the conclusion that the relative content of lignin increases. Furthermore, the cross-linking reaction of the lignin occurs at the same time as its thermal degradation. Absorbance at 1512 cm^{-1} and 1463 cm^{-1} is the benzene ring carbon skeleton vibration peak of lignin and the deformation vibration peak of C-H_2 in lignin and carbohydrates. The two peaks increased with the increase of the heat treatment temperature, indicating an increase in relative lignin content (Table 3). The result is consistent with that of wet chemical analysis discussed above.

The X-ray diffraction pattern is shown in Figure 4(b). Within the scanning interval of 0-35°, two prominent peaks appear on the curve. A peak valley at about 2θ=18° represents the scattering intensity I_{am} of the diffraction angle of the amorphous region. The peak located near 2θ=22° indicates the maximum intensity I_{002} of the diffraction angle of the crystal region. The crystallinity value can reflect the degree of crystal formation during the cellulose condensation process, and it is closely related to elastic modulus, impact toughness, and mechanical properties (Sun *et al.,* 2019). Table 4 lists the crystallization characteristics of *SL* of heat-treated poplar calculated according to I_{am} and I_{002}.

After the heat treatment, the positions of the I_{002} diffraction peaks of the samples were all around 22°, and the crystal layer distance did not change. It means that the heat treatment did not significantly change the crystallization characteristics. When the treatment duration was 2h, the crystallinity of heat-treated wood increased from 42.09 to 56.35, with the increase in heat treatment temperature. It is worth noting that the CI_{XRD} of H_{160-4} increased slightly compared with that of H_{160-2}, and there was no evident difference between H_{190-2} and H_{190-4}. Nevertheless, a significant decline was found for H_{220-4} as the duration increased from 2h to 4h. The degradation of hemicellulose and amorphous-cellulose increases with the increase of heat treatment duration at a relatively low temperature of 160 °C, which accordingly increases the relative percentage of cellulose. Akguiil *et al*. (2007) found that the degradation products of hemicellulose could also be transformed into a crystalline area. With the increase of heat treatment temperature, part of the cellulose began to decompose, and the CI_{XRD} of H_{220-4} declined with the extension of heat treatment duration. Additionally, hemicellulose degradation generates acid substances, accelerating the deterioration of cellulose and lignin and decreases the wood crystallinity (Yang *et al.,* 2021).

3.3 Correlation analysis and prediction of degradation intensity at surface layer
3.3. Korelacijska analiza i predviđanje intenziteta razgradnje površinskog sloja

Figure 5 represents the correlation analysis of the degradation characteristics, including the color, hardness, crystallization, and the change rate of chemical components.

As shown in Figure 5(a), the SPSS analysis results show that the color difference ΔE_1^* and change rate of hemicellulose and benzene-alcohol extractives are all significantly correlated at the 0.05 statistical level. The hardness of wood was significantly correlated with the change rate of hemicellulose, while the relative crystallinity of cellulose was significantly negatively correlated with the change rate of hemicellu-

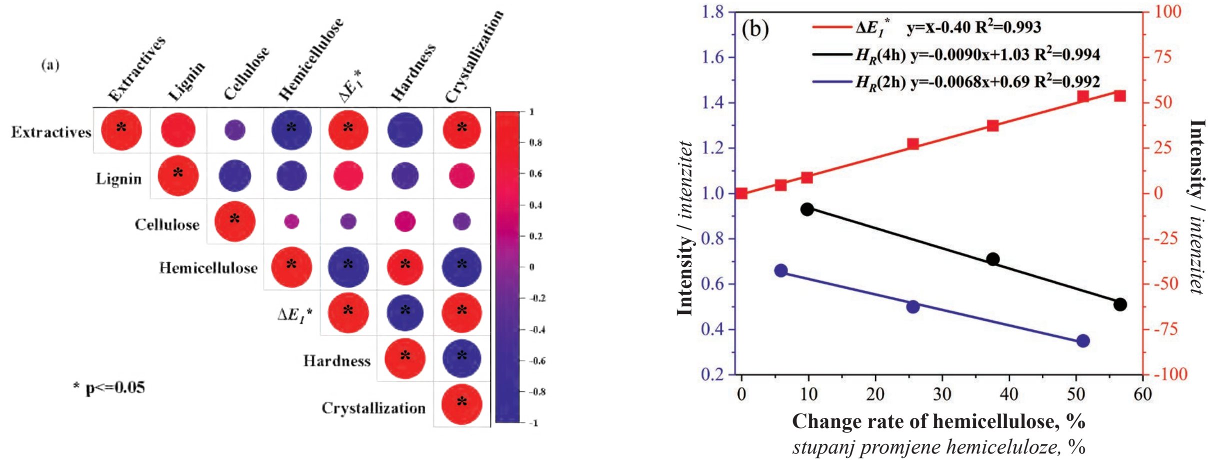

Figure 5 Correlation matrix of chemical component change rate (a) and fitting curve of change rate of hemicellulose with color difference and hardness on surface layer of heat-treated poplar wood (b)
Slika 5. Korelacijska matrica stupnja promjene kemijske komponente (a) i usklađivanje krivulja stupnja promjene hemiceluloze s razlikom u boji i s tvrdoćom površinskog sloja toplinski modificirane topolovine (b)

lose. The lower hemicellulose content, the higher crystallinity of wood; crystallinity also affects the hardness of wood. As shown in Figure 5(b), the H_R of the heat-treated poplar at the surface layer decreases with an increase in hemicellulose change rate; the functional equations of the fitted curve are $y=x-0.40$, (R^2=0.993); $y=-0.0068\cdot x+0.69$, (R^2=0.992).

The color difference ΔE_1^* increases linearly with the changing rate of hemicellulose, and the fitted model is $y=-0.0090\cdot x+1.03$, (R^2=0.994). The degree of fitting is very good, indicating that the hardness and surface color difference of the heat-treated wood surface layer is highly linearly related to the change of hemicellulose content. Therefore, they could characterize the thermal degradation of the heat-treated wood to a certain extent. This result agrees with previous studies, wherein Costa *et al*. (2020) stated that the degradation of polysaccharides could be the reason for the increased modification intensity of the high-temperature treated wood.

4 CONCLUSIONS
4. ZAKLJUČAK

The thermal modification intensity of the 160 °C, 190 °C, and 220 °C heat-treated wood at 0-3 mm surface layer was studied, and wet chemical analysis and correlation analysis were applied to reveal the relationship of surface color and hardness changes with the degree of thermal degradation of the treated wood regarding chemical changes.

The surface layer of the heat-treated wood becomes dark and consequently the surface hardness decreases with the increase of modification intensity, and the colors between the upper-surface and bottom-surface are significantly different. Heat treatment causes a change in the chemical composition of poplar wood, and the thermal mass loss of wood mainly occurs due to hemicellulose degradation, which significantly changes the cell wall structure and could be listed as the main reason for the increase in modification intensity.

The correlation analysis shows that the color difference (ΔE_1^*) and surface hardness (H_R) have a high correlation with the change rate of hemicellulose, and a functional relationship, H_R and ΔE_1^*, could be used as a simple and rapid method to characterize the thermal modification degree of the surface layer of heat-treated wood.

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Influence of Zinc Oxide Nanoparticles on Flame Resistance in Wood Plastic Composites

Utjecaj nanočestica cinkova oksida na vatrootpornost drvno-plastičnih kompozita

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ABSTRACT • *The interest in wood plastic composites (WPCs) has increased in recent years. The utilization of environmentally friendly materials has been of great significance due to the overwhelming pressure on nature. As a widely used material, plastic is, however, easily combustible due to its structure. In this study, WPCs were reinforced with zinc oxide (ZnO) nanoparticles. The effect of higher content of ZnO nanoparticles (1, 3, 5, 10 %) on WPCs thermal stability and fire performance was investigated. Thermogravimetric analysis (TGA) clearly demonstrated that nanoparticles acted as a shield, which inhibited heat transfer and increased the degradation temperature thanks to covering the surface of materials. Free radicals accelerated the thermal degradation of neat-HDPE (high-density polyethylene) by oxidative reactions, while ZnO nanoparticles reduced the degradation velocity. Moreover, the increase in nanoparticle content significantly affected the residue. The fire performance of WPCs was also investigated by the limit oxygen index (LOI) test. While neat-HDPE flamed with dripping, ZnO nanoparticles made flaming difficult for WPCs. Therefore, the LOI values increased with increasing nanoparticle content up to 28.5 %, which indicated the need for more oxygen. The improvement reached up to 54 % compared to neat HDPE. Moreover, the char forming was also improved, which helped enhance the fire resistance. The scanning electron microscope (SEM) investigation indicated that nanoparticles were well dispersed in the matrix. However, the tendency to agglomerate increased with the increase of concentration. The ability of carbonization of wood fiber surface during the combustion also contributed to improving thermal stability and fire performance.*

KEYWORDS: *ZnO nanoparticles; LOI test; TGA analysis; thermal resistance; WPC*

SAŽETAK • *Zanimanje za drvno-plastične kompozite (WPC) posljednjih je godina poraslo. Upotreba ekološki prihvatljivih materijala ima veliko značenje zbog golemog pritiska na okoliš. Plastika je široko rasprostranjen i vrlo često upotrebljavan materijal, ali je zbog svoje strukture lako zapaljiva. U ovom su istraživanju drvno-plastični kompoziti ojačani nanočesticama ZnO. Istraživan je utjecaj većeg udjela nanočestica cinkova oksida (ZnO) (1, 3, 5 i 10 %) na toplinsku stabilnost i vatrootpornost WPC-a. Termogravimetrijska analiza (TGA) jasno je pokazala da nanočestice djeluju kao štit koji inhibira prijenos topline i povećava temperaturu razgradnje. Nadalje, slobodni radikali u reakcijama oksidacije ubrzali su toplinsku razgradnju čistog HDPE-a (polietilena visoke gustoće), dok su nanočestice ZnO smanjile brzinu razgradnje. Štoviše, povećanje udjela nanočestica znatno je utjecalo na*

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ostatak nakon razgradnje. Vatrootpornost WPC-a također je ispitana mjerenjem graničnog indeksa kisika (LOI). Dok je čisti HDPE gorio uz kapanje, nanočestice ZnO otežale su gorenje WPC-a. LOI vrijednosti rasle su s povećanjem udjela nanočestica do 28,5 %, što je upućivalo na veću potrebu za kisikom. Poboljšanje je iznosilo do 54 % u usporedbi s čistim HDPE-om. Štoviše, poboljšano je i stvaranje pougljenjenog sloja koji je pridonio povećanju vatrootpornosti. Istraživanje pretražnim elektronskim mikroskopom (SEM) pokazalo je da su se nanočestice dobro dispergirale u matrici. Međutim, tendencija aglomeraciji povećala se s povećanjem koncentracije nanočestica. Sposobnost karbonizacije površine drvnih vlakanaca tijekom gorenja također je pridonijela poboljšanju toplinske stabilnosti i vatrootpornosti WPC-a.

KLJUČNE RIJEČI: *nanočestice ZnO; LOI test; TGA analiza; toplinska otpornost; WPC*

1 INTRODUCTION
1. UVOD

Climate change is increasing the pressure on the environment, driving manufacturers to produce greener products. Today, however, it is not only the desire for green materials that is more important, but also efficiency. The scarcity of raw materials also makes efficiency vital. Composite materials meet these requirements. Composites consist of two or more materials with different properties than the sum of the properties of two components. Wood-based composites are among the most popular composites because of their green label. Wood is a renewable, biodegradable, and abundant material, distinguishing it from other materials. The properties of wood vary between species, within the same species, and even more between different parts of the same tree. However, composites made from wood have homogeneous properties that attract designers, architects, engineers, etc. Today, wood-based composites are evaluated for everything from furniture to structures, car parts to unique designs.

WPCs, one of the wood-based composites, have existed for over 50 years in various applications: decks, fences, railing, garden furniture, doors, and window frames (Kim and Pal, 2010; Klyosov, 2007). Mixing wood and plastic creates a new material with superior properties. Plastic is one of the most remarkable inventions of the 20th century. Its high resistance to outdoor conditions makes it a more practical material against factors such as UV light, moisture, insects, fungi, etc. However, wood does not have the same advantages. On the contrary, it can be easily degraded by biotic and abiotic factors under the right conditions. Therefore, WPCs have become more desirable in recent years due to their high durability, high strength, low cost, non-corrosion, and environmental friendliness. Moreover, they can be considered an alternative material for most industries such as construction, car, architectural and structural design, aerospace, etc. (Erchiqui *et al.,* 2020; Smith and Wolcott, 2006).

Composites are recognized as superior in comparison with their own constituents. However, there is still a need for improvement to overcome the existing shortcomings. Most research has focused on improving WPCs physical, mechanical, and weathering durability. However, the high flame sensitivity of polymer restricted WPC application, mainly indoors and outdoors, due to higher heat during thermal degradation. Heat or temperature differentiations affect WPCs thermal properties (Guo *et al*., 2019a). Free radicals initiate oxidation reactions during thermal degradation, decreasing the thermal stability of polymers (He *et al*., 2012). The chain scissions resulted in mass losses with increasing temperature due to thermal-oxidative reactions. Eventually, most of the structure is converted to carbon monoxide (CO), carbon dioxide (CO_2), and water (H_2O) (Devi and Maji, 2012). In addition, the polymer threatens structural integrity, especially in structural design, due to its formability at low temperatures.

On the other hand, wood is a biopolymer consisting of cellulose, hemicellulose, and lignin. When exposed to heat or temperature, there are also some changes in the chemical and physical structure of wood. The cross-linked structure makes lignin more resistant to high temperatures, while hemicellulose is vulnerable (Lowden and Hull, 2013). Acetic and formic acid, furfural, and methanol formation occur during high-temperature exposure, which degrades cellulose and hemicellulose amorphous structures (Özgenç *et al*., 2017). Meanwhile, thermal degradation involves liquefaction, gasification, and pyrolysis (Mohan *et al*., 2006). Liquefaction gradually decreases and turns into some gases and solids; CO, CO_2, and H_2O, followed by gasification, which starts below 200 °C with non-combustible gases. Combustible gases also begin to accompany the process as the temperature rises, which starts the combustion of the combustible material. This produces char, which breaks down the wood.

The incompatibility between wood and polymer is also a crucial issue for the thermal stability of WPCs, as well as for their physical and mechanical properties. The bond between the polymer and wood flour, which are hydrophobic and hydrophilic, respectively, has a negative effect on the thermal expansion of WPCs (Yang *et al*., 2005).

The effect of various fillers on the physico-chemical and mechanical properties of polymer composites is determined by many factors. Moreover, some studies have also been carried out to improve the thermal stability of WPC with the reinforcement of synthetic fibers. Rasana *et al*. (2019) stated that glass fibers act as a thermal shield and inhibit degradation. The degradation of carbon fibers above 600 °C, which is higher than polyethylene and wood, improves the thermal stability of WPCs (Eibl, 2017; Guo *et al*., 2019b; Yao *et al*., 2018). As in previous studies, the thermal degradation of WPCs was retarded with the reinforcement of glass and carbon-woven fabrics (Durmaz *et al*., 2021). Moreover, some studies also dealt with improving the thermal stability and fire performance of fibers with aerogels (He *et al*., 2022; Zhang *et al*., 2023).

Nanotechnology applications have been quite prevalent in recent years. The small size of nanoparticles surpasses their traditional counterparts and remarkably improves the materials properties (De Filpo *et al*., 2013). Nanomaterials improve the mechanical and physical properties of WPCs, and also significantly improve the thermal stability and fire performance (Chaharmahali *et al*., 2014). Metal oxide nanoparticles are the preferred nanoparticles for most applications. ZnO nanoparticle is one of the metal oxides evaluated in various applications due to its inherent properties (Franco-Urquiza *et al*., 2020). Zhao and Li (2006) highlighted that the thermal stability of polypropylene-based WPCs with ZnO nanoparticles was maintained even after exposure to UV light. Devi and Maji (2012) also found that the degradation temperature improved with the thermal shielding ability of ZnO nanoparticles. The small size of the nanoparticles allows them to cover the material surface, inhibiting the free radicals that occur during thermal degradation. As a result, the degradation temperature was improved. Jouyandeh *et al*. (2019) also found that increasing the content thickens the nanoparticle layer, which acts as an insulator and reduces heat transfer. Moreover, ZnO nanoparticles can absorb UV light between 200-380 nm (He *et al*., 2009). As in previous studies, ZnO nanoparticles improved weathering resistance with increasing content. The loss of mechanical properties was also reduced with decreasing crack formation (Durmaz *et al*., 2022).

High thermal stability and fire resistance are essential for the expansion of indoor and outdoor applications of WPCs. The main objective of this study was to improve the thermal strength and fire resistance of HDPE-based WPCs with ZnO nanoparticle reinforcement. For this purpose, the effect of ZnO nanoparticles at high contents on the WPCs thermal stability and fire performance was investigated. The distribution of ZnO nanoparticles was investigated by SEM analysis. The thermal behavior of the reinforced WPCs was investigated using TGA. The LOI test also revealed the fire behavior of the reinforced WPCs.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

The pine wood flour (*Pinus sylvestris* L.) with 40-60 mesh dimensions as a lignocellulosic filler and HDPE as a thermoplastic polymer were supplied from commercial suppliers (Ucar Plastic, İzmir, Turkey). HDPE is one of the world's most popular plastics due to its resistance to high temperatures, strong chemicals, and UV light. The fine-grain polymer (~200 mesh) was used to obtain a homogenous mixture. The melt flow index (MFI) and density of HDPE were 5.5 g/10 min (190 °C/2.16 kg) and 0.965 g/cm^3, respectively. Maleic anhydride grafted polyethylene (Licocene PE MA 4351 Fine Grain) was used as a coupling agent to improve the bonding between the materials. The MAPE softening point and density were 123 °C and 0.99 g/cm^3, respectively. The ZnO nanoparticles with dimensions of 30-50 nm and a density of 5.5 g/cm^3 were used as a reinforcement agent (Nanografi, Ankara, Turkey).

2.2 Methods
2.2. Metode

The wood flour, thermoplastic polymer, and nanoparticles were mixed in a mechanical mixer (1200 rev/min) to obtain a homogeneous mixture, according to the scheme in Table 1. Before manufacturing, wood flour was oven-dried until constant weight at 80 °C. Then, the homogeneous mixture was extruded with a single screw extruder (Teknomatik, Turkey). The screw speed was set to 40 rpm. The extruder temperature was from 180 to 195 °C. The extruded samples were cooled in water and pelletized. The pellets were dried at (102±2) °C and hot pressed at 180 °C for 15 min (CemilUsta SSP 125, Istanbul, Turkey). The reinforcement of ZnO nanoparticles decreased the melt flow index of the polymer. The pressure of the hot press was 2.35-

Table 1 Wood flour, polymer, and nanoparticle content (%)
Tablica 1. Sadržaj drvnog brašna, polimera i nanočestica (%)

Groups *Grupe*	**Wood/Polymer ratio** *Omjer drvo / polimer*	**Nanoparticle content** *Sadržaj nanočestica*
Neat-HDPE	0/100	-
WPC40	40/60	-
WPC50	50/50	-
WPC40-1	40/59	1
WPC40-3	40/57	3
WPC40-5	40/55	5
WPC40-10	40/50	10
WPC50-1	50/49	1
WPC50-3	50/47	3
WPC50-5	50/45	5
WPC50-10	50/40	10

2.55 MPa. The dimensions of the panels were 500 mm × 500 mm × 4 mm.

2.3 Thermal properties
2.3. Toplinska svojstva

TGA indicates the difference in the weight of samples as a function of temperature or time. DTGA is also obtained by taking the first derivative of TGA curves as a temperature or time function. The WPC samples underwent grinding utilizing an IKA grinder with a 1 mm sieve. The thermal stability of WPCs was determined with Perkin–Elmer STA 6000 thermogravimetric analyzer 4000 (USA). Samples were heated from 30 °C to 600 °C with a heating rate of 10 °C/min under nitrogen gases. Two samples were tested for each group.

The LOI test is one of the most prominent test methods for characterizing the fire behavior of materials due to its ability to analyze small samples. The LOI test determines the minimum oxygen content for the continuing flame combustion of the samples. The LOI test was performed with the Dynisco LOI analyzer (Franklin, USA) (Figure 1) according to ASTM D 2863-19 (2019). The LOI was measured on five samples for each group (with one sample measuring 127 mm × 12.7 mm × 5 mm). Each sample was placed in the center of a glass tube in which a mixture of oxygen and nitrogen gas was released. After the gas mixture had expanded the glass tube, the sample was burned with a pilot fire. The sample was monitored during the 50 mm of burning or flaming for 180 s. The higher LOI values indicate resistance to flammability, while the lower LOI values are associated with flammable materials.

2.4 SEM analysis
2.4. SEM analiza

The WPC samples were investigated by a scanning electron microscope (Zeiss, Evo LS10). Before the investigation, WPC samples were dried until constant weight and coated with gold (Emitech, SC7620).

Figure 1 LOI instrument
Slika 1. LOI uređaj

2.5 Statistical analysis
2.5. Statistička analiza

A comprehensive statistical analysis was conducted to explore the data, employing a robust approach involving analysis of variance (ANOVA) followed by post-hoc Duncan testing (significance level set at $p<0.05$). This rigorous statistical methodology allowed for a thorough examination of the dataset, and consequently meaningful conclusions could be drawn based on the observed differences among the variables.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 TGA analysis
3.1. TGA analiza

The behavior of materials against gradual temperature increase is essential for applications. The effect of ZnO nanoparticles on the gradual temperature increase (10 °C/min) was examined by TGA analysis, as seen in Table 2. The degradation of WPCs occurred in three temperature stages, as shown in Figure 2. As the temperature increased, the weight loss of the WPCs increased. First, the moisture released from the samples accelerated after 50 °C, causing a slight weight loss (~3-4 %). The primary degradation (2nd stage) was initiated after 180 °C. It is well-known that the main wood cell wall components, hemicellulose, cellulose, and lignin, begin to degrade above this temperature (Jarukumjorn and Suppakarn, 2009; Rowell, 2012).

