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SCIENTIFIC JOURNAL
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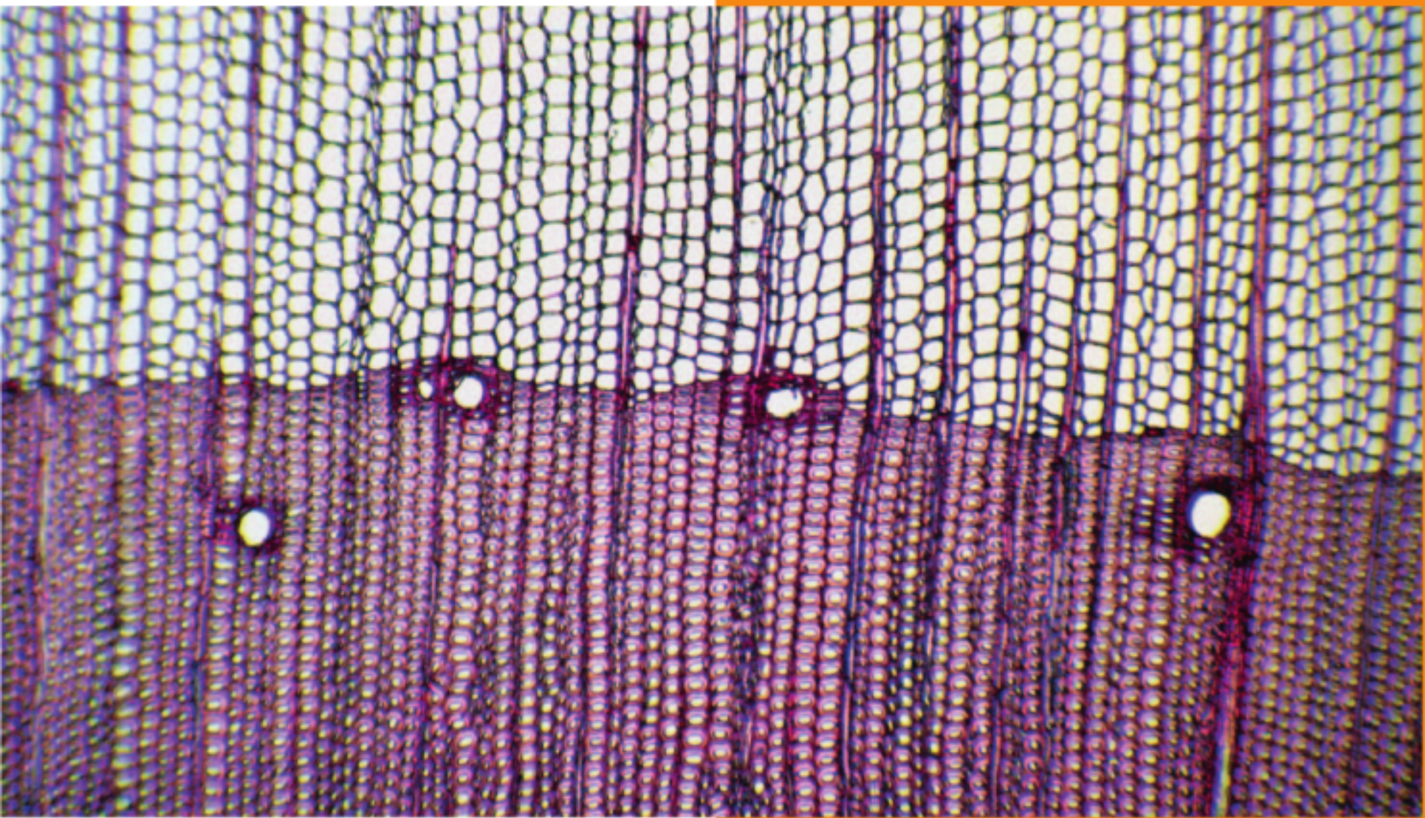

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Effects of CNC Processing Parameters on Surface Quality of Wood-Based Panels Used in Furniture Industry

Utjecaj parametara CNC obrade na kvalitetu površine drvnih ploča koje se upotrebljavaju u industriji namještaja