It is well known that the thermal stability of wood is lower than that of polymer. Therefore, the first degradation was initiated in the wood cell wall components (Ramesh *et al.,* 2022). Hemicellulose is the most sensitive to thermal degradation among the other cell wall components - degradation of hemicellulose followed by cellulose and lignin. In addition, the increase in wood content has a negative effect on the weight loss of WPCs. As shown in Table 2, the higher the wood content, the higher the weight loss. As the wood content increases, the encapsulation of the wood fibers decreases, which weakens the thermal stability. Meanwhile, the carbonization of wood fiber improves the thermal stability of the polymer due to the thermal shielding effect (Guo *et al.*, 2019a). In addition, the wood content also affects the residue. Wood can contain up to ~5 % ash, contributing to the increase in residue.

The degradation temperature of the second stage varied from 175.19 to 204.24 °C, in which hemicelluloses started to degrade first, followed by others. The final degradation stage (above 390 °C) was the decomposition of HDPE. Under the inert atmosphere, the py-

Table 2 TGA values of WPCs
Tablica 2. TGA vrijednosti WPC-a

Groups *Grupe*	**Stage** *Stadij*	***T* onset, °C**	***T* endset, °C**	***T* deg., °C**	**Weight loss, %** *Gubitak mase, %*	**Weight onset, %** *Masa na početku, %*	**Weight endset, %** *Masa na kraju, %*
Neat-HDPE		327.29	526.47	473	96.70	98.0916	1.395
WPC40	2nd stage	179.34	398.47	345.64	27.42	98.703	71.286
	3rd stage	398.47	529.82	473.96	60.72	71.286	10.568
WPC50	2nd stage	175.19	396.71	344.52	34.17	98.095	63.926
	3rd stage	396.71	523.27	449.17	51.44	63.926	12.487
WPC40-1	2nd stage	181.25	398.95	347.4	26.26	98.426	72.169
	3rd stage	398.95	529.18	474.28	60.36	72.23	11.87
WPC40-3	2nd stage	188.6	398.95	346.76	26.76	98.469	71.712
	3rd stage	398.95	530.62	473.32	58.85	71.712	12.867
WPC40-5	2nd stage	191.31	397.51	347.56	26.87	98.81	71.94
	3rd stage	397.51	526.94	469.81	57.04	71.94	14.9
WPC40-10	2nd stage	204.08	395.91	346.28	26.18	98.691	72.51
	3rd stage	395.91	523.11	468.37	52.69	72.51	19.825
WPC50-1	2nd stage	189.39	399.74	347.4	31.98	97.887	65.912
	3rd stage	399.74	530.3	474.44	52.95	65.912	12.959
WPC50-3	2nd stage	180.93	402.3	349.31	34.71	98.189	63.476
	3rd stage	402.3	529.5	473.16	49.01	63.476	14.469
WPC50-5	2nd stage	204.24	399.58	346.28	31.64	98.084	66.445
	3rd stage	399.58	529.98	471.08	48.10	66.445	18.349
WPC50-10	2nd stage	202.32	404.05	350.75	36.21	96.715	60.509
	3rd stage	404.05	544.98	466.46	28.89	60.509	31.624

rolytic degradation starts the cleavage reactions, resulting in rapid weight loss (Peters, 1979). Therefore, the neat HDPE was easily degraded due to its structure. The DTGA plot also showed the rapid degradation of neat HDPE, as seen in Figure 3. As in the previous study, we found that ZnO nanoparticles limit the interaction with active surface groups by covering the surface (Durmaz *et al*., 2022). In addition, the nanoparticles cover the materials, which scavenges oxygen from the surface and inhibits scission reactions. The synergistic effect of carbonization of wood and nanoparticles limits the O_2 penetration, creating a barrier to thermal degradation. Therefore, the degradation temperatures were increased due to the heat-shielding role of nanomaterials as well as the carbonization of wood fibers. The increased degradation temperature is crucial because it retards the softening of the matrix and ceases the relaxation process (Xian *et al*., 2023).

In addition, ZnO nanoparticles inhibited the free radicals that cause thermal oxidative reactions, which reduced the degradation rate and improved thermal stability. Increasing the content of ZnO nanoparticles also

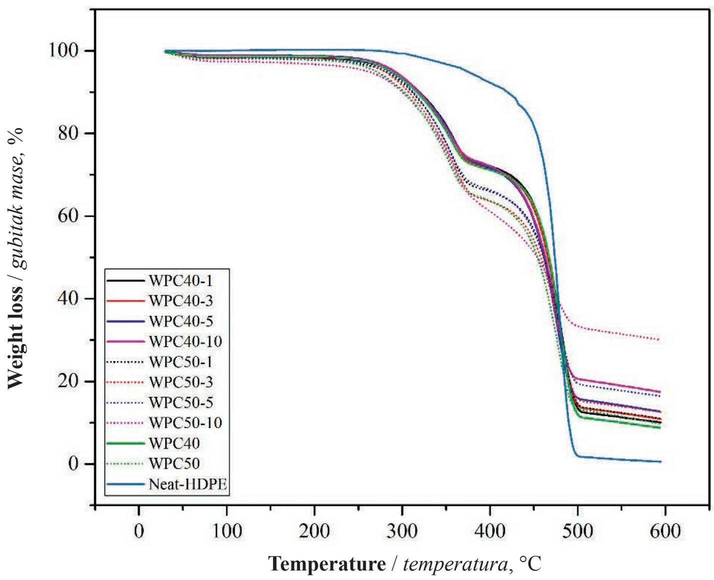

Figure 2 TGA thermograms of WPCs
Slika 2. TGA termogrami WPC-a

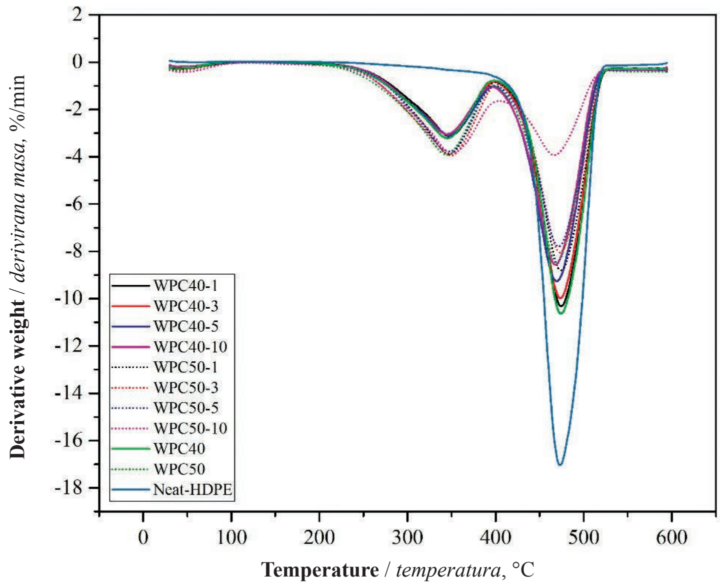

Figure 3 DTG thermograms of WPCs
Slika 3. DTG termogrami WPC-a

affected the degradation rate. The nanoparticle layer becomes thicker with increasing nanoparticle content, which increases the heat requirement for free radicals to cause scission reactions. Similarly, Uyup *et al*. (2019) attributed the higher thermal stability to the higher nanoparticle content. Therefore, the lowest degradation rate was observed for WPC50-10, as shown in the DTGA plot. In addition, HDPE degrades to volatile compounds with no residue above 550 °C (Dorigato *et al*., 2012). However, the addition of wood and nanoparticles increased the residue, which is essential for fire performance, as discussed below. The highest residue was detected from ZnO50-10. As nanoparticle content increases, they tend to agglomerate, affecting the matrix thermal conductivity, which limits the polymer chain scission reactions (Anu and Pillai, 2022). Therefore, this situation could increase the char residue for WPC-10. Xu *et al*. (2019) also highlighted the contribution of ZnO nanoparticles to char residue, which limits heat transfer and oxygen penetration.

3.2 LOI test
3.2. LOI test

The effect of ZnO nanoparticles on the fire performance of WPCs was examined by the LOI test. The oxygen is necessary for continuing flaming combustion. Fire-resistant materials also need much more oxygen for flaming combustion than easily combustible materials. As seen in Figure 4, the LOI values of WPCs were between 18.5 to 28.5. Due to its structure, neat HDPE is recognized as a combustible and flammable droplet thermoplastic. Therefore, it can be easily flamed. Free radicals, such as alkyl or alkyl peroxide, are formed due to oxidative reactions during thermal

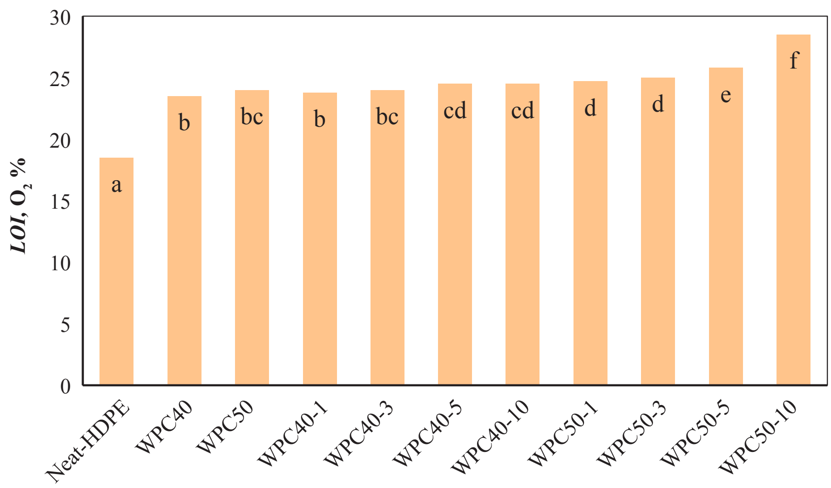

Figure 4 LOI values of WPCs, with letters indicating significant group differences ($p < 0.05$) determined by Duncan test
Slika 4. LOI vrijednosti WPC-a sa slovima koja označavaju značajne grupne razlike ($p < 0,05$) utvrđene Duncanovim testom

Figure 5 LOI samples
Slika 5. Uzorci nakon LOI testa

degradation, which initiates polymer chain degradation (He *et al*., 2012). As the degradation progresses, the free radicals are transformed into CO, CO_2, and H_2O during degradation. Therefore, the fire resistance of neat HDPE was the lowest. In addition, the conversion to CO, CO_2, and H_2O during thermal degradation results in low char formation, which reduces the fire performance of the polymer. The reduced char formation also contributes positively to the polymer dripping during the LOI test.

The lignocellulosic materials are critical in reducing the heat release rate, heat release during combustion, and mass loss rate (Borysiak *et al*., 2006; Kozłowski and Władyka-Przybylak, 2008). Carbonization of wood fiber during carbonization limits O_2 penetration from the surface to the center of the fiber, which improves fire performance. In addition, carbonization of the wood fiber enhances the char formation of WPCs, which limits the dripping of WPCs, as seen in Figure 5. This phenomenon is not neglected for structural applications. The addition of wood flour to the polymer increased the oxygen demand, resulting in higher LOI values. In addition, the polymer penetrates the wood interstices during extrusion. Nanoparticles absorbed by the polymer and deposited in the wood cells provide a barrier that scavenges oxygen from the surface and inhibits oxidation reactions, thereby improving fire performance. The deposited nanoparticles also coat the material, limiting the interaction of components with O_2, which is essential for continued flaming.

The reinforcement with ZnO nanoparticles increased the LOI values up to 28.5 %. Previous studies have also shown that nanoparticles inhibit heat transfer and reduce combustible volatile compounds (Sun *et al.,* 2012; Yin *et al*., 2022). In addition, the burning drop was improved as the nanoparticles acted as a heat shield. While the combustion behavior of neat HDPE was flaming drips, the reinforced WPCs charred without dripping, which improved the fire resistance. Therefore, the fire classification was upgraded from "flammable or combustible material" to "limited fire retardant of fire-resistant material" according to ISO 4589-1 (2017), as a result of reinforced wood fibers and nanoparticles. Adding nanoparticles to WPCs improves fire performance, which is critical for indoor applications.

3.3 SEM analysis
3.3. SEM analiza

The distribution of ZnO nanoparticles in the matrix is crucial in improving fire and thermal performance. Therefore, the structure of WPC samples was investigated by SEM analysis, as seen in Figure 6. SEM images showed that the nanoparticles were well-dispersed in the matrix. It was thought that the powder form of polyethylene played a key role in distribution. Mainly, the lowest content resulted in a good dispersion of nanoparticles (white circles). On the other hand, as nanoparticle content increased, the tendency to agglomerate increased (white arrows). As stated in previous studies, the good dispersion of nanoparticles helped to improve the technological properties of WPCs (Durmaz *et al*., 2022). Likewise, ZnO nanoparticles were well dispersed in the matrix and covered the surface of materials, as clearly seen in the highest content of nanoparticles (Figure 6d, h). As stated above, nanoparticles occurred in thicker layers on the materials with increasing content, which inhibited heat transfer for the scission reaction of the polymer. Therefore, the degradation temperature increased, as explained in the TGA analysis. Moreover, the oxygen penetration was limited. Significantly, the highest nanoparticle content contributed to char formation, which descended the dripping of WPCs during the combustion and improved the fire performance of WPCs.

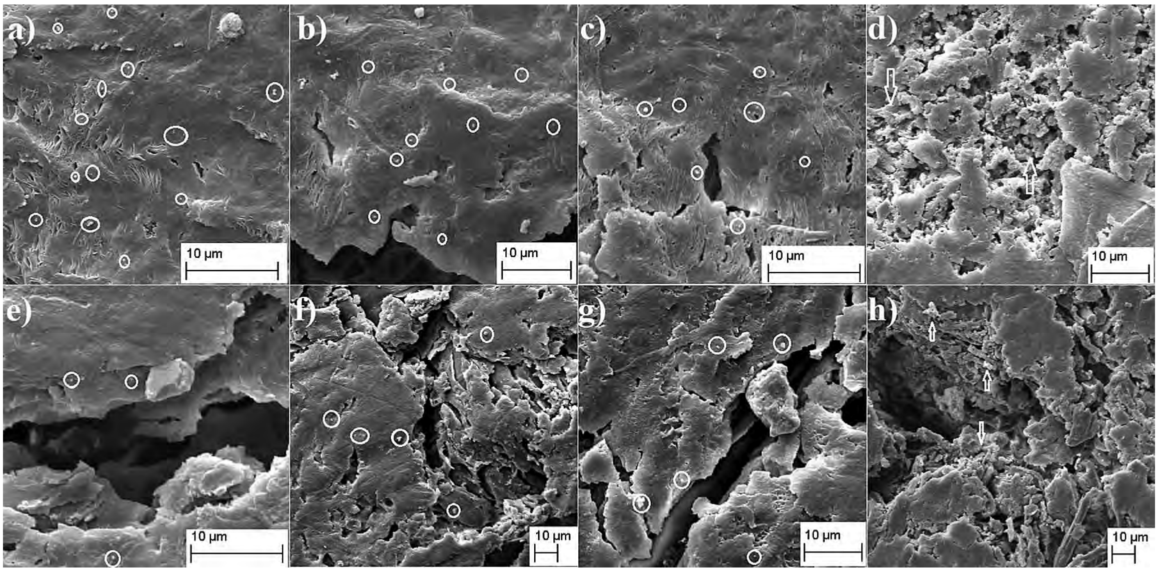

Figure 6 SEM images of WPCs: a) WPC40-1, b) WPC40-3, c) WPC40-5, d) WPC40-10, e) WPC50-1, f) WPC50-3, g) WPC50-5, h) WPC50-10 (White circles show ZnO nanoparticles, while white arrows show agglomeration of ZnO nanoparticles)
Slika 6. SEM slike WPC-a: a) WPC40-1, b) WPC40-3, c) WPC40-5, d) WPC40-10, e) WPC50-1, f) WPC50-3, g) WPC50-5, h) WPC50-10 (bijelim kružićima označene su nanočestice ZnO, a bijele strelice pokazuju aglomeraciju nanočestica ZnO)

4 CONCLUSIONS
4. ZAKLJUČAK

The petroleum-based polymers and wood are susceptible to thermal degradation. Many prior investigations have predominantly focused on assessing the impact of relatively modest quantities of ZnO nanoparticles in WPCs. However, in this study, we have elevated the thermal stability and fire-resistant characteristics of HDPE-based WPCs by augmenting them with ZnO nanoparticles, with enhancements observed at levels of up to 10%. TGA analysis also clearly demonstrated the thermal behavior of neat HDPE. The gradual increase in temperature leads to the formation of free radicals on the chemical chains of the polymer, which ultimately causes chain scission and mass loss. TGA analysis demonstrated that wood thermal stability was lower compared to polymer. However, the degradation pattern was changed with the addition of wood flour. Although the wood fibers thermally degraded at lower temperatures, the char formation improved the thermal stability. In addition, ZnO nanoparticles cover the surface of materials and insulate them from heat by acting as a heat shield. Therefore, the degradation was slowed down, and the degradation temperature increased. The surface coverage of the nanoparticles also inhibited free radicals, a key factor in the degradation rate. There was a rapid degradation for neat HDPE, while it was very moderate for ZnO nanoparticles.

Oxygen is an essential factor for combustion, as determined by the LOI test for WPCs. Carbonization of wood fiber surfaces inhibits oxygen penetration, which improves fire resistance. Therefore, only wood flour significantly improved the LOI values. In addition, nanoparticle reinforcement also supported insulation and increased oxygen demand. Meanwhile, char formation affected the fire performance. Increasing the wood flour and nanoparticle content increased the char formation, which helped to improve the fire performance. Nanoparticle reinforcement also limited dripping. SEM images indicated that nanoparticles were well-dispersed in the matrix. However, the increase in the nanoparticle content increased the tendency for aggregation. On the other hand, WPCs containing the highest nanoparticles were well covered, which clearly explained why thermal and fire performance improved. Consequently, improving thermal stability and fire performance with ZnO nanoparticles could contribute to improving WPC applications on a large scale. As the application range of WPCs widens, thermal stability is vital for safety in use. The improved thermal resistance makes WPCs safer for many applications, such as siding, decking, playgrounds, gardening furniture, windows, doors, etc.

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Bio-Durability and Engineering Characteristics of Heat-Treated Poplar Wood

Biološka trajnost i tehnička svojstva toplinski modificirane topolovine

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ABSTRACT • *The aim of this study was to evaluate the effect of brown rot fungus Coniophora puteana activity on physical and mechanical properties as well as biological resistance of heat-treated poplar wood. Two poplar wood species (Populus deltoids and Populus nigra) were heat-treated by thermo-wood (Thermo-D) method. Control and heat-treated specimens were exposed to brown rot fungus C. puteana for 16 weeks. Physical and mechanical characteristics of specimens including density, compression strength parallel to the grain and impact strength were evaluated before and after exposure to fungus. Mass loss of specimens caused by fungal activity (MLF) was also calculated. In addition, the effect of thermal modification on laccase production by C. puteana was assayed. The highest mass loss due to fungal deterioration was observed in control specimens, coinciding with the highest substrate-enzyme interactions and constant decrease in detectable laccase levels. According to the results, thermal modification can be used effectively to protect poplar wood against brown rot fungus attack.*

KEYWORDS: *poplar; thermo-wood; fungus; physical and mechanical properties; biological resistance*

SAŽETAK • *Cilj ovog istraživanja bio je procijeniti utjecaj gljive smeđe truleži Coniophora puteana na fizička i mehanička svojstva te na biološku otpornost toplinski modificirane topolovine. Dvije vrste topolovine (Populus deltoids i Populus nigra) toplinski su modificirane postupkom thermo wood (Thermo D). Kontrolni i toplinski modificirani uzorci bili su izloženi 16 tjedana gljivi smeđe truleži C. puteana. Prije i nakon izlaganja gljivama određena su fizička i mehanička svojstva uzoraka uključujući gustoću, čvrstoću na tlak paralelno s vlakancima i čvrstoću na udarac. Također je izračunan gubitak mase uzoraka kao posljedica aktivnosti gljiva (MLF). Osim toga, ispitan je utjecaj toplinske modifikacije na stvaranje lakaze zbog djelovanja gljive C. puteana. Najveći gubitak mase, tj. najveća razgradnja nastala djelovanjem gljive zabilježena je na kontrolnim uzorcima, a to se podudara s najjačim međusobnim djelovanjem supstrata i enzima te s konstantnim smanjenjem detektirane razine lakaze. Prema dobivenim rezultatima, toplinska se modifikacija može učinkovito iskoristiti za zaštitu topolovine od napada gljiva smeđe truleži.*

KLJUČNE RIJEČI: *topolovina; thermo-wood; gljive; fizička i mehanička svojstva; biološka otpornost*

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1 INTRODUCTION
1. UVOD

Wood as a natural polymer has been widely used in structures for many years. However, some properties of this natural material such as moisture absorption, dimensional instability, biological degradation, weathering, etc., limit its use. So, numerous new methods of wood modification have been developed to remove these disadvantages and expand its functionality. Wood heat treatment is one of the modification methods, which is carried out by different processes. This kind of modification was first studied scientifically by Stamm and Hansen in the 1930s in Germany and in the 1940s by White in the United States. The most comprehensive research in this field has been carried out by VTT (International Thermo-Wood Association) in Finland, whose product is called thermo-wood. A temperature of 160-260 °C is usually used to produce thermo-wood (Militz, 2002). Thermal modifications have some positive and negative effects on wood. In some cases, thermal treatments lead to positive changes in chemical structure of wood, colour, dimensional stability, thermal insulation properties, biological resistance to biological degradation. However, these treatments also reduce some mechanical properties such as wood bending strength.

Heat treatment of wood in the temperature range of 140-260 ºC with a long holding time causes an irreversible reduction of moisture absorption (González-Peña *et al.,* 2004; Obataya and Tomita, 2002); it leads to dimensional stability (Krause *et al.,* 2004), less moisture movement (Militz and Tjeerdsma, 2001), and improved biological resistance (Welzbacher and Rapp, 2004; Farahani *et al.,* 2001). However, one of the major drawbacks of these treatments is the reduction of wood mechanical properties (González-Peña and Hale, 2007).

Brown rot fungi have an enzyme system that produces hydrolytic and oxidative enzymes, which act on cell wall component degradation such as lignin, cellulose and hemicelluloses (Baldrian and Gabriel, 2003). Cellulases can be classified into three types: endoglucanases, exoglucanases and 1, 4-β-glucosidases (Gielkens *et al.,* 1999). Lignin peroxidase (LiP), manganese peroxidase (MnP) and laccase (Lac) are the most widely distributed lignin-degrading enzymes, which are responsible for the biodegradation of lignin (Vicuna, 2000).

Evaluation of heat treatment effect on bio-durability of a wood species showed that it significantly increases wood durability against fungal action due to the reduction of hydroxyl groups (Mburu *et al.,* 2007). Kamperidou (2019) evaluated the biological durability of thermo-chemically modified pine and spruce wood against white and brown rot fungi. The results showed that thermal modification presented lower mass loss caused by used fungi compared to the unmodified wood. In other words, heat treatment increases the biological resistance of wood.

According to Gao *et al.* (2016), the colour of wood became darker and there was an improvement in dimensional stability as well as reduction in modulus of elasticity of heat-treated poplar wood at temperatures of 140 to 200 °C for 1-3 hours. In addition, bio-durability of heat-treated specimen was better against white rot fungus than brown rot fungus.

Furthermore, Corleto *et al.* (2020) investigated the effect of thermal modification on properties of padauk wood, indicating that heat treatment at high temperature caused significant loss in bending strength and bending stiffness of padauk wood. Moreover, the results of chemical analysis showed that cellulose and lignin proportion increased, while that of hemicellulose decreased substantially following thermal modification. Another study showed that durability and dimensional stability of European birch, European aspen, Norway spruce as well as Scots pine improved by thermal modification (Karlsson *et al.,* 2011; Militz and Altgen, 2014).

Kaygin *et al.* (2009) investigated the effect of mass loss of mechanical properties of heat-treated Paulownia at 160, 180 and 200 °C. The results showed that mechanical properties including compression strength, modulus of elasticity, bending strength and impact strength decreased with increasing of heat treatment temperature. In fact, it was determined that, due to thermal modification, the mass loss significantly affected mechanic properties of Paulownia wood.

Heat treatment can be used effectively against fungal attack for Scotch pine, oak and beech wood species (Ayata *et al.,* 2017). At high temperature of wood thermal modification, the lignin content increases because of autocondensation and its higher thermal stability. There is a slight decrease in the extractives content. Moreover, the cellulose and its DP decrease, while cross-linking reactions occur in the cellulose (**Čabalová** *et al.,* 2019).

Brown rot fungi have the ability to degrade cellulose by producing extracellular fungal cellulose degrading enzymes. The extracellular enzymes produced by these fungi are greatly affected by the type of substrate being decayed. Our study focuses on measuring oxidative enzyme laccases secreted by *C. puteana* in the presence of poplar woods (control and heat-treated specimens) as substrates. Therefore, this study aimed to find the effect of heat treatment on biological resistance of thermo-wood against degradation by brown rot fungus *Coniophora puteana*. Meanwhile, physical and mechanical properties of thermo-wood and control specimen after exposure to *C. puteana* were investigated. In addition, levels of laccase enzyme on both substrates (control and modified specimens) were assayed.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

The lumbers of two poplar wood species (*Populus deltoids* and *Populus nigra*) were prepared and treated in Mazand wood company. After kiln loading, the temperature was rapidly raised to a level of around 100 ºC and then increased steadily to 130 ºC. Thereafter, the temperature inside the kiln was increased to around 212 °C and remained constant for 3 hours. The final stage was cooling and moisture conditioning by using water spray system. When the temperature reached 80 ºC, re-moisturizing took place to bring the wood moisture content to a level of around 6 %. In order to evaluate biological resistance as well as physical and mechanical properties of treated timbers, six specimens were prepared in accordance with EN 113-2:2020 standard.

In this study, brown rot fungus *Coniophora puteana* was used to evaluate biological resistance of control and modified specimens. Modified and control specimens were exposed to fungal mycelium in Kolle-flasks. As a nutrient medium for fungal mycelium, Malta Extract Agar (Merck KGaA, Darmstadt, Germany) was used. After preparing nutrient medium and sterilization in autoclave (temperature 120 °C, time 20 minutes, pressure 1.5 kg/cm^2), small pieces of fungal mycelium were inoculated in Kolle flasks. Then the flasks with nutrient medium and mycelium were incubated in the climate chamber at a temperature of 22 °C and 65±5 % relative humidity for 2 to 3 weeks until the entire surface of the nutrient medium was overgrown by mycelium (as shown in Figure 1). Six modified and control specimens were placed in each flask and were incubated in the same climate chamber for another 16 weeks according to EN 113-2: (2020) standard (Figure 1). After 16 weeks, all Kolle flasks were taken out from the climate chamber.

Ligninolytic enzyme laccase was extracted at 25 ºC in two extraction cycles, 5 and 24 hours, with 50 mM sodium acetate buffer (pH=5.5) with 0.1 g/L Polysorbate 20 (Tween® 20; from Sigma Aldrich: Steinheim, Germany). It should be noted that two extraction cycles of 5 and 24 hours were used to obtain the optimal time for maximum enzyme extraction. An amount of 1.5 gr chips prepared from decayed specimens were first soaked in 50 mL extraction buffer for 5 hours. The second extraction was performed with 25 mL extraction buffer for 24 hours. For this purpose, six repetitions were considered. Then, the supernatants collected from all extractions were filtered, centrifuged at 5.000 rpm for 15 minutes and used to test enzyme activity.

Laccase activity was assayed by measuring the oxidation of 2,6-DMP. An aliquot of enzyme solution was incubated in 1 ml of 5 mM of 2,6-Dimethoxyphenol (2,6-DMP) in 0.1 M sodium acetate buffer (pH= 3.6) at 30 ºC. Absorbance was monitored at 469 nm in a spectrophotometer. One unit of laccase activity was defined as the amount of the laccase that oxidized 1 µmol of 2,6-DMP per minute at 30 ºC (Field *et al.,* 1993).

In order to evaluate the durability of wood specimens against brown rot fungus, the mass loss was calculated. After 16 weeks of incubation, the wood specimens were taken out of the Kolle flasks and carefully cleaned from the mycelium and then oven dried for 24 hours at the temperature of 103±2 °C until the constant mass was reached, and then weighed. The mass loss

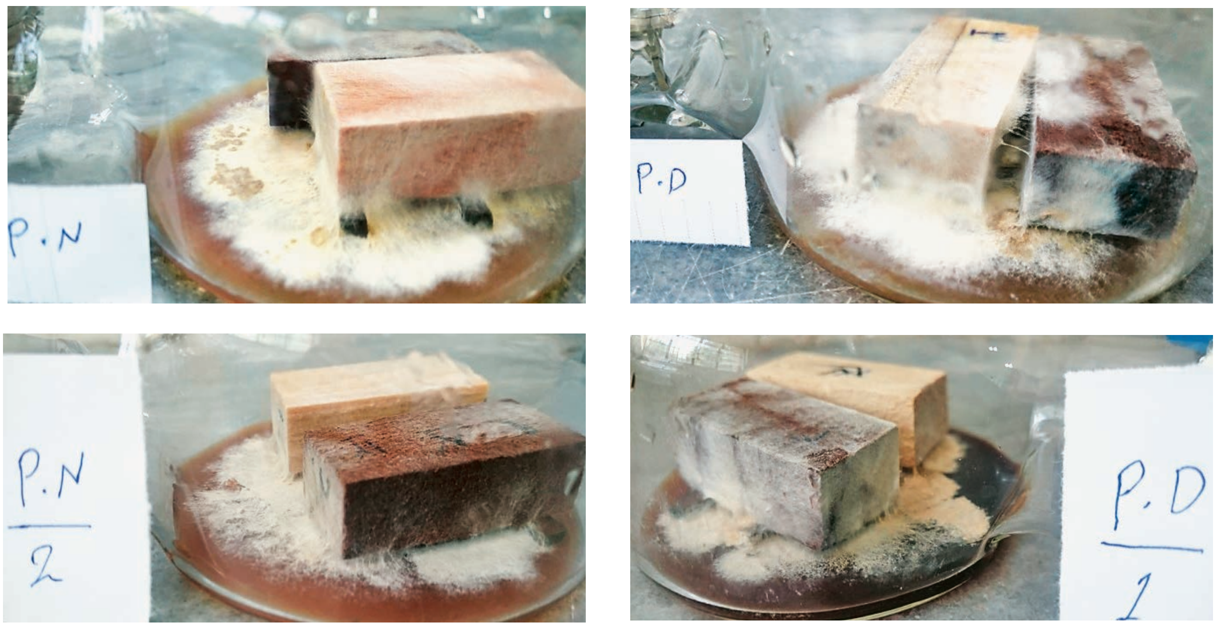

Figure 1 Control and heat-treated specimens after exposure to brown rot fungi
(Up: after 3 weeks, Down: after 16 weeks)
Slika 1. Kontrolni i toplinski modificirani uzorci nakon izlaganja gljivama smeđe truleži
(gore: nakon tri tjedna, dolje: nakon 16 tjedana)

and the repellent effect of thermal modification on *MLF* of each specimen were calculated according to equation 1 and 2, respectively:

$$M.L= ((D_1-D_2)/D_1) \times 100 \qquad (1)$$

Where:
M.L – mass loss (%)
D_1 – oven dry mass of specimen before exposure to fungi (g)
D_2 – oven dry mass of specimen after exposure to fungi (g)

$$R.E= ((M.L_1 - M.L_2)/ M.L_1) \times 100 \qquad (2)$$

Where:
R.E – repellent effect of thermal modification on *MLF* (%)
$M.L_1$ – mass loss of control specimen caused by fungi (%)
$M.L_2$ – mass loss of heat-treated specimen caused by fungi (%)

In this research, only the properties that could be determined based on the specimens that can be placed in Kolle flasks, based on standard dimensions, were considered. The physical and mechanical properties of control and heat-treated specimens were tested according to ASTM D 143-09. Also, the standard ASTM D256 was used to measure impact strength, because the dimensions of the specimens in this standard were suitable to be placed in the Kolle flask. Moreover, the standard EN 113 was used to evaluate bio-durability. It should be noted that 6 replications were considered for each parameter. The obtained data from the tests were statistically analyzed based on One-Way ANOVA method. This method was used to determine any statistically significant differences between the means of the two groups (control and modified specimens) for each wood species separately.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

In this research, the physical and mechanical properties of two wood species (*P. deltoids* and *P. nigra*) were evaluated in two stages: the first after thermal modification and the second after fungal degradation to show the effect of heat treatment on fungal functionality as well as physical and mechanical properties. Table 1 summarizes the results of statistical analysis of One-Way ANOVA test for physical and mechanical properties of two poplar wood species, specifying significant levels. The numbers presented in Table 1 show whether the heat treatment had a significant effect on the investigated properties.

3.1. Laccase activity assessment
3.1. Procjena pojave lakaze

The results of laccase evaluation of specimens after 16 weeks of exposure to *C. puteana* showed that the laccase value in controls was lower than in heat-treated specimens in both poplar wood species (Figure 2). In fact, the reduction of this enzyme in controls compared to thermo-wood indicates greater consumption and effectiveness of this enzyme in untreated specimens. As heat treatment reduces the amount of hemicellulose and holocellulose, hydroxyl groups, which are an active site for chemical reactions, are reduced. As a result, the activity and effectiveness of laccase enzyme produced by brown rot fungi in modified wood is reduced (Gaff *et al.,* 2019; Mburu *et al.,* 2007; Wentzel *et al.,* 2019). Faraz *et al.* (2003) reported that, when *Eucalyptus grandis* wood chips were exposed to *Ceriporiopsis subvermisporaon*, the greatest mass loss was directly associated with a significant reduction of laccase enzyme, which supports the results of this study.

3.2. Mass loss caused by fungi (*MLF*)
3.2. Gubitak mase zbog djelovanja gljiva (*MLF*)

The results revealed that heat treatment had a significant effect on the decay action of brown rot fungi, leading to mass loss (Table 1). The *MLF* in thermo-wood specimens were significantly lower than in control specimens. This indicates the inhibition of heat

Table 1 Summarized statistical results of One-Way ANOVA for physical and mechanical properties of treated poplar wood
Tablica 1. Sažeti statistički rezultati jednosmjerne ANOVA-e za fizička i mehanička svojstva toplinski modificirane topolovine

S.V	Wood species *Vrste drva*	**After thermal modification** *Nakon toplinske modifikacije*			**After fungal degradation** *Nakon razgradnje uzrokovane gljivama*			
		Density **Gustoća**	**Compression strength parallel to grain** *Čvrstoća na tlak paralelno s vlakancima*	**Impact strength** *Čvrstoća na udarac*	**Mass loss** *Gubitak mase*	**Density** *Gustoća*	**Compression strength parallel to grain** *Čvrstoća na tlak paralelno s vlakancima*	**Impact strength** *Čvrstoća na udarac*
Heat treatment *toplinska modifikacija*	*Populus deltoids*	0.008**	0.017*	0.025*	0.020*	0.284 ns	0.122 ns	0.016*
	Populus nigra	0.024*	0.231 ns	0.028*	0.001**	0.386 ns	0.200 ns	0.034*

ns, * and ** – Non-significant and significant at 5 % and 1 % probability levels, respectively / *nije značajno i značajno je pri razinama vjerojatnosti od 5 %, odnosno 1 %*

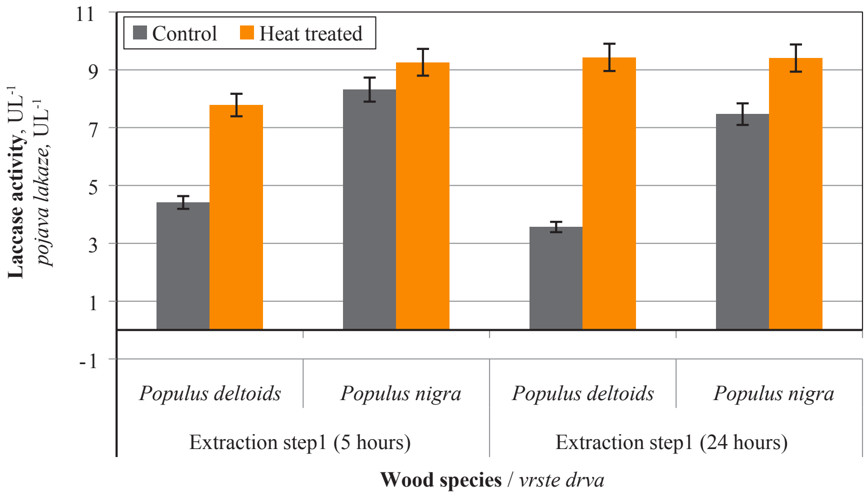

Figure 2 Laccase activity in heat-treated and control wood specimens during decay by *Coniophora puteana*
Slika 2. Pojava lakaze u toplinski modificiranim i kontrolnim uzorcima drva tijekom truljenja uzrokovanoga gljivom *Coniophora puteana*

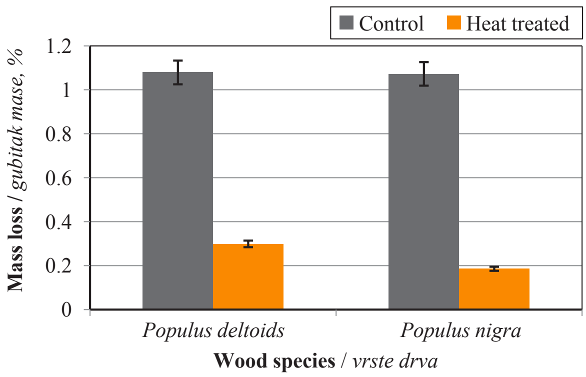

Figure 3 Effect of thermal modification on MLF in two wood species
Slika 3. Utjecaj toplinske modifikacije na MLF za dvije vrste drva

treatment on fungal activity (Figure 3). As indicated in Figure 3, the repellent effect of thermal modification on MLF obtained 72.28 and 82.69 % in *P. deltoids* and *P. nigra*, respectively. On the other hand, as shown in Figure 2, laccase activity in heat-treated specimens is greater than in controls, which is a sign of thermo-wood resistance against fungal degradation. Considering the substrate-enzyme interactions, it seems that in the control specimens, laccase enzyme had more interaction with the substrate and therefore the mass in these specimens was reduced to a greater extent compared to the treated specimens. According to the results of this study, there are other reports that show the deep adsorption of laccases in substrates suitable for their degradation (Tu *et al.*, 2009). Moreover, improvement of bio-durability of thermo-wood against fungal degradation is related to the reduction of hydroxyl groups due to heat treatment (Mburu *et al.,* 2007).

3.3. Density
3.3. Gustoća

This physical property was evaluated as follows.

3.3.1 After thermal modification
3.3.1. Nakon toplinske modifikacije

The results showed that heat treatment had a significant effect on the density (Table 1), so that the thermo-wood process reduced the density of both wood species (Figure 4). The reduction of density due to heat treatment in *P. deltoids* and *P. nigra* was 8.81 and 5.17 %, respectively. The change of wood chemical structure at high temperature causes mass loss, which leads to density decrease. The reduction of density due to heat treatment is related to destruction and change of chemical structure of wood, which intensifies at high temperatures (Militz, 2002).

3.3.2 After fungal degradation
3.3.2. Nakon razgradnje gljivama

After density assessment of control and heat-treated specimens in section 3.3.1, these specimens were exposed to brown rot fungus *C. puteana.* The results

Table 2 Average reduction values of physical and mechanical properties due to fungal degradation
Tablica 2. Prosječne redukcijske vrijednosti fizičkih i mehaničkih svojstava zbog razgradnje gljivama

Property *Svojstvo*	**Density** / *Gustoća, %*		**Compression strength parallel to grain, %** *Čvrstoća na tlak paralelno s vlakancima, %*		**Impact strength, %** *Čvrstoća na udarac, %*	
Wood species / *vrste drva*	*P. deltoids*	*P. nigra*	*P. deltoids*	*P. nigra*	*P. deltoids*	*P. nigra*
Control / *kontrolni uzorak*	3.27	5.94	6.04	11.71	4.71	7.45
Thermo-wood / *toplinski tretirano drvo*	2.23	5.52	3.69	9.28	6.41	21.88

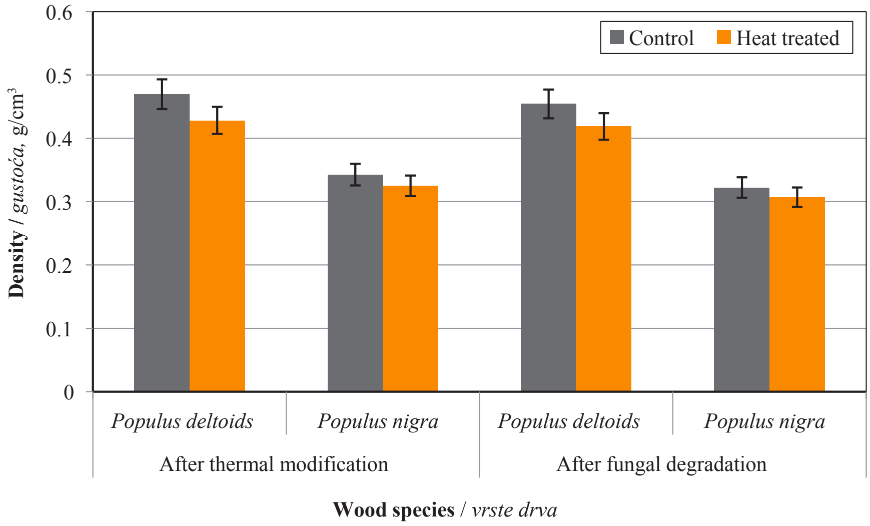

Figure 4 Effect of thermal modification on wood density and its variation under fungal attack
Slika 4. Utjecaj toplinske modifikacije na gustoću drva i promjene gustoće zbog napada gljiva

showed that heat treatment had no significant effect on fungal functionality in reduction of density in both poplar wood species (Table 1). As shown in Figure 4, the density difference between the control and thermo-wood specimens is little in both poplar species. However, heat treatment has been able to reduce the activity of brown rot fungus in both wood species, so that the density reduction due to fungal action in thermo-wood specimen was less than in controls (Table 2). This indicates that heat treatment has a repellent effect on fungal activity.

3.4. Compression strength parallel to grain
3.4. Čvrstoća na tlak paralelno s vlakancima

This mechanical property was evaluated as follows.