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ABSTRACT • *The processing of wood-based panels such as plywood, particleboard and fiberboard, which are widely used in the furniture industry, with CNC (Computer Numerical Control) milling machines has been increasing recently. Even though CNC milling machines have many advantages for furniture producers, it is difficult to set process parameters to obtain the desired surface quality of the material. Therefore, it is necessary to determine the most suitable of these parameters for the surface quality of each wood-based panel. This study aimed to determine the effects of processing parameters on the surface quality of plywood, particleboard and medium density fiberboard (MDF) panels processed in CNC milling machines. Furthermore, the average surface roughness values of these panels were compared after CNC processing. Three spindle rotational frequencies (10.000, 14.000 and 18.000 rpm), three feed rates (5, 7, and 9 m/min) and two cutting tool diameters (2 and 5 mm) were selected as CNC processing parameters. To determine the surface quality of wood-based panels, the surface roughness measurements were performed according to DIN 4768 standard and three surface roughness parameters (R_a, R_{max} and R_z) were determined. According to the results of this study, it can be concluded that the surface roughness values of wood-based panels decreased with increasing spindle rotational frequency and feed rate, while they increased with increasing cutting tool diameter. Among the wood-based panels used in this study, the lowest average roughness values were obtained for plywood samples.*

KEYWORDS: *wood-based panel; CNC milling machine; surface roughness; processing parameters*

SAŽETAK • *Za obradu ploča na bazi drva kao što su furnirske ploče, iverice i vlaknatice, koje imaju široku primjenu u industriji namještaja, u posljednje se vrijeme sve češće primjenjuju CNC (Computer Numerical Control) glodalice. Iako CNC glodalice imaju mnoge prednosti za proizvođače namještaja, teško je odrediti parametre procesa za postizanje željene kvalitete površine obrađivanog materijala. Stoga je potrebno odrediti najprikladnije parametre obrade za svaku vrstu ploče na bazi drva. Cilj ovog istraživanja bio je utvrditi utjecaj parametara obra-*

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de CNC glodalicama na kvalitetu površine furnirske ploče, iverice i ploče vlaknatice srednje gustoće (MDF ploče). Nadalje, uspoređene su srednje vrijednosti hrapavosti površine tih ploča nakon CNC obrade. Kao parametri CNC obrade odabrane su tri frekvencije vrtnje vretena (10 000, 14 000 i 18 000 okr./min), tri posmične brzine (5, 7 i 9 m/min) te dva promjera reznog alata (2 i 5 mm). Za određivanje kvalitete površine ploča na bazi drva provedena su mjerenja hrapavosti površine prema normi DIN 4768, a hrapavost je iskazana trima parametrima hrapavosti površine (R_a, R_{max} i R_z). Prema rezultatima ovog istraživanja može se zaključiti da su se vrijednosti hrapavosti površine ploča na bazi drva smanjivale s povećanjem frekvencije vrtnje vretena i posmične brzine, dok su se povećavale s povećanjem promjera reznog alata. Među pločama na bazi drva na kojima je provedeno ovo istraživanje najniže srednje vrijednosti hrapavosti dobivene su za uzorke furnirske ploče.

KLJUČNE RIJEČI: *ploče na bazi drva; CNC glodalica; hrapavost površine; parametri obrade*

1 INTRODUCTION
1. UVOD

Wood-based panels such as plywood, particleboard and fibreboard, especially medium density fiberboard (MDF), are intensely used in furniture production and interior decoration (Akbulut and Ayrilmis, 2019). The production of wood-based panels in Europe (+EFTA) grew by 3 % in 2018 to 57.6 million cubic metres. The fastest growth was in plywood, and particleboard was the largest category by volume (32 million cubic metres), followed by MDF (12.3 million cubic metres) (European Panel Federation, 2018). Turkey is now one of the world's top wood-based panel manufacturers. Turkey is ranked first in the production of MDF in Europe and second in the world, and third in Europe and fourth in the world in the production of particleboard (ORSIAD, 2019). In 2018, the production quantity of particleboard, MDF and plywood in Turkey was 4.36, 4.91 million cubic metres, and 112.000 cubic metres, respectively (FAO, 2020). It is known that among the panels produced every year, approximately 80 % of the particleboard and 70 % of the MDF panels are used in the furniture industry, and the furniture market in Turkey is growing rapidly due to the increase in the amount of wood-based panel production. Furthermore, it was stated that plywood panels are frequently used in both construction and furniture industries (Ferreira *et al.*, 2017).