3.4.1 After thermal modification
3.4.1. Nakon toplinske modifikacije

The results showed that thermal modification had no significant effect on the compression strength parallel to the grain of *P. nigra* but it did in *P. deltoids* (Table 1). However, as indicated in Figure 5, heat treatment has improved this mechanical property in both wood species, so that the value of compression strength parallel to the grain in *P. deltoids* and *P. nigra* was 27.46 and 11.21 %, respectively (Figure 5). As a matter of fact, wood chemical structure changes at high temperature during thermal modification. Consequently, heat treatment causes cross-linking of lignin as well as increasing of lignin and cellulose values in wood structure, which leads to the improvement of compression

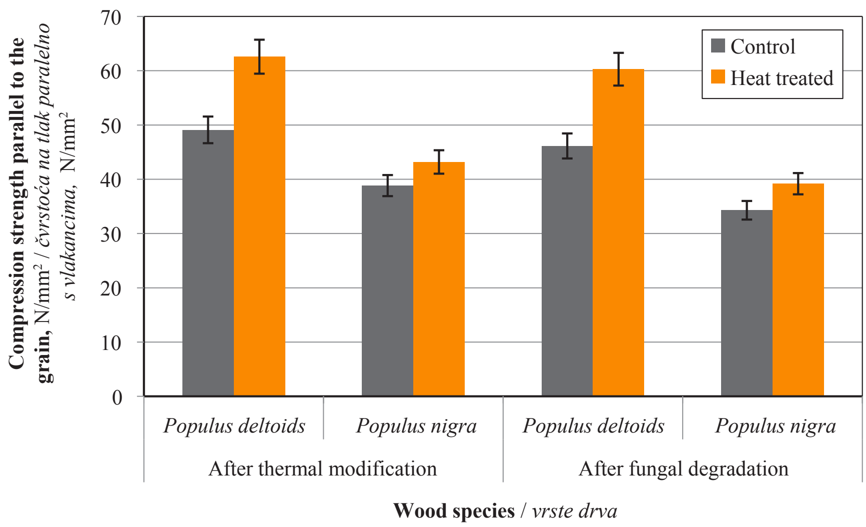

Figure 5 Effect of thermal modification on compression strength parallel to grain and its variation under fungal attack
Slika 5. Utjecaj toplinske modifikacije na čvrstoću na tlak paralelno s vlakancima i promjene čvrstoće zbog napada gljiva

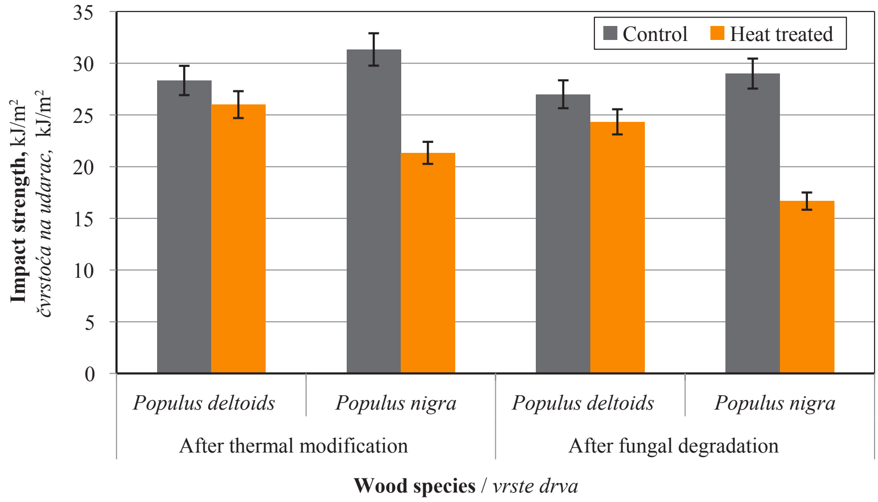

Figure 6 Effect of thermal modification on impact strength and its variation under fungal attack
Slika 6. Utjecaj toplinske modifikacije na čvrstoću na udarac i promjene te čvrstoće zbog napada gljiva

strength parallel to the grain. In other words, the change of wood chemical components due to the heat treatment is an effective agent for changing physical and mechanical properties of wood (Gaff *et al.,* 2019; Wentzel *et al.,* 2019).

3.4.2 After fungal degradation
3.4.2. Nakon razgradnje gljivama

The results revealed that heat treatment had no significant effect on fungal efficiency in terms of compression strength parallel to the grain (Table 1). However, comparison of the results in Figure 5 shows that this kind of modification was able to reduce the activity of brown rot fungi in both wood species. Consequently, the reduction of compression strength parallel to the grain due to fungal activity in thermo-wood specimen was less than that in the control, which indicates a repellent effect of heat treatment on fungal activity (Table 2). As shown in Figure 2, laccase activity in heat-treated specimens was more than that in the control group, which indicates bio-durability of thermo-wood. Heat treatment of wood at high temperatures reduces hydroxyl groups, which leads to the reduction of enzymatic hydrolysis (Mburu *et al.,* 2007).

3.5. Impact strength
3.5. Čvrstoća na udarac

This mechanical property was evaluated as follows:

3.5.1 After thermal modification
3.5.1. Nakon toplinske modifikacije

The results revealed that heat treatment had a significant effect on impact strength of both wood species (Table 1). Thermal modification had negative effect on this mechanical property in both species, as shown in Figure 5. The reduction of impact strength was 8.24 and 31.91 % in *P. deltoids* and *P. nigra*, respectively (Figure 6). The reduction of impact strength of thermo-wood might be related to brittle structure of heat-treated specimens. In fact, the reduction of this mechanical property is directly related to chemical structure changes, while mass loss of specimens occurs due to heating (Kaygin *et al.,* 2009).

3.5.2 After fungal degradation
3.5.2. Nakon razgradnje gljivama

The results showed that heat treatment had a significant effect on fungal action in terms of impact strength (Table 1). Although thermal modification can reduce the activity of the brown rot fungi, the reduction of impact strength in thermo-wood exposed to brown rot fungi was greater than in control group (Table 2). The effect of heat treatment on structural changes of wood as well as destruction of cellulose and holocellulose by brown rot fungi can be effective agents in the reduction of impact strength of thermo-wood specimens.

4 CONCLUSIONS
4. ZAKLJUČAK

The results of the present study revealed that the degradation of two poplar wood species (*P. deltoids* and *P. nigra*) by *C. puteana* could be affected by thermal modification. Although the activity of *C. puteana* and laccase production occurred in both control and heat-treated specimens, the lowest detectable laccase levels were observed in controls, coinciding with the highest mass loss. Considering the substrate-enzyme interactions, it seems that in the control specimens, laccase enzyme had more interaction with the substrate and therefore the mass in these specimens was reduced to a greater extent compared to the treated specimens. Therefore, the result of laccase activity assessment also showed that the laccase value in controls was less than that in modified specimens. Overall, the results revealed that thermal modification improves bio-durability of both poplar wood species; however, it reduces some mechanical properties. On the other hand, this kind of modification prevents the reduction of mechanical properties due to biological degradation.

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Influence of Pre-Impregnation Acetic Anhydride and Heating Time on Mechanical and Physical Properties of Wood-Plastic Composites

Utjecaj predimpregnacije anhidridom octene kiseline i vremena zagrijavanja na mehanička i fizička svojstva drvno-plastičnih kompozita

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ABSTRACT • *This research aimed to assess the influence of different pre-impregnation times (PITs) (60, 180, and 300 min), heating or reaction times (H/RTs) (60, 90, and 120 min), and chemical modification of wood flour (WF) on the mechanical and physical properties of wood-plastic composites (WPCs). The study employed acetylated beech (Fagus orientalis L.) flour as the filler and polypropylene (PP) as the matrix phase producing of WPC samples through melt compounding and injection molding. The resulting composites underwent testing for their physical and mechanical properties. The findings revealed that WPCs derived from acetylated wood with PITs of 60 min and H/RTs of 60 min exhibited the highest mechanical properties, except for the bending modulus. Moreover, the lowest water absorption (WA) was observed in the PITs-H/RTs combination of 60-120 min, while the lowest thickness swelling (TS) occurred in the PITs-H/RTs combination of 300-60 min. The simultaneous utilization of pre-impregnation and reaction times demonstrated a synergistic effect on the physical and mechanical properties. Consequently, the chemical modification of wood flour and the application of suitable reaction times improved the interfacial adhesion, thereby enhancing the overall performance of the WPCs.*

KEYWORDS: *WPCs; wood flour; acetylation; chemical modification; mechanical properties*

SAŽETAK • *Cilj ovog istraživanja bio je procijeniti utjecaj različitih vremena predimpregnacije (PIT) (60, 180 i 300 min), vremena zagrijavanja (H/RT) (60, 90 i 120 min) i kemijske modifikacije drvnog brašna (WF) na mehanička i fizička svojstva drvno-plastičnih kompozita (WPC). U istraživanju je za proizvodnju uzoraka WPC-a taljenjem i injekcijskim prešanjem upotrijebljeno drvno brašno acetilirane bukovine (Fagus orientalis L.) kao punilo i polipropilen (PP) kao matrica. Zatim su ispitana fizička i mehanička svojstva dobivenih kompozita. Rezultati su*

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pokazali da drvno-plastični kompoziti dobiveni od acetiliranog drva s vremenom predimpregnacije i zagrijavanja od 60 min imaju najbolja mehanička svojstva, osim modula na savijanje. Nadalje, najmanje upijanje vode (WA) zabilježeno je za kombinaciju PIT-H/RT 60-120 min, dok je najmanje debljinsko bubrenje (TS) izmjereno za kombinaciju PIT-H/RT 300-60 min. Istodobno je primjena predimpregnacije i zagrijavanja pokazala sinergijski utjecaj na fizička i mehanička svojstva. Posljedično su kemijska modifikacija drvnog brašna i odabir odgovarajućih vremena zagrijavanja poboljšali adheziju između faza, a time i svojstva drvno-plastičnih kompozita.

KLJUČNE RIJEČI: *WPC; drvno brašno; acetilacija; kemijska modifikacija; mehanička svojstva*

1 INTRODUCTION
1. UVOD

Chemical modification is considered an active approach as the transformation occurs in the macromolecules of the wood cell wall, utilizing one or more chemicals that react to form a material integrated into the wood cell wall structure (Rowell, 2005). This alteration of the cell wall macromolecules in solid wood leads to a reduction in dimensional stability and moisture content, while enhancing resistance to biological degradation (Hill, 2006; Lyon *et al.*, 2008; Ermeydan, 2014; Thybring and Fredriksson, 2021; Zhou and Liu, 2022). A prominent challenge arises when hydrophilic lignocellulosic fibers are used as reinforcements in a hydrophobic synthetic matrix, as the lack of compatibility between the two materials results in disparate behavior and poor adhesion (Özmen *et al.*, 2013a).

The properties and performance of wood and wood-based composites were improved by chemical modification, which is defined as chemical reaction between some reactive site of a lignocellulose material and a simple single chemical reagent, with or without catalyst, to form a covalent bond between them. Acetylation is a reaction that leads to a change in the chemical and physical properties of the wood substrate and results in the substitution of hydrophilic hydroxyl groups with hydrophobic acetyl groups (Rowell, 2006a, 2006b; Rowell, 2007; Bodîrlău *et al.*, 2012).

The poor compatibility between the reinforcing material and the matrix contributes to a decline in the mechanical properties of the composite (Özmen *et al.*, 2013b; Kallakas *et al.*, 2015). To address this issue and enhance the interfacial adhesion between the polymer matrix and wood flour (WF), various chemical modifications such as alkaline treatment, acetylation, epoxidation, benzoylation, silane treatment, or other WF modifications have been employed (Rowell, 2006a; Farsi, 2010; Gwon *et al.*, 2010; Tesinova, 2011; Teacă *et al.*, 2014; Kallakas *et al.*, 2015; de Prá Andrade and Poletto, 2021).

Chemical modification of *Abies alba* L. softwood samples was conducted by succinic anhydride (SA) and maleic anhydride (MA) reagents in the presence of xylene as solvent (reaction time 1 h and 90 °C), where MA exhibited lower reactivity towards wood than SA, presumably due to chemical structure (Teacă *et al.*, 2014).

The conventional acetylation process involves the impregnation of dried wood with liquid-phase acetic anhydride, followed by the application of external heat (Hung *et al.*, 2016). Acetylation of wood is a widely used method for modifying wood with acetic anhydride, which leads to the esterification of accessible hydroxyl groups in the cell wall with acetyl groups (Figure 1).

Larsson-brelid *et al.* (2006) conducted a study on WPCs using acetic anhydride-modified solid wood. Previous research has demonstrated that WPC materials reinforced with acetylated wood fibers exhibit improved mechanical properties, reduced moisture content, and enhanced resistance to brown-rot decay compared to unmodified WPC materials (Abdul Khalil *et al.*, 2002; Segerholm *et al.*, 2005).

The properties of acetylated wood are influenced by the specific method of acetylation. Factors such as reaction temperature, reaction time, type of catalysts, and their quantities significantly affect the extent of fiber degradation during the treatment process (Rowell, 1983). The weight percent gain (*WPG*) increases as the reaction temperature and time increase. For instance, in the chemical modification of pine wood using various chemicals, such as liquid acetic anhydride, the *WPG* of acetylated pine wood increases with higher temperature values (from 100 °C to 160 °C) and longer reaction times (from 60 min to 300 min) (Rowell, 1983).

This study aimed to investigate the mechanical and physical properties of polypropylene composites (WPCs) reinforced with acetylated and unmodified beech WF. Three different pre-impregnation times (*PITs*) and three different heating or reaction times (*H/RTs*) were evaluated during the acetylation process using acetic anhydride. The obtained results were compared with those of unmodified WPCs.

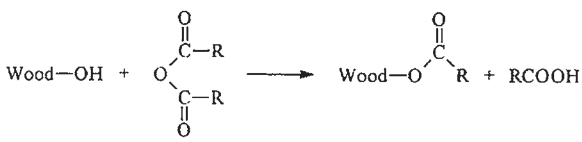

Figure 1 Anhydride modification scheme, where R = CH_3 (acetic anhydride) (Papadopoulos *et al.*, 2019)
Slika 1. Shema modifikacije anhidrida, pri čemu je R = CH_3 (anhidrid octene kiseline) (Papadopoulos *et al.*, 2019.)

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

Beech wood (*Fagus orientalis* L.) was sourced from the Kelardasht forest in northern Iran. The wood was initially cut into small pieces and then further reduced in size using a hammer mill. The resulting WF had a particle size ranging from 0.25 mm to 0.42 mm. Before impregnation and reaction processes, the wood samples were subjected to oven drying at (103±2) °C for 24 hours. Subsequently, the samples were allowed to cool to room temperature in a desiccator containing phosphorous pentoxide before their weight was determined. The moisture content of WF before reaction process was 0 %. Oven-dried WF was stored in sealed plastic bags.

Polypropylene (PP) with a melt flow index (MI) of 10 g/10 min and a density of 0.95 g/cm^3 was supplied by the Tabriz Petrochemical Company in Iran.

The coupling agent used in this study was maleated polypropylene (MAPP), obtained from Eastman Chemical Products, Inc.; as Epolene G-3003TM polymer with 8 % acid anhydride and a molecular weight of 103,500 (Nourbakhsh and Ashori, 2008).

Acetic anhydride (AA), a reagent required for the acetylation process, was purchased from Merck Company in Germany. Its molecular weight, purity, density, and melting point were 102.1 g/mol, 96 %, 1.08 g/cm^3, and -73.1 °C, respectively (Kown and Nadir, 2015).

2.2 Acetylation of beech wood flour
2.2. Acetilacija drvnog brašna bukovine

A total of 500 g of oven-dried beech wood flour was placed in 2-L Erlenmeyer flasks along with acetic anhydride (96 %) for pre-impregnation. Catalysts were not used during the pre-impregnation process, which took place at room temperature for 60, 180, or 300 minutes as pre-impregnation times (*PITs*). The acetic anhydride treatment of the WF was conducted through a reaction at (103±2) °C for 60, 90, or 120 minutes as heating or reaction times (*H/RTs*). These reaction times were determined based on previous studies by Serin (2005), Dizman (2005), and Cavdar *et al.* (2014), respectively. After the reaction, the WF was thoroughly washed with semi-warm deionized water to remove any residual chemicals and by-products. The acetylated beech WF was then dried in an oven at (103±2) °C for 24 hours. Subsequently, it was placed in a desiccator containing phosphorous pentoxide until it reached room temperature, and the weight percentage gain (*WPG*) was determined using Eq. 1:

$$WPG\ (\%) = [(W2 - W1)/W1] * 100 \qquad (1)$$

In the equation, *W1* represents the weight of the sample before treatment, and *W2* represents the weight of the sample after treatment.

2.3 Production of WPCs
2.3. Proizvodnja WPC-a

The composition of acetic anhydride-treated WF/PP composites and the corresponding weight percentage gain (*WPG*) are presented in Table 1.

Before the sample preparation, both unmodified and modified beech wood flours were subjected to drying in an oven at a temperature of (103±2) °C for 24 hours. The mixing of the components was conducted using a Hake internal mixer (HBI System 90, USA) at a temperature of 180 °C and a rotation speed of 60 rpm.

The process involved feeding the PP into the mixing chamber, followed by the addition of the coupling agent (PP-g-MA) once the PP had melted. After five minutes, the WF was introduced, and the total mixing time was 11 minutes. The resulting mixture was then ground using a pilot scale grinder (Wieser, WGLS 200/200 Model, Germany). The obtained granules were subsequently dried at a temperature of 70 °C for 24 hours. Test samples were prepared by injection

Table 1 Procedure of chemical modification of wood flour
Tablica 1. Postupak kemijske modifikacije drvnog brašna

WPC or Treatment code *WPC ili oznaka tretmana*	**WF, wt %**	**PP, wt %**	**MAPP, wt %**	***PITs*, min**	***H/RTs*, min**
*PITs*60-*H/RTs*60	60	40	2	60	60
*PITs*60- *H/RTs*90	60	40	2	60	90
*PITs*60- *H/RTs*120	60	40	2	60	120
*PITs*180- *H/RTs*60	60	40	2	180	60
*PITs*180- *H/RTs*90	60	40	2	180	90
*PITs*180- *H/RTs*120	60	40	2	180	120
*PITs*300- *H/RTs*60	60	40	2	300	60
*PITs*300- *H/RTs*90	60	40	2	300	90
*PITs*300- *H/RTs*120	60	40	2	300	120
Control	60	40	2	-	-

PITs – Pre-impregnation times, *H/RTs* – Heating or reaction times, WF – Wood flour, PP – Polypropylene, MAPP – Maleic anhydride polypropylene / *PITs – vrijeme predimpregnacije, H/RTs – vrijeme zagrijavanja ili reakcije, WF – drvno brašno, PP – polipropilen, MAPP – anhidrid maleinske kiseline – polipropilen*

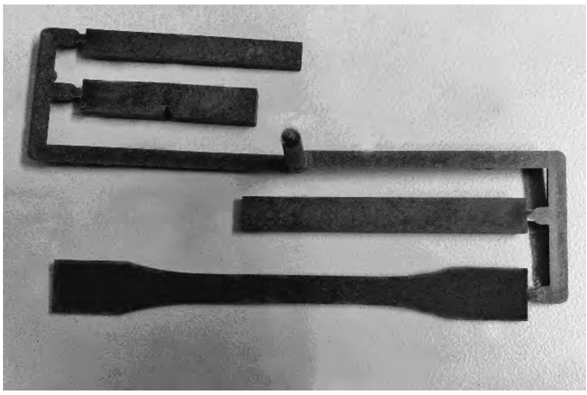

Figure 2 Mechanical testing samples
Slika 2. Uzorci za mehanička ispitivanja

molding according to the ASTM D 3641 (2015) standard using an injection molding machine at a temperature of 185 °C and a pressure of 10 MPa (Iman machine, Iran). Finally, the specimens were stored in controlled conditions with a relative humidity of 50 % and a temperature of 23 °C for at least 40 hours prior to testing. A sample product ready for mechanical testing is shown in Figure 2. Three replicated samples were tested for each property at each treatment level.

2.4 Measurements
2.4. Mjerenja

2.4.1 Mechanical properties
2.4.1. Mehanička svojstva

Flexural and tensile tests were carried out in accordance with ASTM D 790 (2007) and D 638 (2007) standards, respectively, using an Instron machine (Model 1186, England). The tests were performed at a crosshead speed of 5 mm/min. For the Izod impact test, a Zwick impact tester (Model SIT 20 D, Santam Co., Iran) was employed. To prepare the samples, a notch was made at the center of one longitudinal side, in accordance with the specifications provided in ASTM D 256 (2007).

2.4.2 Physical properties
2.4.2. Fizička svojstva

Water absorption (*WA*) measurements were conducted following the guidelines outlined in ASTM D 570-98 (2010) standard. To determine the water absorption of the acetylated wood-plastic composite (WPC) specimens, three specimens of each WPC type were first dried in an oven at (103±2) ºC for 24 hours. The dried specimens were weighed with a precision of 0.001 g and then immersed in distilled water at room temperature for a period of 24 hours. After the immersion time elapsed, the specimens were carefully removed from the water and any excess moisture on the surface was removed using blotting paper. The weight of the specimens was then measured again after 24 hours of immersion in water at room temperature. The mean percentages of water absorption (*WA*) and thickness swelling (*TS*) over time were calculated for each composite (both modified and unmodified) and for each of the 10 formulations using Eqs. 2 and 3 as follows:

$$WA\,(t)\,\% = [(Wt - Wo) / Wo] \times 100 \tag{2}$$

$$TS\,(t)\,\% = [(Tt - To) / To] \times 100 \tag{3}$$

Where *WA* (t) represents the water absorption at time *t*, Wo is the initial weight of the specimens, *Wt* is the weight of the specimens at time *t*, *TS* (*t*) denotes the thickness swelling at time *t*, *To* is the initial thickness of the specimens, and *Tt* is the thickness of the specimens at time *t*.

2.5 Statistical analysis
2.5. Statistička analiza

The statistical analysis was performed using the SPSS software (Version 24) through the implementation of a general linear model (Univariate). To understand the combined effect of *PITs* (min) and *H/RTs* (min), a two-way ANOVA (analysis of variance) technique was used in the experiments. To assess the statistical significance at a significance level of $p<0.05$, the Duncan multiple range test was employed.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Weight percent gain
3.1. Povećanje mase

The result of the reaction between beech wood flour hydroxyl groups and acetic anhydride (AA), in other words the weight percentage gain of the acetic anhydride chemically modified WF is presented in Figure 3.

The individual effects of pre-impregnation times and heating/reaction times on the weight percent gain (*WPG*) of PP composites filled with AA-modified WF are summarized in Table 2.

Statistically, results showed that the interaction between the variables *PITs* and *H/RTs* was negative and had a significant effect on the weight percent gain of samples.

According to a statistical analysis, the individual effect of variables *PITs* and *H/RTs* showed significant effects on the *WPG* measured. The interaction between *PITs* and *H/RTs* was negative and indicated a significant effect on the *WPG* factor within the range of 95 % confidence for the experimental WPC samples made from AA-treated wood flour investigated.

The highest (7.18 %) and lowest (0.69 %) *WPG* levels were achieved for 300-120 min and 180-60 min of *PITs-H/RTs*, respectively (Figure 3).

Table 2 Individual effect of pre-impregnation times and heating/reaction times on weight percent gain of PP composites filled with AA-modified WF
Tablica 2. Individualni utjecaj vremena predimpregnacije i vremena zagrijavanja/reakcije na povećanje mase PP kompozita ispunjenih AA modificiranim WF-om

	Pre-impregnation times / *Vremena predimpregnacije*		
Property / *Svojstvo*	60 min	180 min	300 min
Weight percent gain, % / *povećanje mase,* %	1.48	3.11	4.31
	Heating/reaction times / *Vremena zagrijavanja/reakcije*		
Property / *Svojstvo*	60 min	90 min	120 min
Weight percent gain, % / *povećanje mase,* %	1.04	3.98	3.92

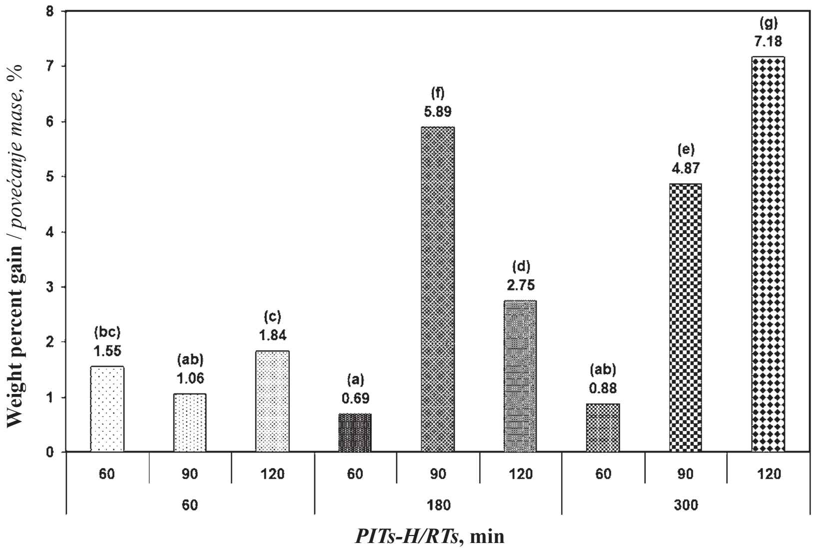

Figure 3 Interaction effect of pre-impregnation (*PITs*) and heating or reaction times (*H/RTs*) on weight percent gain of PP composites filled with AA-modified WF; Groups with the same letters in each row indicated no statistical difference (p < 0.05) between samples according to Duncan's multiple range test
Slika 3. Utjecaj interakcije predimpregnacije (*PITs*) i vremena zagrijavanja ili reakcije (*H/RTs*) na povećanje mase PP kompozita ispunjenih AA modificiranim WF-om; prema Duncanovu testu, grupe s istim slovima u svakom stupcu nisu pokazale statistički značajnu razliku (p < 0,05) među uzorcima

3.2 Mechanical properties
3.2. Mehanička svojstva

The mechanical properties of PP composites filled with acetic anhydride WF and Duncan's multiple range test results are shown in Figures 4, 5, 6, 7, and 8.

The individual effects of pre-impregnation times and heating/reaction times on the mechanical properties of PP composites filled with AA-modified WF are summarized in Tables 3 and 4, respectively.

3.2.1 Flexural strength
3.2.1. Čvrstoća na savijanje

Statistically, it was shown that the interaction between the variables *PITs* and *H/RTs* was negative and exhibited a significant effect on the flexural strength

Table 3 Individual effect of pre-impregnation times on mechanical properties of PP composites filled with AA-modified WF
Tablica 3. Individualni utjecaj vremena predimpregnacije na mehanička svojstva PP kompozita ispunjenih AA modificiranim WF-om

Mechanical properties *Mehanička svojstva*	**Pre-impregnation times** / *Vremena predimpregnacije*		
	60 min	180 min	300 min
Flexural strength, MPa / *čvrstoća na savijanje,* MPa	55.68 (2.4) [b]	52.02 (1.3) [a]	52.46 (3.4) [a]
Flexural modulus, MPa / *modul savijanja,* MPa	4648 (2262) [a]	5996 (417) [a]	4958 (2239) [a]
Tensile strength, MPa / *vlačna čvrstoća,* MPa	35.73 (4.1) [b]	29.87 (2.2) [a]	29.66 (4.2) [a]
Tensile modulus, MPa / *modul elastičnosti pri vlačnom naprezanju,* MPa	5561 (2387) [b]	4373 (1348) [ab]	3285 (1400) [a]
Impact strength, J/m / *čvrstoća na udarac,* J/m	0.42 (0.1) [a]	0.41 (0.1) [a]	0.38 (0.0) [a]

Groups with the same letters in each row indicated no statistical difference (p < 0.05) between samples according to Duncan's multiple rang test. The values in parentheses are standard deviations.
Prema Duncanovu testu, grupe s istim slovima u svakom retku nisu pokazale statistički značajnu razliku (p < 0,05) među uzorcima. Vrijednosti u zagradama standardna su odstupanja.

Table 4 Individual effect of heating/reaction times on mechanical properties of PP composites filled with AA-modified WF
Tablica 4. Individualni utjecaj vremena zagrijavanja/reakcije na mehanička svojstva PP kompozita ispunjenih AA modificiranim WF-om

Mechanical properties *Mehanička svojstva*	**Heating/reaction times** / *Vremena zagrijavanja/reakcije*		
	60 min	90 min	120 min
Flexural strength, MPa / *čvrstoća na savijanje,* MPa	53.55 (3.4) [a]	52.92 (3.0) [a]	53.69 (2.7) [a]
Flexural modulus, MPa / *modul savijanja,* MPa	4007 (2376) [a]	5334 (1657) [ab]	6261 (381) [b]
Tensile strength, MPa / *vlačna čvrstoća,* MPa	33.26 (4.5) [a]	30.03 (3.1) [a]	31.97 (5.4) [a]
Tensile modulus, MPa / *modul elastičnosti pri vlačnom naprezanju,* MPa	5351 (1907) [b]	3655 (2491) [a]	4214 (930) [ab]
Impact strength, J/m / *čvrstoća na udarac,* J/m	0.40 (0.1) [a]	0.42 (0.0) [a]	0.39 (0.0) [a]

Groups with the same letters in each row indicated no statistical difference ($p < 0.05$) between samples according to Duncan's multiple rang test. The values in parentheses are standard deviations.
Prema Duncanovu testu, grupe s istim slovima u svakom retku nisu pokazale statistički značajnu razliku ($p < 0,05$) među uzorcima. Vrijednosti u zagradama standardna su odstupanja.

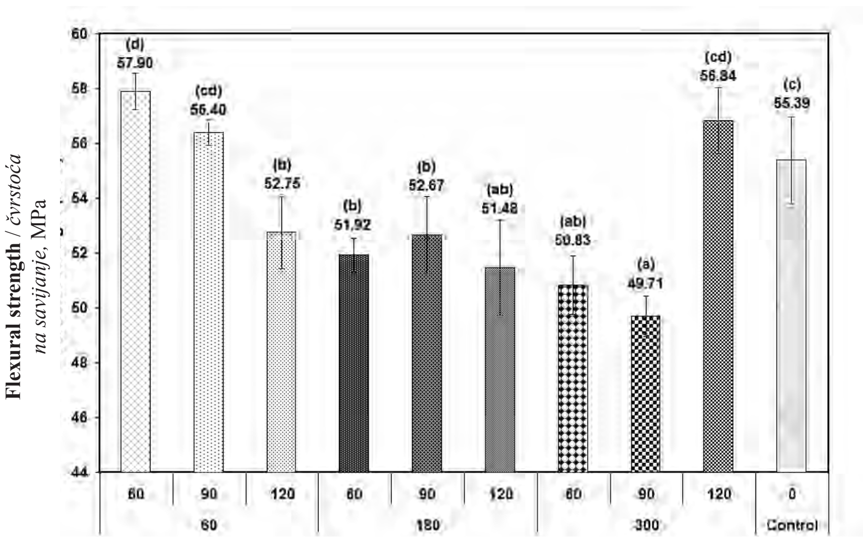

Figure 4 Interaction effect of pre-impregnation (*PITs*) and heating or reaction times (*H/RTs*) on flexural strength of PP composites filled with AA-modified WF; Groups with the same letters in each column indicated no statistical difference ($p < 0.05$) between samples according to Duncan's multiple range test
Slika 4. Utjecaj interakcije predimpregnacije (*PITs*) i vremena zagrijavanja ili reakcije (*H/RTs*) na čvrstoću na savijanje PP kompozita ispunjenih AA modificiranim WF-om; prema Duncanovu testu, grupe s istim slovima u svakom stupcu nisu pokazale statistički značajnu razliku ($p < 0,05$) među uzorcima

(*FS*) of WPC samples. According to a statistical analysis, the individual effect of variable *PITs* showed a significant effect on the *FS* measured (Table 3), while the individual effect of variable *H/RTs* had no significant effect on the *FS* measured (Table 4). The interaction between *PITs* and *H/RTs* was negative and showed a significant effect on the *FS* factor within the range of 95% confidence for the experimental WPC samples made from AA-treated wood flour investigated (Figure 4).

The test results showed that the *PITs* and *H/RTs,* as reaction variables of chemical modification with AA, affected the mechanical and physical properties of the composite samples. Flexural strength (*FS*) values of samples increased with the decrease in pre-impregnation times considering the individual effect of *PITs*, while the decreases were not significant considering the individual effect of *H/RTs*. The *FS* values were determined to be between 55.39 MPa (control) and 57.9 MPa of the samples produced with WF modification at 60 minutes of *PITs* (Figure 4).

The greatest increase in FS was found in the samples modified with 60-60 min of *PITS-H/RTs* in the interaction effect of reaction variables (57.90 MPa) (Figure 4).

3.2.2 Flexural modulus
3.2.2. Modul savijanja

Statistically, it was shown that the interaction between the variables *PITs* and *H/RTs* was positive and had no significant effect on the flexural modulus (*FM*) of WPC samples (Figure 5).

According to a statistical analysis, the individual effect of variable *H/RTs* showed significant effect on the *FM* measured (Table 4), while the individual effect of variable *PITs* had no significant effect on the *FM* measured (Table 3). The interaction between *PITs* and

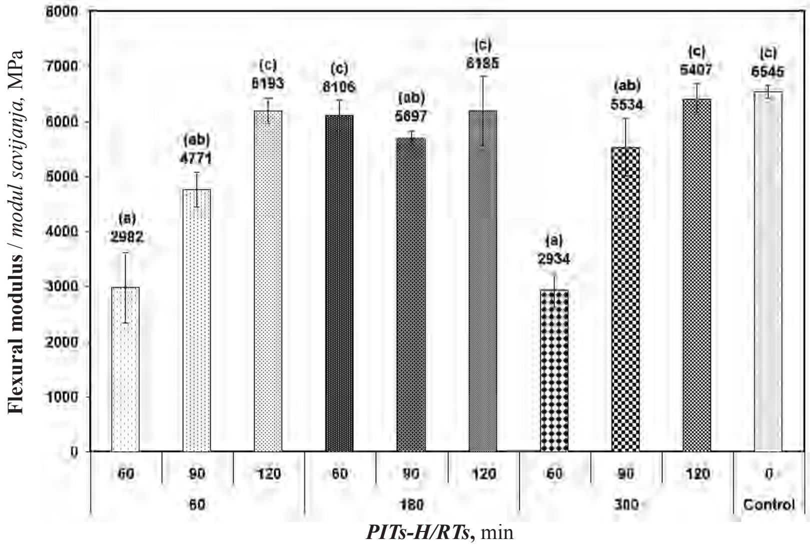

Figure 5 Interaction effect of pre-impregnation (*PITs*) and heating or reaction times (*H/RTs*) on flexural modulus of PP composites filled with AA-modified WF; Groups with the same letters in each column indicated no statistical difference ($p < 0.05$) between samples according to Duncan's multiple range test
Slika 5. Utjecaj interakcije predimpregnacije (*PITs*) i vremena zagrijavanja ili reakcije (*H/RTs*) na modul savijanja PP kompozita ispunjenih AA modificiranim WF-om; prema Duncanovu testu, grupe s istim slovima u svakom stupcu nisu pokazale statistički značajnu razliku ($p < 0,05$) među uzorcima

H/RTs was positive and showed no significant effect on the *FM* factor within the range of 95 % confidence for the experimental WPC samples made from AA-treated wood flour investigated (Figure 5).

The use of different heating or reaction times (*H/RTs*) in the chemical modification process with AA led to an increase in the flexural modulus (*FM*). Among the various *H/RTs* tested, the highest increase in *FM* was observed with 120-minute *H/RTs* (6261 MPa) as individual effect; however, there was a significant difference in *FM* values between the 60-minute and 120-minute *H/RTs*. The greatest value in *FM* was found in the unmodified or control samples (6545 MPa) (Figure 5).

The lowest values in *FS* and *FM* were found in the samples modified with 300-90 min (49.71 MPa) and 300-60 min (2934 MPa) of *PITS-H/RTs* in the interaction effect of reaction variables, respectively (Figures 4 and 5). Compared to the control group, the WPCs produced in 60-60 min and 300-120 min of *PITs-H/RTs* provided about 4% higher *FS* and 2% lower *FM* values, respectively.

3.2.3 Tensile strength
3.2.3. Vlačna čvrstoća

Statistically, it was shown that the interaction between the variables *PITs* and *H/RTs* was positive and had no significant effect on the tensile strength (*TS*) of WPC samples. According to a statistical analysis, the individual effect of variable *PITs* showed a significant effect on the *TS* measured (Table 3), while the individual effect of variable *H/RTs* had no significant effect on the *TS* measured (Table 4). The interaction between *PITs* and *H/RTs* was positive and showed no significant effect on the *TS* factor within the range of 95 % confidence for the experimental WPC samples made from AA-treated wood flour investigated (Figure 6).

The tensile strength (*TS*) values varied depending on the reaction variables, ranging from 29.62 MPa to 38.39 MPa in the control samples and samples produced by modifying WF in 60 min of *PITs* and 60 min of *H/RTs*, respectively (Figure 6).

The highest *TS* value was obtained from samples produced through a 60-minute pre-impregnation time (*PIT*). Remarkable changes were observed in *TS* values between the control samples and the modified ones.

3.2.4 Tensile modulus
3.2.4. Modul elastičnosti pri vlačnom naprezanju

Statistically, it was shown that the interaction between the variables *PITs* and *H/RTs* was negative and had a significant effect on the tensile modulus (*TM*) of WPC samples. According to a statistical analysis, the individual effect of variables *PITs* and *H/RTs* showed significant effects on the *TM* measured (Tables 3 and 4). The interaction between *PITs* and *H/RTs* was negative and showed a significant effect on the *TM* factor within the range of 95 % confidence for the experimental WPC samples made from AA-treated wood flour investigated (Figure 7).

In addition, the tensile modulus of the samples also exhibited an increase caused by variations in both reaction variables, namely *PITs* and *H/RTs*, during the acetylation of WF with AA. This increase in the *TM* can be attributed to the enhanced stiffness of WPCs (Figure 7). The increasing severity of pre-

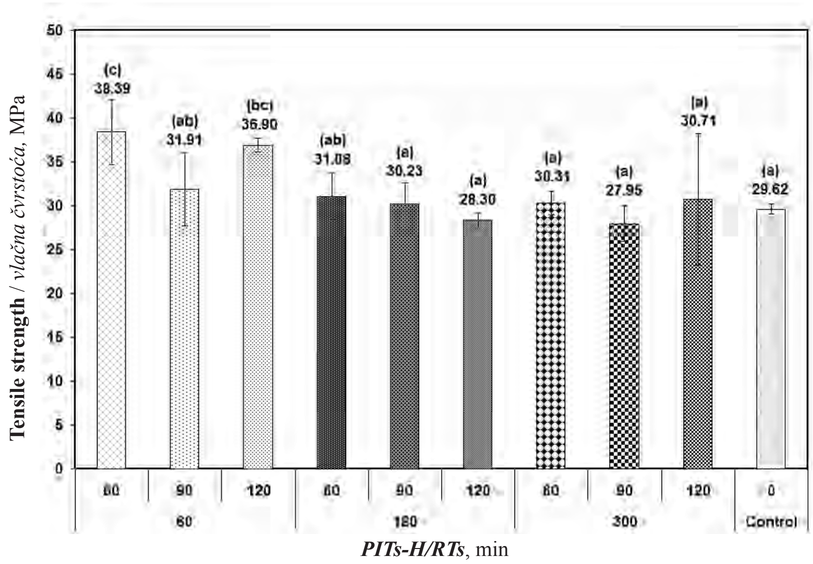

Figure 6 Interaction effect of pre-impregnation (*PITs*) and heating or reaction times (*H/RTs*) on tensile strength of PP composites filled with AA-modified WF; Groups with the same letters in each column indicated no statistical difference ($p < 0.05$) between samples according to Duncan's multiple range test
Slika 6. Utjecaj interakcije predimpregnacije (*PITs*) i vremena zagrijavanja ili reakcije (*H/RTs*) na vlačnu čvrstoću PP kompozita ispunjenih AA modificiranim WF-om; prema Duncanovu testu, grupe s istim slovima u svakom stupcu nisu pokazale statistički značajnu razliku ($p < 0,05$) među uzorcima

impregnation times and heating/reaction times negatively affected the tensile modulus and other mechanical properties.

3.2.5 Impact strength
3.2.5. Čvrstoća na udarac

Statistically, it was shown that the interaction between the variables *PITs* and *H/RTs* was positive and had no significant effect on the impact strength (*IS*) of WPC samples. According to a statistical analysis, the individual effect of variables *PITs* and *H/RTs* showed no significant effects on the *IS* measured (Tables 3 and 4). The interaction between *PITs* and *H/RTs* was positive and showed no significant effect on the IS factor within the range of 95 % confidence for the experimental WPC samples made from AA-treated wood flour investigated (Figure 8).

The impact strength (*IS*) values of samples increased with the decrease in pre-impregnation times in the individual effect of *PITs*, while the values did not

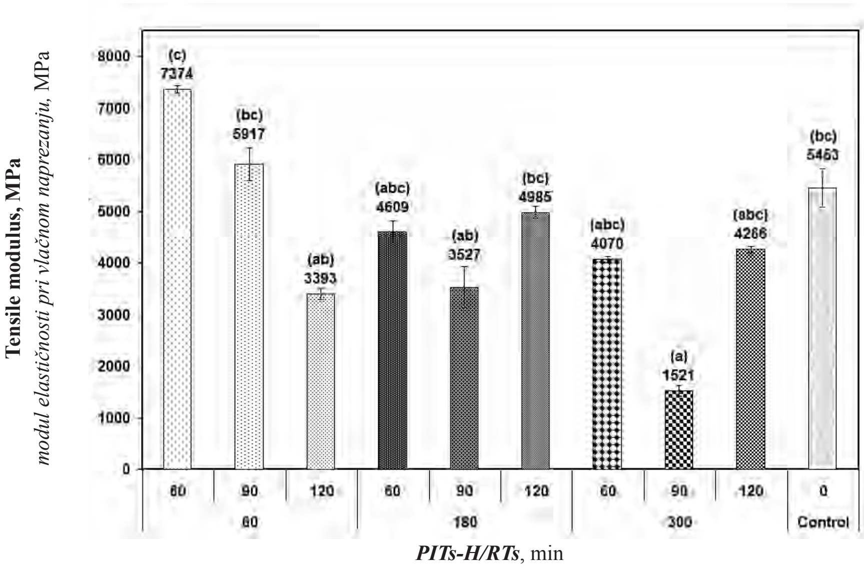

Figure 7 Interaction effect of pre-impregnation (*PITs*) and heating or reaction times (*H/RTs*) on tensile modulus of PP composites filled with AA-modified WF; Groups with the same letters in each column indicated no statistical difference ($p < 0.05$) between samples according to Duncan's multiple range test
Slika 7. Utjecaj interakcije predimpregnacije (*PITs*) i vremena zagrijavanja ili reakcije (*H/RTs*) na modul elastičnosti pri vlačnom naprezanju PP kompozita ispunjenih AA modificiranim WF-om; prema Duncanovu testu, grupe s istim slovima u svakom stupcu nisu pokazale statistički značajnu razliku ($p < 0,05$) među uzorcima

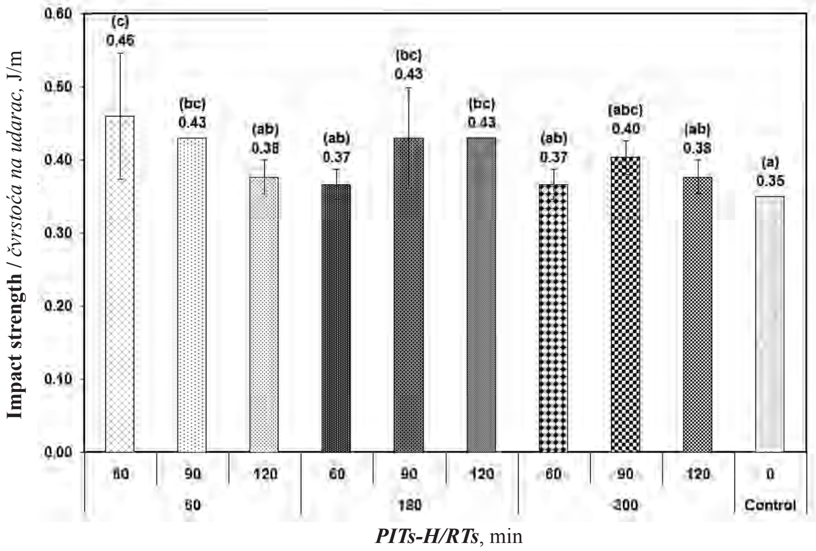

Figure 8 Interaction effect of pre-impregnation (*PITs*) and heating or reaction times (*H/RTs*) on impact strength of PP composites filled with AA-modified WF; Groups with the same letters in each column indicated no statistical difference ($p < 0.05$) between samples according to Duncan's multiple range test
Slika 8. Utjecaj interakcije predimpregnacije (*PITs*) i vremena zagrijavanja ili reakcije (*H/RTs*) na čvrstoću na udarac PP kompozita ispunjenih AA modificiranim WF-om; prema Duncanovu testu, grupe s istim slovima u svakom stupcu nisu pokazale statistički značajnu razliku ($p < 0,05$) među uzorcima

show significant changes for the individual effect of *H/RTs*. The *IS* values were found to be between 0.35 J/m (control) and 0.46 J/m from the samples produced by modifying WF in 60 min of *PITs* and 60 min of *H/RTs* (Figure 8).