The application of high technology such as CNC machines and automation systems is one of the main reasons of the growth of the furniture industry. In the furniture industry, CNC machines have been preferred considerably in the processes such as patterning, milling, drilling and grooving (Sofuoglu, 2017). As the integration of CNC machines with other automation systems is very flexible, time loss in furniture production decreases and productivity increases 2.5 times (Koc *et al.,* 2017). Moreover, they improve surface quality of the materials and reduce labour cost (Sutcu, 2013). The surface quality of the processed materials is one of the most important factors affected by finishing processes such as coating, painting and varnishing, machining properties, mechanical properties such as adhesion strength and bending strength of wood-based panels (Sofuoglu, 2017; Zhong *et al.,* 2013). Surface roughness measurement is the most important quality control tool that determines the surface quality of the processed materials. The surface roughness of wood and wood-based panels is affected by many factors. The most important of these factors are the anatomical and physical properties of wood materials such as wood species, density, hardness, moisture content, fiber direction (Tabarsa *et al*., 2011). Moreover, the main operational machining parameters such as spindle rotational frequency, depth of cut, tool sharpness, cutting circle radius, cutting direction, cutting angle and vibration are among the factors that significantly affect surface roughness values (Csanády *et al*., 2015).

Many different parameters have to be adjusted on the CNC machines for the processing of material using code file. These are the spindle rotational frequency, feed rate, cutter step over, cutter plunge speed, tool strategy, etc. (Bal and Akcakaya, 2018). Most of the problems that occur in the processing of wood and based panels are caused by errors in setting the appropriate parameters. Processing parameters adjusted correctly are the most important factors that increase the surface quality of wood and wood-based boards (Koc *et al.,* 2017). For this reason, many researchers have studied the effects of CNC processing parameters such as spindle rotational frequency, step over, feed rate, cutting tool diameter and depth of cut on surface roughness of solid wood (Gawronski, 2013; Hazir and Koc, 2019; Iskra and Hernández, 2009; Sofuoglu, 2017; Sutcu and Karagoz, 2013) and MDF (Koc *et al.,* 2017; Bal, 2018; Davim *et al.,* 2009; Deus *et al.,* 2018; İşleyen and Karamanoğlu, 2019) processed with CNC machines. In general, it was found in these studies that higher values of spindle rotational frequency caused smoother surface of material whereas higher values of feed rate, step over and depth of cut resulted in the increased surface roughness of materials.

There is no information about the influence of CNC processing parameters on the surface quality of plywood and particleboard while there are many studies on solid wood and MDF in the literature. The aim of this study was to determine the effects of processing parameters on the surface quality of plywood, particleboard and MDF panels processed in CNC milling machines. In addition, the average values of surface roughness of these panels were compared after CNC processing.

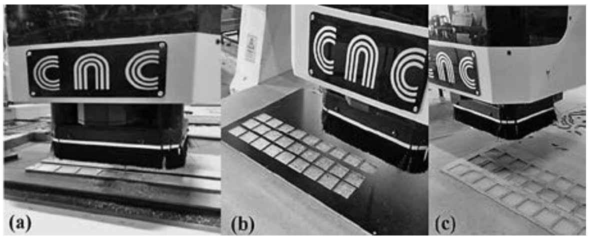

Figure 1 Grooving of plywood (a), particleboard (b) and MDF (c) panels with CNC machine
Slika 1. Izrada utora na furnirskoj ploči (a), ploči iverici (b) i MDF-u (c) CNC strojem

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

Particleboard and MDF panels with the thickness of 18 mm and five-ply birch plywood panels with the thickness of 10 mm have been obtained from a supplier as research materials in the study, because these wood-based panel products are largely used as raw materials in the furniture industry in Turkey. Test samples with the dimensions of 50 cm × 110 cm (width × length) were prepared from the particleboard, MDF and plywood panels. Before the processing with CNC machine, the samples of panels were conditioned at (20 ± 2) °C and (65 ± 5) % relative humidity until they reached a moisture content of (12 ± 1) % in a climate chamber.