Figure 4 clearly shows that the acetylation of beech WF with acetic anhydride had a positive effect on flexural strength, while the variation in the pre-impregnation and heating or reaction times had no positive effect on flexural modulus in comparison with unmodified or control samples (Figure 5).

The greatest increase in TS was found for samples modified with 60-60 min of *PITS-H/RTs* in the interaction effect of reaction variables (38.39 MPa) (Figure 6), and the greatest value in *TM* was also found in samples modified with 60-60 min of *PITS-H/RTs* in the interaction effect of reaction variables (7374 MPa) (Figure 7). The lowest values in *TS* and *TM* were found in samples modified with 300-90 min (27.95 MPa) and (1521 MPa) of *PITS-H/RTs* in the interaction effect of reaction variables, respectively. Compared to the control group, composites produced with 60-60 min of *PITs-H/RTs* WF provided about 22 % and 26 % higher *TS* and *TM* values, respectively (Figures 6 and 7).

The greatest increase in the *IS* was found in samples modified with 60-60 min of *PITS-H/RTs* in the interaction effect of reaction variables (0.46 J/m), while the lowest values in the IS were found in the unmodified samples (0.35 J/m), 180-60 (0.37 J/m), and 300-60 min (0.37 J/m) of *PITS-H/RTs* in the interaction effect of reaction variables, respectively (Figure 8). Compared to the control group, the WPCs produced in 60-60 min of *PITs-H/RTs* WF provided about 23 % higher impact strength values.

3.3 Physical properties
3.3. Fizička svojstva

The individual effects of pre-impregnation times and heating/reaction times on the physical properties of PP composites filled with AA-modified WF are summarized in Tables 5 and 6, respectively.

Table 5 Individual effect of pre-impregnation times on physical properties of PP composites filled with AA-modified WF
Tablica 5. Individualni utjecaj vremena predimpregnacije na fizička svojstva PP kompozita ispunjenih AA modificiranim WF-om

Physical properties *Fizička svojstva*	**Pre-impregnation times** / *Vremena predimpregnacije*		
	60 min	180 min	300 min
Water absorption (24 h), % / *upijanje vode (24 h),* %	1.86 (1.5) [b]	1.06 (0.2) [a]	1.66 (0.5) [b]
Thickness swelling (24 h), % / *debljinsko bubrenje (24 h),* %	0.69 (4.3) [a]	0.19 (4.4) [a]	0.65 (4.1) [a]

Groups with the same letters in each row indicated no statistical difference ($p < 0.05$) between samples according to Duncan's multiple rang test. The values in parentheses are standard deviations.
Prema Duncanovu testu, grupe s istim slovima u svakom retku nisu pokazale statistički značajnu razliku ($p < 0,05$) među uzorcima. Vrijednosti u zagradama standardna su odstupanja.

Table 6 Individual effect of heating/reaction times on physical properties of PP composites filled with AA-modified WF
Tablica 6. Individualni utjecaj vremena zagrijavanja/reakcije na fizička svojstva PP kompozita ispunjenih AA modificiranim WF-om

Physical properties *Fizička svojstva*	**Heating/Reaction times** / *Vremena zagrijavanja/reakcije*		
	60 min	90 min	120 min
Water absorption (24 h), % / *upijanje vode (24 h),* %	2.29 (1.3) [b]	1.20 (0.3) [a]	1.08 (0.2) [a]
Thickness swelling (24 h), % / *debljinsko bubrenje (24 h),* %	-0.59 (5.7) [a]	1.19 (1.1) [a]	0.94 (4.4) [a]

Groups with the same letters in each row indicated no statistical difference ($p < 0.05$) between samples according to Duncan's multiple rang test. The values in parentheses are standard deviations.
Prema Duncanovu testu, grupe s istim slovima u svakom retku nisu pokazale statistički značajnu razliku (p < 0,05) među uzorcima. Vrijednosti u zagradama standardna su odstupanja.

The water absorption (*WA*) values of samples decreased with the increase in pre-impregnation times and heating or reaction times for the individual effects of *PITs* and *H/RTs* (Tables 5 and 6), where the values showed significant differences for both reaction variables. The same happened for thickness swelling (*TSw*), where the values showed significant differences in the individual effect of both reaction variables, but without special order. The greatest decrease in water absorption (*WA*) of 24 h was found for samples modified with 60-120 min of *PITS-H/RTs* (0.94 %) in the interaction effect of reaction variables (Table 7).

In contrast, the greatest increase value in *WA* of 24 h was found for samples modified with 60-60 min of *PITS-H/RTs* in the interaction effect of reaction variables (3.45 %). Compared to the control group, composites produced in 60-120 min and 60-60 min of *PITs-H/RTs* provided about 25 % lower and 63% higher *WA* values, respectively (Table 7).

The results showed that a significant reduction in *TSw* and *WA* properties of the WPCs was produced by using acetylated wood. This phenomenon may be explained by the fact that the amount of hydroxyl groups was substituted by acetyl moieties following the acetylation treatment, which leads to the decrease in the hydrophilicity of wood particles (Hung *et al.*, 2016). The greatest decrease in thickness swelling (*TSw*) of 24 h was found in samples modified with 180-60 min of *PITS-H/RTs* in the interaction effect of reaction variables (-2.58 %). In contrast, the greatest increase value in *WA* of 24 h was found in the samples modified with 60-60 min of *PITS-H/RTs* in the interaction effect of reaction variables (2.43 %). Compared to the control group, composites produced in 180-60 min and 60-60 min of *PITs-H/RTs* provided about 197 % lower and 8 % higher *TSw* values, respectively (Table 7).

The acetylation process significantly impacted the mechanical properties of wood-polymer composites (WPCs), including flexural strength (*FS*), tensile strength (*TS*), tensile modulus (*TM*), and impact strength (*IS*), with variations observed based on weight percentage gain (*WPG*) and pre-impregnation and heating or reaction times. Lower levels of *WPG* and shorter reaction times resulted in improved properties, indicating enhanced interfacial adhesion and polymer-WF interaction. However, higher levels of *WPG* and longer reaction times exhibited poor interfacial adhesion. Previous studies have highlighted the better bonding achieved between acetylated WF and the polymer matrix, although this improvement did not necessarily translate into improved mechanical properties of the composites.

This study introduced the use of acetic anhydride (AA) in different pre-impregnation and heating or reaction times, leading to enhancements in flexural strength, tensile strength, tensile modulus, and impact strength of WPCs. Similar results were found in previous studies (Abdul Khalil *et al.*, 2002; Segerholm *et al.*, 2007; Kwon and Ayrilmis, 2015). Acetylated wood particles contribute to the sufficient interfacial adhesion with the polymer matrix, which leads to a more efficient transfer of stress along the wood particle/polymer interface as compared to the control wood/polymer (Hung *et al.*, 2016). However, no significant change was observed in the flexural modulus. This can be attributed to the altered failure mode, shifting from fiber pullout to fiber breakage, caused by improved bonding between the polymer and WF. Acetylated WF

Table 7 Individual effect of pre-impregnation and heating or reaction times on physical properties of PP composites filled with AA-modified WF
Tablica 7. Individualni utjecaj vremena predimpregnacije i zagrijavanja ili reakcije na fizička svojstva PP kompozita ispunjenih AA modificiranim WF-om

Physical properties *Fizička svojstva*	**Treatment code (*PITs*/HRTs)** / *Oznaka tretmana (PIT/HRT),* min									
	60- 60	60- 90	60-120	180-60	180-90	180-120	300-60	300-90	300-120	Control
WA (24 h), %	3.45[c] (0.4)	1.19[a] (0.1)	0.94[a] (0.1)	1.12[a] (0.1)	0.95[a] (0.4)	1.11[a] (0.1)	2.32[b] (0.0)	1.46[ab] (0.2)	1.20[a] (0.1)	1.26[a] (0.3)
TSw (24 h), %	2.43[a] (0.0)	0.00[a] (0.0)	-0.35[a] (0.8)	-2.58[a] (0.8)	1.85[a] (0.8)	1.30[a] (0.3)	-1.63[a] (0.7)	1.73[a] (0.1)	1.87[a] (0.8)	2.65[a] (0.6)

WA – Water absorption; *TSw* – Thickness swelling. Groups with the same letters in each row indicated no statistical difference ($p < 0.05$) between samples according to Duncan's multiple range test. The values in parentheses are standard deviations.
WA – upijanje vode; TSw – debljinsko bubrenje. Prema Duncanovu testu, grupe s istim slovima u svakom retku nisu pokazale statistički značajnu razliku (p < 0,05) među uzorcima. Vrijednosti u zagradama standardna su odstupanja.

exhibited higher flexural strength compared to unmodified WF, with a slight reduction in flexural modulus potentially attributed to the weight gain caused by acetylation. The density is one of the significant parameters affecting the mechanical properties.

The compatibility between WF and the polymer matrix improved due to reduced polarity after acetylation, resulting in increased mechanical properties. However, excessive acetylation levels could hinder effective stress transmission from the matrix to the fibers, leading to reduced composite properties. The flexural strength, tensile strength, and moduli, as well as impact strength, were significantly increased in samples treated with AA, even with lower *WPG* compared to other treatments. However, a linear correlation between pre-impregnation and reaction times and mechanical properties was not observed. Inadequate washing to remove unreacted chemicals and by-products after modification may have contributed to the reduction and fluctuation in properties. The application of a Soxhlet cleaning procedure after acetylation was found to have a positive effect on the mechanical properties of WPCs compared to simple water washing.

The success of chemical modification in WF for WPCs depends on various factors, such as the method used, reaction time and temperature, and removal of unreacted chemicals. Substituting hydroxyl groups in WF with acetyl groups increased the hydrophobicity of the WF surface, improving compatibility with the polymer matrix. Additionally, the upward trend observed in flexural strength, tensile strength, and moduli, and impact strength with increasing *WPG* may be attributed to reduced moisture sorption and increased brittleness of the samples. The use of anhydrides and higher reaction temperatures influenced the cross-linking of lignin in WF during the formation of cellulose-reinforced thermoplastic composites.

As a result of the chemical reaction between wood and acetic anhydride as the reagent, an increase in dimensions of the reacted wood species has occurred, because of swelling of the wood cell wall. In most cases, chemically modified wood has a lower capacity for water absorption, with lower equilibrium moisture content at a specified atmospheric relative humidity, compared to unmodified wood (Teaca *et al.*, 2014).

4 CONCLUSIONS
4. ZAKLJUČAK

In this research, WPCs were made from acetylated wood flour. The highest and lowest *WPG* levels were achieved for 300-120 min for *PITs-H/RTs* and 180-60 min for *PITs-H/RTs*, respectively. The test results revealed that the chemical modifications of WF in different *PITs* and *H/RTs* improved the mechanical and physical properties of the WPCs. The highest mechanical properties were found in the WPCs produced with wood acetylated at *PITs-H/RTs* for 60-60 min with the exception of impact strength, while the least *WA* and *TSw* were found in the *PITs-H/RTs* for 60-120 min and *PITs-H/RTs* for 300-60 min, respectively. The WPCs produced with wood acetylated at *PITs-H/RTs* for 180-60 min affected the physical characteristics, while the positive effect did not affect all the mechanical properties compared to unmodified or control samples. Regarding the physical and mechanical properties of the WPCs, it was observed that surface modification by both anhydride acetic and reaction times improved the tensile and flexural strengths as well as *WA* and *TSw* of the composites. This was because the surface modification could improve the compatibility between wood flour and PP matrix, leading to fewer microvoids and fiber-PP debonding in the interphase region. Simultaneous use of pre-impregnation and reaction times had a synergic effect on the physical and mechanical properties.

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Effect of Epoxy Resin Reinforcement on Screw Withdrawal Strength of Fiberboard and Particleboard Used in Furniture Industry

Utjecaj ojačanja epoksidnom smolom na izvlačnu silu vijka u ploča vlaknatica i iverica koje se rabe u proizvodnji namještaja

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ABSTRACT • *The study aimed to increase the screw withdrawal strength of medium density fiberboard and particleboard used in furniture strength by using epoxy resin in the screw pilot hole. Therefore, the effects of pilot hole diameters, screw diameter, and amount of epoxy resin on screw withdrawal strength of medium density fiberboard and particleboard from face and edge were investigated. According to TS EN 13446, 50 mm × 50 mm specimens were cut from commercial medium density fiberboard and particleboard boards. A static load was applied parallel to the screw direction. The screw withdrawal strength of medium density fiberboard was higher than the screw withdrawal strength of particleboard because of its density. Besides, the screw withdrawal strength of medium density fiberboard and particleboard samples with a 3.5 mm screw diameter was higher compared to those with a 4.5 mm screw diameter. A decrease in pilot hole diameter and an increase in the amount of epoxy resin provided higher screw withdrawal strength of materials. Using 20 % epoxy resin of the volume of the pilot hole resulted in two times better screw withdrawal strength values. The study showed that a higher amount of epoxy resin, smaller pilot hole diameter, and smaller screw diameter contribute to better screw withdrawal strength of both medium density fiberboard and particleboard from the face and edge.*

KEYWORDS: *epoxy resin; reinforcement; screw withdrawal strength; medium density fiberboard; particleboard*

SAŽETAK • *Cilj ovog istraživanja bio je povećati izvlačnu silu vijka u ploča vlaknatica i iverica koje se rabe u proizvodnji namještaja, i to upotrebom epoksidne smole u pilot-rupama za vijke. Stoga je ispitan utjecaj promjera pilot-rupa, promjera vijka i količine epoksidne smole na izvlačnu silu vijka na plohi i rubu ploče vlaknatice i iverice. Uzorci dimenzija 50 mm × 50 mm prema TS EN 13446 izrađeni su od komercijalne srednje guste ploče vlaknatice i ploče iverice. Statičko opterećenje djelovalo je paralelno na smjer vijka. Zbog razlika u gustoći ploča*

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izvlačna sila vijka bila je veća za srednje gustu ploču vlaknaticu nego za ploču ivericu. Osim toga, izvlačna sila vijka promjera 3,5 mm u srednje gustoj ploči vlaknatici i ploči iverici bila je veća od izvlačne sile vijka promjera 4,5 mm. Smanjenje promjera pilot-rupe i povećanje količine epoksidne smole omogućilo je veću otpornost materijala na izvlačenje vijaka. Upotrebom 20 % epoksidne smole u odnosu prema volumenu pilot-rupe rezultiralo je dvostruko boljim vrijednostima izvlačne sile vijka. Istraživanje je pokazalo da veća količina epoksidne smole, manji promjer pilot-rupe i manji promjer vijka pridonose boljoj izvlačnoj sili vijka na plohi i rubu srednje guste ploče vlaknatice i ploče iverice.

KLJUČNE RIJEČI: *epoksidna smola; ojačanje; izvlačna sila vijka; srednje gusta ploča vlaknatica; ploča iverica*

1 INTRODUCTION
1. UVOD

Joints are the weakest component of furniture construction (Smardzewski, 2009). In the case of any joint failure, all construction fails. Therefore, the reusability of the furniture decreases over time due to the fact that furniture joints loosen or cannot provide its serviceability. Especially, in case furniture, screw joints are a typical example where a furniture member or screw is not damaged, but the screw hole enlarge. Besides, screws are used for demountable furniture to be easily assembled and disassembled. However, screw holes enlarge after screwing off, so the screw pilot hole diameter increases and the joint does not have the initial strength after re-assembly. Larger screws could be preferred for re-assembly efforts, which increases the strength of the screw joints because the pilot hole diameter was increased in that screw withdrawn from the material (Uysal *et al.,* 2015). However, the use of larger joints may not be suitable for wood-composite materials, especially edgewise, because of their thickness and internal bond strength. On the contrary, the use of adhesive in screw pilot holes is another method to increase the screw withdrawal strength (*SWS*) of materials. Epoxy resin (EP) is a better selection to reinforce screw withdrawal strength compared to other adhesives because it provides higher mechanical properties for adhesives containing EP as reinforcement.

An increase in joint strength decreases the environmental impact of furniture because its serviceability and life span are increased since the rate of discarded furniture in landfills decreases while the rate of reusability increases. Besides, the repairability of the furniture construction with larger screws or the use of adhesive could increase the life span of the furniture. Remanufacturing furniture is another way to increase furniture life span and decrease its environmental impact before being discarded in landfills. However, the remanufacturing market of furniture is responsible for 0.4 % of the furniture industry, and the number of employees is less compared to employees in the reuse network (Parker *et al.,* 2015). In addition, furniture remanufacturing requires more energy than furniture reuse, according to the product recovery hierarchy in the end-of-life options (Östlin *et al.,* 2009). Hence, increasing the service life of furniture with screw joints could be provided by increasing the *SWS* of materials used in construction, so the second users can reuse furniture after it fulfills its service life for the first users.

Screw-type joints are mainly used in demountable furniture for easy assembly and disassembly. Therefore, it should be considered how joint strength would be increased after the first use or dissembling. Fasteners like screws, minifixes, confirmats, etc. are effective joinery systems to join elements made of wood-based composites and are used in case furniture. The strength of joints depends on the material, joinery system, screw pilot hole, number of fasteners and distance between fasteners. However, it is significant to have a self-locking system for each fastener to hold material. In addition, freezing and heating significantly affect material withdrawal capacity (Gašparík *et al.,* 2023). Thus, increasing the *SWS* of materials comes into prominence to improve the overall strength of furniture joints. It depends on their density and internal bonding strength, diameter of pilot hole, length of screw penetration, diameter of screw shank and thread, and filling material injected in a pilot hole (Smardzewski *et al.,* 2016). Besides, thread geometry such as thread height, flank distance, teeth angle, and thread angle significantly affect the *SWS* of the material (Hoelz *et al.,* 2022).

Fiberboard (MDF) and particleboard (PB) are widely used as raw materials for Ready-to-Assembly (RtA) furniture. Screw-type fasteners are not only used in joints but also in handles, hinges and locks (Örs *et al.,* 1998). Studies related to joints with screws have been conducted with the technological development in wood composites and engineered wood since the strength design of furniture was defined in the 1940s (Eckelman, 1973; Smardzewski, 2009; Uysal and Haviarova, 2019). Such studies will continue to be conducted to enhance the strength of screw joints as long as the furniture is the greatest need of people's living spaces and as long as new technological developments emerge in the field. The *SWS* of material increased by 9-45 % and 7-39 % with injected polyvinyl acetate adhesive in pilot holes for MDF and PB compared to those without adhesive. In addition, an increase in the thread depth of the screw provided higher *SWS* (Örs *et al.,* 1998). The correlation between *SWS* and internal bonding strength is better than that of density and was studied to predict the internal bond strength of PB from its *SWS* (Semple and Smith, 2005). The larger-diameter screws had a greater *SWS* than smaller-diameter screws, but thread depth is more critical than screw diameter for the *SWS* of materials (Abu and Ahmad, 2015). Polyvinyl acetate and polyurethane adhesive were used in screw pilot holes to increase joint strength in RtA furniture (Imirzi *et al.,* 2016). The type of material and type of screw affected the *SWS* of the material (Wolpiuk and Sydor, 2016). The *SWS* of a material increases with higher material density (Bal *et al.,* 2017; Jivkov *et al.,* 2017). The *SWS* of wood-based materials was inversely proportional to the modulus of elasticity because of high stiffness (Yunus *et al.,* 2019). The effects of screw type, screw orientation, and adhesive on the *SWS* of MDF and PB were studied. Results showed that the use of adhesive in pilot holes increased the *SWS* of both MDF and PB (Yorur *et al.,* 2020). The pilot hole diameter and density effects on the *SWS* of MDF were studied. Results revealed that pilot diameter had more impact on *SWS* than material density. Besides, coarse thread screws have lower *SWS* than fine thread screws; namely, the distance between screw threads is also a significant factor. *SWS* from the face was greater than *SWS* from the edge because the face density of MDF is higher than the core density. *SWS* is also affected by the adhesive composition used in particleboard (Farajollah Pour *et al.,* 2022). The reinforcement type in a material increases its *SWS* due to higher density (Perçin and Uzun, 2022). Different size and amount of waste melamine impregnated paper (WMIP) as adhesive in particle boards were studied (Başboğa *et al.,* 2023). *SWS* of particleboard increased with narrower size and higher amount of WMIP.

EP was recommended for frame construction with biscuit joints (Yildirim *et al.,* 2017). EP provides a more durable adhesion with its mechanical adhesion force. It achieves more and faster penetration to the adhesion surface pores on the material than other adhesives due to its higher elasticity (Karaman *et al.,* 2017). Also, an increase in the mechanical interlocking mechanism between wood material and the surface of metal fasteners with EP enhances the *SWS* of wood-based composite materials. In light of this information, the present study was carried out with the idea of adding EP as reinforcement to the screw joint to increase the *SWS* of materials. In doing so, the effects of pilot hole diameters, screw diameter, and amount of EP on *SWS* of MDF and PB from face and edge were investigated.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Design of experiment
2.1. Postavke eksperimenta

In this study, MDF and PB materials, widely used in panel-type furniture and commercially produced, were investigated. A complete four-factor factorial experiment with five replications in each sample group was conducted to determine the *SWS* of the MDF and PB. For this purpose, two materials (MDF and PB), two screw diameters (3.5 mm and 4.5 mm), three pilot-hole diameters (80 %, 90 % and 100 % of screw diameter) and four levels of the EP amount (no-resin, 20 %, 40 % and 60 % of volume of the pilot-hole) factors were selected for edge and face withdrawal from specimens. In doing so, 480 specimens in 96 sample groups were tested to examine the effect of the EP on the *SWS* of the materials.

2.2 Preparation of specimens
2.2. Priprema uzoraka

According to TS EN 13446, all specimens were cut into 50 mm × 50 mm nominal dimensions from 18 mm × 2100 mm × 2800 mm of MDF and PB boards obtained from a local store in the Inegol furniture cluster in Turkey. Table 1 shows the average density and moisture content of the MDF and PB used in the study. The dimensions of the specimens and orientations for the pilot holes are given in Figure 1.a and b. All pilot holes were drilled according to screw diameter and depth of screw penetration in MDF and PB. The depth of the screw penetration is equal to 8d (d: screw diameter) or at most 30 mm for *SWS* from the edge and 16 mm for *SWS* from the face of the materials. Besides, the depth of the pilot hole was 70 % of the depth of the screw penetration.

Table 1 Average density and moisture content of materials
Tablica 1. Prosječna gustoća i sadržaj vode u pločama

Material *Materijal*	**Density** / *Gustoća,* g/cm³						**Moisture content, %** *Sadržaj vode, %*		
	Air-dry			Oven-dry					
	Mean	SD*	CoV*	Mean	SD*	CoV*	Mean	SD*	CoV*
MDF	0.70	0.004	0.57 %	0.67	0.004	0.60 %	6.98	0.07	1.01 %
PB	0.62	0.006	0.91 %	0.59	0.006	1.01 %	8.78	0.03	0.30 %

*SD – standard deviation, CoV – coefficient of variation / **SD – standardna devijacija, CoV – koeficijent varijacije*

Table 2 Dimensions of pilot hole and amount of EP for each sample group
Tablica 2. Dimenzije pilot-rupe i količina epoksidne smole za svaku skupinu uzoraka

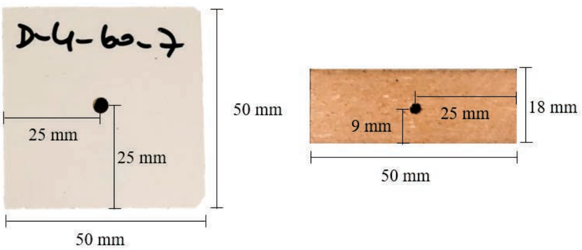
Screw orientations *Orijentacija vijka*	**For 3.5 × 45 mm screw (4.5 × 45 mm screw)** *Za vijak 3,5 × 45* mm *(vijak 4,5 × 45* mm*)*				
	Depth of pilot hole, mm *Dubina pilot-rupe,* mm	**Diameter of pilot hole, mm** *Promjer pilot-rupe,* mm	**Amount of EP, μl** *Količina epoksidne smole,* μl		
			20 %	40 %	60 %
Edge / *rub*	19.60 (21.00)	2.80 (3.60)	26 (50)	50 (100)	75 (150)
		3.15 (4.05)	32 (60)	60 (120)	90 (180)
		3.50 (4.50)	40 (80)	80 (160)	115 (240)
Face / *ploha*	11.20 (11.20)	2.80 (3.60)	20 (32)	40 (65)	60 (100)
		3.15 (4.05)	25 (40)	50 (80)	75 (120)
		3.50 (4.50)	30 (50)	60 (100)	90 (150)

* Values in parathesis are for screws with a diameter of 4.5 mm. / * *Vrijednosti u zagradama odnose se na vijke promjera 4,5 mm.*

Table 3 Length, penetration depth, diameter, and root diameter of screws used in the study
Tablica 3. Duljina, dubina prodiranja, promjer i promjer unutarnjeg navoja vijaka upotrijebljenih u istraživanju

		3.5 × 45 mm screw (4.5 × 45 mm screw) / *Vijak 3,5 × 45 mm (vijak 4,5 × 45 mm)**			
		Length *Duljina*	Penetration depth *Dubina prodiranja*	Diameter *Promjer*	Root diameter *Unutarnji promjer*
Edge *Rub*	Mean	44.14 (44.21)	27.88 (29.24)	3.54 (4.46)	2.33 (2.80)
	SD	0.39 (0.35)	0.73 (0.93)	0.07 (0.04)	0.04 (0.02)
	CoV	0.88 % (0.78 %)	2.61 % (3.19 %)	1.86 % (0.84 %)	1.79 % (0.60 %)
Face *Ploha*	Mean	44.15 (44.19)	15,96 (16.05)	3.53 (4.48)	2.30 (2.80)
	SD	0.40 (0.34)	0.67 (0.69)	0.07 (0.02)	0.02 (0.05)
	CoV	0.90 % (0.76 %)	4.21 % (4.29 %)	2.02 % (0.41 %)	0.78 % (1.78 %)

* Values in parathesis are for screws with a diameter of 4.5 mm. / * *Vrijednosti u zagradama odnose se na vijke promjera 4,5 mm.*



a) b)

Figure 1 Pilot hole position for a) face withdrawal and b) edge withdrawal
Slika 1. Položaj pilot-rupe: a) za izvlačenje iz plohe, b) za izvlačenje iz ruba

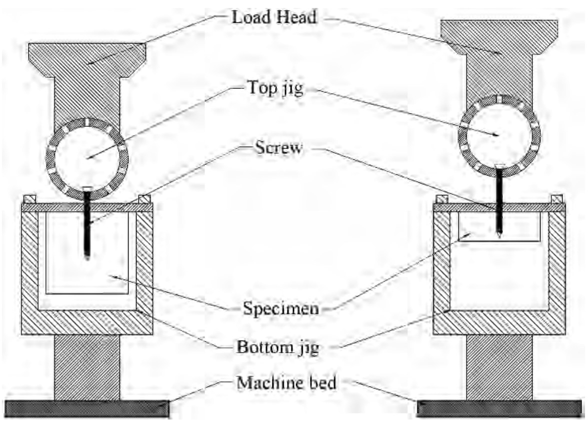
Table 2 shows pilot hole dimensions and the amount of EP for each sample group. Epoxy and hardener, obtained from a local store, were mixed to a 5/2 ratio for 3 minutes. Epoxy and hardener mix were exposed to air for at most 15 minutes before the screws were driven. After each specimen was drilled for a pilot hole and an appropriate amount of EP was injected, 3.5 and 4.5 mm diameter screws were driven into pilot holes to the above length. Table 3 shows the average length, penetration depth, diameter, and root diameter of the screws used in the study.

2.3 Determination of screw withdrawal strength
2.3. Određivanje izvlačne sile vijka

All tests for the *SWS* were conducted on the SHIMADZU universal test machine according to TS EN 13446. Withdrawal loads were obtained by applying a withdrawal load parallel to the screw axis from the edge and face of specimens with a rate of 0.8 mm/min and 2 mm/min, respectively, and continued until the ultimate load was reached (Figure 2). *SWS*, *f* (MPa), was calculated by using Eq. 1.

$$f = \frac{F_{ult}}{d \cdot l_p} \quad (1)$$

F_{ult} – Ultimate withdrawal load (N)
d – Diameter of screw tread (mm)
l_p – depth of screw penetration (mm)



a) b)

Figure 2 Test configuration for a) edge withdrawal and b) face withdrawal
Slika 2. Ispitna konfiguracija: a) za izvlačenje iz plohe, b) za izvlačenje iz ruba

2.4 Statistical analysis
2.4. Statistička analiza

Data collected for all specimens were checked for determining statistical significance through Four-way ANOVA analysis, and Tukey pairwise comparisons were carried out in Minitab Statistical Analysis Software (Minitab Inc. 2013).

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Screw withdrawal strength from the edge
3.1. Izvlačna sila vijka na rubu

Results for *SWS* from the edge are given in Figure 3. According to test results, the average *SWS* from the edge of MDF was 9.26 MPa with a standard deviation of 0.56 MPa provided that the pilot hole was 80 % of the 3.5 mm screw diameter. An increase in the pilot hole diameter from 80 % to 90 % and 100 % decreased *SWS* of the MDF by 7.89 MPa and 3.42 MPa with standard deviations of 1.32 MPa and 0.47 MPa, respectively. If 4.5 mm diameter screws were used, the average *SWS* from the edge of MDF was 8.05 MPa with a standard deviation of 0.50 MPa, 6.80 MPa with a standard deviation of 0.28 MPa and 2.88 MPa with a standard deviation of 0.13 MPa for 80 %, 90 % and 100 % pilot hole of 4.5 mm-screw-diameter, respectively. If PB was used, the average *SWS* from the edge was 6.91 MPa with a standard deviation of 0.63 MPa, 4.92 MPa with a standard deviation of 0.44 MPa, and 3.50 MPa with a standard deviation of 0.22 MPa for 80 %, 90 % and 100 % pilot hole of 3.5 mm-screw-diameter, respectively. Moreover, those of 4.5 mm-screw diameter had 6.27 MPa with a standard deviation of 0.69 MPa, 5.17 MPa with a standard deviation of 0.78 MPa, and 2.80 MPa with a standard deviation of 0.19 MPa, respectively.

It could be observed that the average *SWS*s from the of MDF are higher than those of PB not only because of internal bond strength but also because the density of the material directly affected the *SWS* (Semple and Smith, 2005; Bal *et al.,* 2017; Jivkov *et al.,* 2017; Başboğa *et al.,* 2023). Besides, statistical analysis results

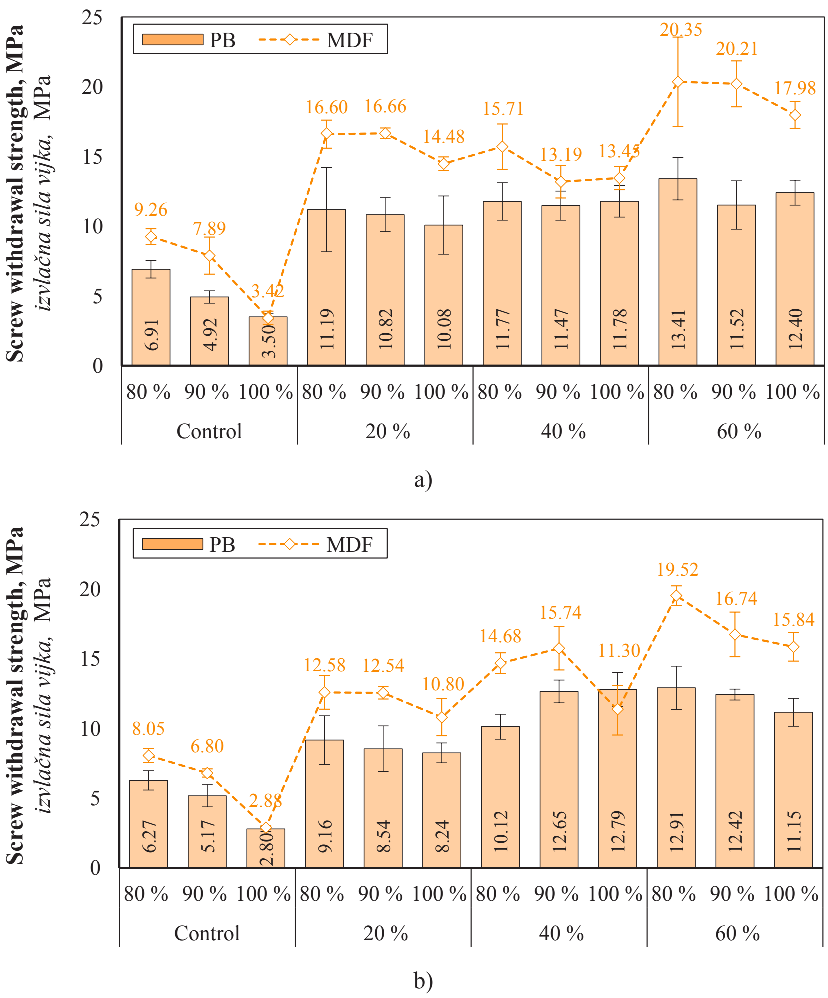


Figure 3 Effect of amount of EP on *SWS* from edge of material for a) 3.5 mm and b) 4.5 mm screw diameter
Slika 3. Utjecaj količine epoksidne smole na izvlačnu silu vijka na rubu materijala: a) za promjer vijka od 3,5 mm, b) za promjer vijka od 4,5 mm

shown in Table 4 and Table 5 documented that the type of material significantly affected *SWS* from the edge (p-value = 0.000 < 0.05), and that the mean values of *SWS* from the edge of MDF and PB were significantly different from each other (α = 0.05), respectively. Furthermore, larger screw diameter levels yielded decreasing *SWS* values from the edge of both MDF and PB. Although larger screws provide higher *SWS* for materials, screw diameter affected *SWS* when drilling a pilot hole but not the screw insertion technique (Abu and Ahmad, 2015). Therefore, 4.5 mm screw sizes might have an adverse impact on *SWS* because of using EP. The average *SWS* from the edge for a 3.5 mm-screw diameter was 11.3 % higher than those of 4.5 mm. Also, Table 4 and Table 5 showed that screw diameter had a significant impact on the *SWS* from the edge (*p*-value = 0.000 < 0.05) and that the mean values of *SWS* from the edge for 3.5 mm and 4.5 mm screw diameter were significantly different (α = 0.05), respectively.

Pilot hole diameter is vital to the strength of joints with screws because the difference between its shank diameter and screw teeth length affects *SWS* depending on the material. Therefore, it should be close to the shank diameter so that the screw is easily driven into the material without damaging it (Eckelman, 2003). On the other hand, joinery systems with screws allow easily assembled and disassembled furniture construction. However, every effort to disassemble furniture construction enlarges the pilot hole, and re-assembling the furniture results in lower strength of joints. The test results showed that larger pilot hole diameters decreased the *SWS* of materials, while the increased levels of EP injected in the pilot hole enhanced *SWS*. The amount of EP was increased by 20 %, 40 % and 60 % of the pilot hole diameter. In the case of MDF samples with a 3.5 mm screw diameter and 60 % EP, *SWS* from the edge was increased by 119.67 %, 156.56 %, and 425.59 % for 80 %, 90 % and 100 % pilot hole diameters, respectively. Those with 40 % and 20 % EP, increased *SWS* as much as 69.49 %, 67.33 %, 292.66 % and 79.32 %, 111.55 % and 322.94 %, respectively.

Furthermore, *SWS* was increased by 94.24 % when the pilot hole diameter increased from 80 % to 100 %, and the amount of EP increased from 0 to 60 %. *SWS* increase in those with 40 % and 20 % EP was 45.08 % and 56.27 %, respectively. Not only did both the pilot hole (p-value =0.000) diameter and amount of EP (*p*-value = 0.000) have a significant effect on *SWS* from the edge, but the interaction of pilot hole diameter and amount of EP (p-value = 0.000) also significantly affected *SWS* from the edge (Table 4). Furthermore, as shown in Table 5, based on Tukey pairwise comparisons, the mean values of *SWS* from the edge for pilot hole diameter and amount of EP significantly differed at the 95 % confidence level, respectively. An *SWS* increase should be achieved with a screw pilot hole close to the root diameter (Farajollah Pour *et al.,* 2022). The pilot hole diameter should be at least 70 % of the screw diameter (Eckelman, 2003). The holding mechanism of the screw on materials depends on the screw thread rather than the whole screw diameter. In this study, specimens with 70 % pilot hole diameter and EP failed in the case of drilling screws, so these sample groups were omitted from the experiment.

As can be observed in Table 4, interactions of (i) screw diameter and pilot hole diameter, (ii) screw diameter, amount of EP, and material, (iii) screw diameter, amount of EP, and pilot hole diameter, (iv) screw diameter, material, and pilot hole diameter, (v) amount of EP, material, and pilot hole diameter, and (vi) screw diameter, amount of EP, material, and pilot hole diameter had no significant effects on *SWS* from the edge.

3.2 Screw withdrawal strength from the face
3.2. Izvlačna sila vijka na plohi

Test results for *SWS* from the face are given in Figure 4. According to test results, the average *SWS* from the face of MDF was 11.80 MPa with a standard

Table 4 Four-way ANOVA for *SWS* from the edge
Tablica 4. Četverosmjerna ANOVA za izvlačnu silu vijka na rubu

Source / *Izvor*	***DF***	**Adj *SS***	**Adj *MS***	***F*-Value**	***P*-Value**
Screw diameter (D) / *promjer vijka (D)*	1	89.59	89.59	50.09	0.000
Epoxy resin (EP) / *epoksidna smola (EP)*	3	3068.02	1022.67	571.79	0.000
Material (M) / *materijal (M)*	1	746.91	746.91	417.61	0.000
Pilot hole (P) / *pilot-rupa (P)*	2	206.88	103.44	57.84	0.000
D*EP	3	73.63	24.54	13.72	0.000
D*M	1	20.85	20.85	11.66	0.001
EP*M	3	199.32	66.44	37.15	0.000
EP*P	6	86.00	14.33	8.01	0.000
M*P	2	55.96	27.98	15.64	0.000
Error	217	388.11	1.79		
Lack-of-Fit	25	88.71	3.35	2.11	0.003
Pure Error	192	304.40	1.59		
Total	239	4935.27			

Table 5 Tukey pairwise comparisons for *SWS* from the edge of material
Tablica 5. Tukey usporedbe parova za izvlačnu silu vijka na rubu materijala

Variables / *Varijable*		*N*	**Average** *Prosjek*	**Grouping** / *Grupiranje*			
Material / *materijal*	MDF	120	13.19	A			
	PB	120	9.67		B		
Screw diameter / *promjer vijka*	3.5 mm	120	12.04	A			
	4.5 mm	120	10.82		B		
Amount of EP / *količina epoksidne smole*	60 %	60	15.37	A			
	40 %	60	12.88		B		
	20 %	60	11.81			C	
	Control	60	5.66				D
Pilot hole diameter / *promjer pilot-rupe*	80 %	80	12.41	A			
	90 %	80	11.71		B		
	100 %	80	10.18			C	

deviation of 0.85 MPa if the pilot hole was 80 % of the 3.5 mm screw diameter. Increasing pilot hole diameter from 80 % to 90 % and 100 % decreased *SWS* of the MDF by 9.45 MPa and 7.34 MPa with standard deviations of 0.85 MPa and 0.31 MPa, respectively. If 4.5 mm diameter screws were used, the average *SWS* from the face of MDF was 9.71 MPa with a standard deviation of 0.47 MPa, 7.96 MPa with a standard deviation of 0.85 MPa and 5.26 MPa with a standard deviation of 0.31 MPa for 80 %, 90% and 100 % pilot hole of 4.5 mm-screw-diameter, respectively. If PB was used, the average *SWS*s from the face were 8.97 MPa with a standard deviation of 0.57 MPa, 6.49 MPa with a standard deviation of 0.72 MPa, and 5.93 MPa with a standard deviation of 0.60 MPa for 80 %, 90 % and 100 % pilot hole of 3.5 mm-screw-diameter, respec-

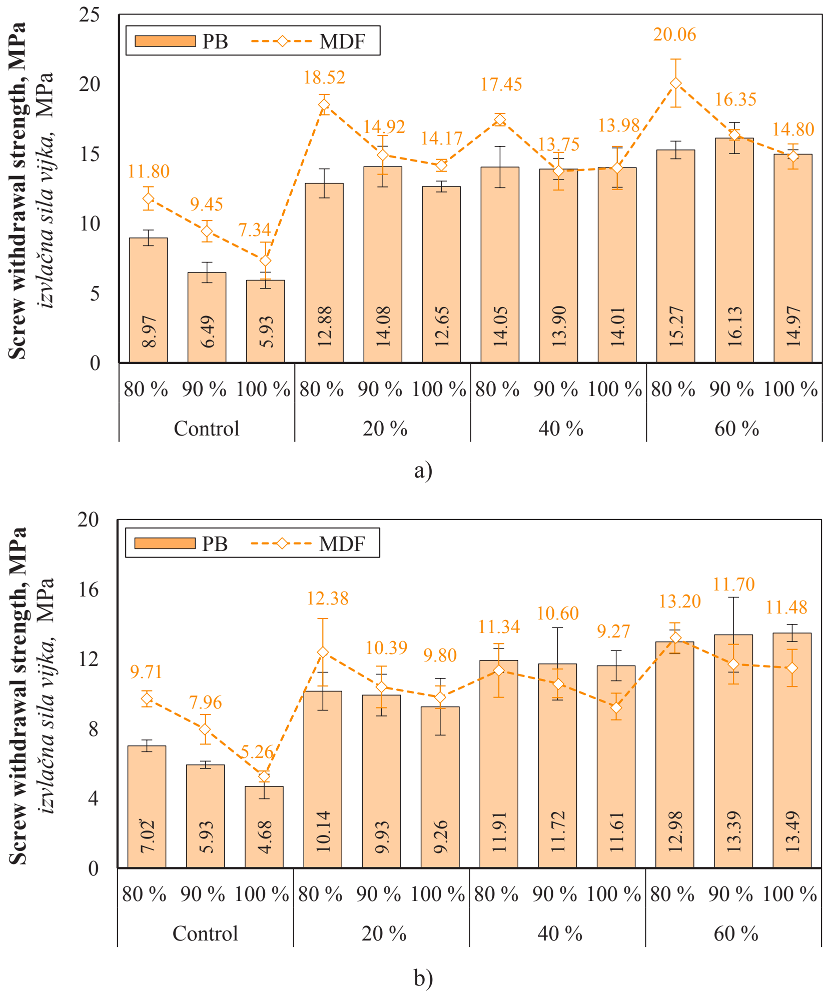


Figure 4 Effect of amount of EP on *SWS* from the face of material for a) 3.5 mm and b) 4.5 mm screw diameter
Slika 4. Utjecaj količine epoksidne smole na izvlačnu silu vijka na plohi materijala: a) za promjer vijka od 3,5 mm, b) za promjer vijka od 4,5 mm

tively. Furthermore, those of 4.5 mm-screw diameters had 7.02 MPa with a standard deviation of 0.35 MPa, 5.93 MPa with a standard deviation of 0.22 MPa, and 4.68 MPa with a standard deviation of 0.69 MPa, respectively.

Four-Way ANOVA results presented in Table 6 showed that all independent variables, namely, screw diameter (p-value = 0.000), material type (p-value = 0.000), amount of EP (p-value = 0.000), and pilot hole diameter, significantly affected the mean values of *SWS* from the face of specimens. In terms of the impact of material type on *SWS* from the face, a similar pattern was obtained with those from the edge; namely, the mean *SWS* from the face of MDF (11.80 MPa) was higher than that of PB (8.97 MPa) because MDF is a denser material with a higher internal bonding strength than PB. Tukey pairwise comparisons identified and documented the presence of *SWS* from the face differences across various levels of each independent factor. Table 7 demonstrated that the mean *SWS* from the face of MDF was significantly different from that of PB (α = 0.05). Table 5 also showed that changing screw diameter levels resulted in significantly different mean *SWS* from the face values. Besides, the mean *SWS* from the face for 3.5 mm and 4.5 mm screw diameters were significantly different from each other (α = 0.05). As given in Table 6, the interaction of screw diameter and material also significantly affected the average *SWS* from the face (p-value = 0.000).

A smaller pilot hole diameter and higher amount of EP resulted in higher mean *SWS* from the face of both MDF and PB. According to Table 6, the interaction of pilot hole diameter and amount of EP (p-value = 0.001) significantly impacted *SWS* from the face. The average *SWS* from the face of MDF increased by 69.94 %, 73.09 %, and 101.28 % for 80 %, 90 % and 100 % pilot hole diameter when using a 3.5 mm screw diameter, and the amount of EP was 60 % of pilot hole diameter, respectively. For the same pilot hole diameters, the increase in *SWS* from the face for the samples with 4.5 mm screw diameter and the amount of EP of 60 % of pilot hole diameter was 68.74 %, 46.85 % and 117.86 %, respectively. Similar increases in the mean *SWS* from the face of MDF and PB were observed for the samples with the amount of EP equal to 20 % and 40 % of the pilot hole diameter. According to Tukey pairwise comparison results, the mean *SWS* from the face for the samples with the amount of EP equal to 60 % of the pilot hole diameter and without resin was significantly different. However, there was not enough statistical evidence to claim that the mean *SWS* from the face values for the groups with an amount of EP equal to 20 % and 40 % of the pilot hole diameter (Table 7) were different. Furthermore, Table 7 also demonstrated that the mean *SWS* from the face values for 80 %, 90 % and 100 % pilot hole diameters were significantly different from each other. Table 6 also showed that interactions of (i) screw diameter, amount of EP, and pilot hole diameter and (ii) all parameters had no significant effects on the *SWS* from the face.

4 CONCLUSIONS
4. ZAKLJUČAK

This study investigated the effect of EP in the pilot hole on the *SWS* of MDF and PB. For this purpose, three pilot hole diameters, four amounts of EP and two screw diameters were investigated.

Internal bond strength and density of material have a significant effect on *SWS*. No matter what pilot

Table 6 Four-way ANOVA for *SWS* from the face
Tablica 6. Četverosmjerna ANOVA za izvlačnu silu vijka na plohi

Source / *Izvor*	*DF*	Adj *SS*	Adj *MS*	*F*-Value	*P*-Value
Screw diameter (D) / *promjer vijka (D)*	1	613.29	613.29	522.98	0.000
Epoxy resin (EP) / *epoksidna smola (EP)*	3	1602.82	534.27	455.60	0.000
Material (M) / *materijal (M)*	1	61.67	61.67	52.59	0.000
Pilot hole (P) / *pilot-rupa (P)*	2	193.89	96.94	82.67	0.000
D*EP	3	58.40	19.46	16.60	0.000
D*M	1	51.42	51.42	43.84	0.000
D*P	2	10.50	5.25	4.48	0.012
EP*M	3	57.38	19.13	16.31	0.000
EP*P	6	28.09	4.68	3.99	0.001
M*P	2	83.46	41.73	35.59	0.000
D*EP*M	3	10.27	3.43	2.92	0.035
D*M*P	2	10.75	5.37	4.58	0.011
EP*M*P	6	15.95	2.66	2.27	0.039
Error	204	239.23	1.17		
Lack-of-Fit	12	18.22	1.52	1.32	0.210
Pure Error	192	221.01	1.15		
Total	239	3037.11			

Table 7 Tukey pairwise comparisons for *SWS* from the face of material
Tablica 7. Tukey usporedbe parova za izvlačnu silu vijka na plohi materijala

Variables / *Varijable*		*N*	Average *Prosjek*	Grouping / *Grupiranje*		
Material / *materijal*	MDF	120	12.32	A		
	PB	120	11.31		B	
Screw diameter / *promjer vijka*	3.5 mm	120	13.41	A		
	4.5 mm	120	10.21		B	
Amount of EP / *količina epoksidne smole*	60 %	60	14.48	A		
	40 %	60	12.80		B	
	20 %	60	12.43		B	
	Control	60	7.54			C
Pilot hole diameter / *promjer pilot-rupe*	80 %	80	12.98	A		
	90 %	80	11.67		B	
	100 %	80	10.79			C

hole diameter, amount of EP and screw diameter were used, the *SWS* of MDF was higher than the *SWS* of PB. An increase in screw diameter from 3.5 to 4.5 mm significantly affects the *SWS* of MDF and PB. In the edge and face withdrawal of screws from materials, smaller screw diameter has higher *SWS*. Increase in pilot hole diameter decreases the *SWS* of materials. 80 % pilot hole diameter had greater *SWS* than that of 90 % and 100 % because it was the closest to the root diameter. In addition, an increase in the amount of EP in the pilot hole increases the *SWS* of the material. EP dispenses more and faster in material pores, so it works better after drilling screws in pilot holes. If a piece of furniture with screw-type fasteners were disassembled, its pilot hole would be close to 100 % of or larger than the screw diameter. Using EP in the pilot hole for the following assembly would be beneficial. *SWS* of materials with 80 % pilot hole diameter and no-EP increased by 40 % compared to that of 100 % pilot hole with 60 % EP of the volume of the pilot hole.

The study showed that the existence of EP in the pilot hole increases the *SWS* of both MDF and PB. In this way, it could be used to repair furniture with screw-type fasteners. Moreover, it has a longer life span before being discarded in landfills and increases the reusability and repairability of case furniture to decrease its environmental impacts.

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Drvo topole
Populus spp.

MIKROSKOPSKA OBILJEŽJA

Drvo topole rastresito je porozno. Traheje su mu sitne (promjera od 80 do 100 µm), brojne i guste (od 40 do 180 na 1 mm^2 poprečnog presjeka). Raspoređene su pojedinačno, u parovima i kratkim radijalnim nizovima te u skupinama. Perforacija članaka traheja potpuna je. Volumni udio traheja iznosi 22 do 44 %. Staničje drvnih trakova je homogeno. Drvni su traci isključivo jednoredni, visoki od 10 do 15 stanica. Gustoća drvnih trakova je od 8 do 13 na milimetar tangentnog smjera, a udio im je 10 do 14 %. Drvna su vlakanca libriformska, duljine od 0,3 do 2,1 mm. Debljina staničnih stijenki kreće se od 2,2 do 4,7 µm, a širina lumena od 11,5 do 23,5 µm. Udio drvnih vlakanaca je od 56 do 63 %. Uzdužni je parenhim zanemariv. Iako su makroskopski slični, drvo roda *Populus* može se strukturom drvnih trakova mikroskopski razlikovati od drva roda *Salix* (drvo roda *Salix* ima heterocelularne, a drvo roda *Populus* homocelularne drvne trakove).

RELEVANTNE SPOZNAJE O STRUKTURI I VARIJACIJAMA DRVA TOPOLE

Određena anatomska obilježja drva topole poput duljine i promjera drvnih vlakanaca, debljine stanične stijenke (Ehrenberg, 1970.) te udjela različitih elemenata drva (Harris, 1970.) prepoznati su kao važna svojstva već u ranom razvoju programa oplemenjivanja šumskog drveća.

S obzirom na komercijalno rasprostranjenu specifičnu namjenu sirovine, postojanje traheja u drvu mnogih vrsta listača, pa tako i topola, veliki je problem u proizvodnji papira (Lundqvist, 2002.). Iako je uloga traheja u živom stablu nezamjenjiva, iz perspektive proizvodnje papira one su iznimno nepoželjne. U razvijanju vrsta predviđenih za takvu namjenu, osobito pri oplemenjivanju brzorastućih klonova i hibrida topola, treba težiti postizanju što manje veličine i broja traheja. Sve to upućuje na važnost istraživanja koja će obuhvatiti karakteriziranje stanica drva (drvnih vlakanaca i traheja), odnosno određivanje njihovih dimenzija, rasporeda i udjela u strukturi drva.

Pregled istraživanja varijabilnosti unutar i između godova drva topole donosi ove spoznaje: a) da je rast duljine drvnih vlakanaca drva topole u prvih deset do dvadeset godina starosti drveta izrazito pojačan (Boyce i Kaiser, 1961.); b) da za promjer drvnih vlakanaca i tra-

MICROSCOPIC CHARACTERISTICS

Poplar wood is diffuse porous. Vessels are small (diameter 80 to 100 µm), numerous and dense (40 to 180 on 1 mm^2 of cross section). They are distributed individually, in pairs and in short radial rows. Perforation of vessel members is simple. Wood rays are exclusively uniseriate, 10 to 15 cells in height. The density of wood rays is 8 to 13 per tangential mm, and the volume fraction 10 to 14 %. Wood fibres are libriform, with a length of 0.3 to 2.1 mm. The cell wall thickness ranges from 2.2 to 4.7 µm, and lumen diameter from 11.5 to 23.5 µm. The volume fraction of wood fibres is from 56 to 63 %. Axial parenchyma is negligible. Although they are macroscopically similar, wood of the genus *Populus* can be microscopically distinguished from wood of the genus *Salix* according to the structure of the wood rays (wood of the genus *Salix* has heterocellular, and wood of the genus *Populus* homocellular wood rays).

RELEVANT KNOWLEDGE ABOUT POPLAR WOOD STRUCTURE AND VARIATIONS

Certain anatomical characteristics of poplar wood, such as fibre length and fibre diameter, cell wall thickness (Ehrenberg, 1970) and proportions of different wood elements (Harris, 1970) were recognized as important already in the early development of forest tree breeding programmes.

Considering the commercially widespread specific use of the raw material, in many hardwood species, such as poplar, the presence of vessels represents a major problem in paper production (Lundqvist, 2002). Although their function in the living tree is indispensable, from the perspective of paper production they are extremely undesirable. When developing species intended for such a purpose, especially when breeding fast-growing poplar clones and hybrids, the aim should be to achieve the smallest possible size and number of vessels. All the above points to the importance of research that includes the characterization of wood cells (fibres and vessels), that is, the determination of their dimensions, distribution and proportion in the wood structure.

A review of the research on the intra- and inter-ring variability of poplar wood provides knowledge on: (a) a pronounced growth in the fibre length of poplar wood in the first ten to twenty years of age (Boyce heja te za debljine njihovih stijenki vrijedi trend suprotan onome koji se odnosi na rast duljine vlakanaca (Bendtsen, 1978.); c) da se broj traheja u smjeru od srčike prema kori smanjuje, a njihov promjer između godova u istom smjeru raste (Kroll i dr., 1992.); d) da su udjeli stijenki stanica drva, udjeli lumena traheja i drvnih vlakanaca te udjeli drvnih trakova pokazali najmanje promjene duž radijusa (Pezlen, 1994.); e) da su radijalne varijacije određene rastom duljine vlakanaca i starošću drva, dok se dvostruka debljina stijenki i promjer lumena vlakanaca mijenjaju prema promjenjivom modelu među godovima (Ištok i dr., 2017.); f) da je odnos širine godova i duljine te promjera vlakanaca u drvu topole značajno negativan (Zhang i dr., 2022.).

Jedno od najvažnijih svojstava drva za tradicionalnu upotrebu topole jest mali udio tenzijskog drva. To se drvo po nizu biokemijskih, anatomskih i mehaničkih obilježja razlikuje od normalnog drva nastalog zbog nepostojanja mehaničkog podražaja, kao i od suprotnog drva koje se nalazi na donjoj strani nagnutog stabla (Pilate i dr., 2004.). Rane i nešto novije spoznaje o toj temi upućuju na zaključak: a) da je udio nepoželjnoga tenzijskog drva u križancima topole velik (Šefc i dr., 2009.); b) da su želatinozna vlakanca sadržana u tenzijskom drvu relativno uža od vlakanaca u normalnom drvu uočenih na poprečnom presjeku (Jouarez i dr., 2001.); c) da se od normalnog drva do drva s udjelom blagoga i jakog tenzijskog drva smanjila debljina stanične stijenke, isključujući želatinozni sloj (Fang i dr., 2008.); d) da je udio traheja u tenzijskom drvu manji (De Zio i dr., 2020.).

and Kaiser, 1961); (c) decrease in the number of vessels in the direction from the pith to the bark, while their diameter increased with cambial age in the same direction (Kroll *et al*. 1992); (d) proportions of cell walls, proportions of vessel and fibre lumens, and proportions of wood rays that showed the least changes along the radius (Pezlen, 1994); (e) the variation in radial pattern was characterized by increase in fibre length with cambial age, while double cell wall thickness and fibre lumen diameter varied in non-consistent radial pattern (Ištok *et al*. 2017); (f) a significantly negative relationship of tree-ring width to fibre length and fibre width in poplar wood (Zhang *et al*. 2022).

One of the most important wood properties for the traditional use of poplar is a small proportion of tension wood. Tension wood differs from normal wood formed in the absence of mechanical stimulus, and from opposite wood located on the lower side of the inclined stem, in a number of biochemical, anatomical and mechanical characteristics (Pilate *et al*. 2004). Early and more recent knowledge on the topic refers to: (a) large amount of undesired tension wood (Šefc *et al*. 2009); (b) gelatinous fibres present in tension wood were relatively narrower than the opposite wood fibres observed on cross section (Jouarez *et al*. 2001); (c) from normal wood to mild and severe tension wood, the thickness of cell wall excluding G-layer decreased (Fang *et al*. 2008); (d) tension wood was characterized by a low number of vessels (De Zio *et al*. 2020).

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Predani radovi smiju sadržavati najviše 15 jednostrano pisanih A4 listova s dvostrukim proredom (30 redaka na stranici), uključujući i tablice, slike te popis literature, dodatke i ostale priloge. Dulje je članke preporučljivo podijeliti na dva ili više nastavaka. Tekst treba biti u *doc formatu*, u potpunosti napisan fontom *Times New Roman* (tekst, grafikoni i slike), normalnim stilom, bez dodatnog uređenja teksta.

Prva stranica poslanog rada treba sadržavati puni naslov, ime(na) i prezime(na) autora, podatke o zaposlenju autora (ustanova, grad i država) te sažetak s ključnim riječima (duljina sažetka približno 1/2 stranice A4).

Posljednja stranica treba sadržavati titule, zanimanje, zvanje i adresu (svakog) autora, s naznakom osobe s kojom će Uredništvo biti u vezi.

Znanstveni i stručni radovi moraju biti sažeti i precizni. Osnovna poglavlja trebaju biti označena odgovarajućim podnaslovima. Napomene se ispisuju na dnu pripadajuće stranice, a obrojčavaju se susljedno. One koje se odnose na naslov označuju se zvjezdicom, a ostale uzdignutim arapskim brojkama. Napomene koje se odnose na tablice pišu se ispod tablica, a označavaju se uzdignutim malim pisanim slovima, abecednim redom.

Latinska imena trebaju biti pisana kosim slovima (italicom), a ako je cijeli tekst pisan kosim slovima, latinska imena trebaju biti podcrtana.

U uvodu treba definirati problem i, koliko je moguće, predočiti granice postojećih spoznaja, tako da se čitateljima koji se ne bave područjem o kojemu je riječ omogući razumijevanje ciljeva rada.

Materijal i metode trebaju biti što preciznije opisane da omoguće drugim znanstvenicima ponavljanje pokusa. Glavni eksperimentalni podaci trebaju biti dvojezično navedeni.

Rezultati trebaju obuhvatiti samo materijal koji se izravno odnosi na predmet. Obvezatna je primjena metričkog sustava. Preporučuje se upotreba SI jedinica. Rjeđe rabljene fizikalne vrijednosti, simboli i jedinice trebaju biti objašnjeni pri njihovu prvom spominjanju u tekstu. Za pisanje formula valja se koristiti Equation Editorom (programom za pisanje formula u MS Wordu). Jedinice se pišu normalnim (uspravnim) slovima, a fizikalni simboli i faktori kosima (italicom). Formule se susljedno obrojčavaju arapskim brojkama u zagradama, npr. (1) na kraju retka.

Broj slika mora biti ograničen samo na one koje su prijeko potrebne za objašnjenje teksta. Isti podaci ne smiju biti navedeni i u tablici i na slici. Slike i tablice trebaju biti zasebno obrojčane, arapskim brojkama, a u tekstu se na njih upućuje jasnim naznakama ("tablica 1" ili "slika 1"). Naslovi, zaglavlja, legende i sav ostali tekst u slikama i tablicama treba biti napisan hrvatskim i engleskim jezikom.

Slike je potrebno rasporediti na odgovarajuća mjesta u tekstu, trebaju biti izrađene u rezoluciji 600 dpi, crno-bijele (objavljivanje slika u koloru moguće je na zahtjev autora), formata jpg ili tiff, potpune i jasno razumljive bez pozivanja na tekst priloga.

Svi grafikoni i tablice izrađuju se kao crno-bijeli prilozi (osim na zahtjev). Tablice i grafikoni trebaju biti na svojim mjestima u tekstu te originalnog formata u kojemu su izrađeni radi naknadnog ubacivanja hrvatskog prijevoda. Ako ne postoji mogućnost za to, potrebno je poslati originalne dokumente u formatu u kojemu su napravljeni (*excel* ili *statistica* format).

Naslovi slika i crteža ne pišu se velikim tiskanim slovima. Crteži i grafikoni trebaju odgovarati stilu časopisa (fontovima i izgledu). Slova i brojke moraju biti dovoljno veliki da budu lako čitljivi nakon smanjenja širine slike ili tablice. Fotomikrografije moraju imati naznaku uvećanja, poželjno u mikrometrima. Uvećanje može biti dodatno naznačeno na kraju naslova slike, npr. "uvećanje 7500 : l".

Diskusija i zaključak mogu, ako autori žele, biti spojeni u jedan odjeljak. U tom tekstu treba objasniti rezultate s obzirom na problem postavljen u uvodu i u odnosu prema odgovarajućim zapažanjima autora ili drugih istraživača. Valja izbjegavati ponavljanje podataka već iznesenih u odjeljku *Rezultati*. Mogu se razmotriti naznake za daljnja istraživanja ili primjenu. Ako su rezultati i diskusija spojeni u isti odjeljak, zaključke je nužno napisati izdvojeno. Zahvale se navode na kraju rukopisa. Odgovarajuću literaturu treba citirati u tekstu, i to prema harvardskom sustavu (*ime – godina*), npr. (Bađun, 1965). Nadalje, bibliografija mora biti navedena na kraju teksta, i to abecednim redom prezimena autora, s naslovima i potpunim navodima bibliografskih referenci. Popis literature mora biti selektivan, a svaka referenca na kraju mora imati naveden DOI broj, ako ga posjeduje (http://www.doi.org) (provjeriti na http://www.crossref.org).

Primjeri navođenja literature

Članci u časopisima: Prezime autora, inicijal(i) osobnog imena, godina: Naslov. Naziv časopisa, godište (ev. broj): stranice (od – do). Doi broj.

Primjer

Kärki, T., 2001: Variation of wood density and shrinkage in European aspen (*Populus tremula*). Holz als Roh- und Werkstoff, 59: 79-84. http://dx.doi.org/10.1007/s001070050479.

Knjige: Prezime autora, inicijal(i) osobnog imena, godina: Naslov. (ev. izdavač/editor): izdanje (ev. svezak). Mjesto izdanja, izdavač (ev. stranice od – do).

Primjeri

Krpan, J., 1970: Tehnologija furnira i ploča. Drugo izdanje. Zagreb, Tehnička knjiga.

Wilson, J. W.; Wellwood, R. W., 1965: Intra-increment chemical properties of certain western Canadian coniferous species. U: W. A. Cote, Jr. (Ed.): Cellular Ultrastructure of Woody Plants. Syracuse, N.Y., Syracuse Univ. Press, pp. 551- 559.

Ostale publikacije (brošure, studije itd.)

Müller, D., 1977: Beitrag zür Klassifizierung asiatischer Baumarten. Mitteilung der Bundesforschungsanstalt für Forstund Holzvvirt schaft Hamburg, Nr. 98. Hamburg: M. Wiederbusch.

Web stranice

***1997: "Guide to Punctuation" (online), University of Sussex, www.informatics.sussex.ac.uk/department/docs/punctuation/node00.html. First published 1997 (pristupljeno 27. siječnja 2010).

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Kärki, T., 2001: Variation of wood density and shrinkage in European aspen (*Populus tremula*). Holz als Roh- und Werkstoff, 59: 79-84. http://dx.doi.org/10.1007/s001070050479.

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Examples:
Krpan, J. 1970: Tehnologija furnira i ploča. Drugo izdanje. Zagreb: Tehnička knjiga.
Wilson, J.W.; Wellwood, R.W. 1965: Intra-increment chemical properties of certain western Canadian coniferous species. U: W. A. Cote, Jr. (Ed.): Cellular Ultrastructure of Woody Plants. Syracuse, N.Y., Syracuse Univ. Press, pp. 551-559.

Other publications (brochures, studies, etc.):
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***1997: "Guide to Punctuation" (online), University of Sussex, www.informatics.sussex.ac.uk/department/docs/punctuation/node00.html. First published 1997 (Accessed Jan. 27, 2010).

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HRVATSKA KOMORA INŽENJERA ŠUMARSTVA I DRVNE TEHNOLOGIJE

HRVATSKA KOMORA INŽENJERA ŠUMARSTVA I DRVNE TEHNOLOGIJE

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