2.2 CNC machine and cutting conditions
2.2. CNC stroj i uvjeti obrade

The samples of panels were processed on Megatron 2128, four-axis CNC Milling Machine with 9 kW spindle power, and a maximum rotational frequency of 24.000 rpm. The processing of panel samples with CNC machine is shown in Figure 1.

Three spindle rotational frequencies (10.000, 14.000, and 18.000 rpm) and feed rates (5, 7, and 9 m/min) were applied for CNC processing. Solid carbide barrel milling cutters in conical form were also used with two different diameters (2 mm and 5 mm). The tool dimensions of cutters are presented in Figure 2. The cutting depth for plywood was 2 mm and 10 mm for particleboard and MDF.

2.3 Surface roughness measurements
2.3. Mjerenje hrapavosti površine

Five specimens (5 cm × 5 cm) were obtained from panel groups and five measurements were made for each specimen. Mitutoyo Surftest SJ-301 Surface Roughness Tester was used for the surface roughness tests. Cut-off length was 2.5 mm, sampling length was 12.5 mm and detector tip radius was 5 μm in the surface roughness measurements. The measurements were made perpendicular to the fiber direction of the plywood samples, whereas the fiber direction was not taken into account in the particleboard and MDF samples due to their homogeneous structure. Three roughness parameters R_a (absolute arithmetic mean), R_{max} (maximum two-point height of profile) and R_z (arithmetic mean of the 10-point height of irregularities) were measured to evaluate surface roughness of the sample surfaces according to DIN 4768 standard (1990).

Analysis of variance (ANOVA) was performed for the statistical evaluation of changes in the surface

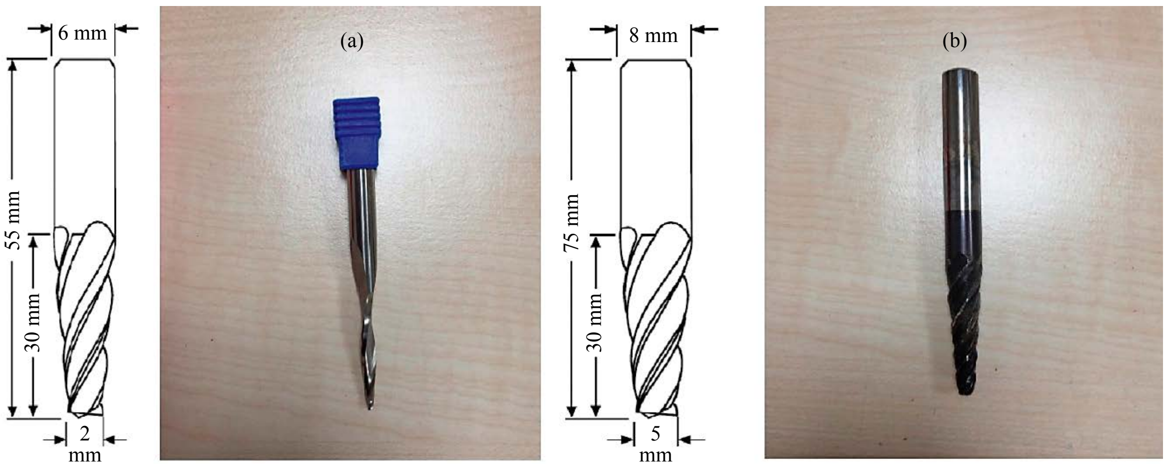

Figure 2 Cutting tool dimensions of 2 (a) mm and 5 (b) mm diameters
Slika 2. Dimenzije reznog alata promjera 2 mm (a) i 5 mm (b)

roughness parameters (R_a, R_{max} and R_z) depending on the cutting tool diameter, spindle rotational frequency and feed rate. After ANOVA, Student–Newman–Keuls test with 95 % confidence level was used to compare the mean values of variance sources.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

The average values and standard deviation of R_a, R_{max}, and R_z parameters of surface roughness are given in Figures 3 and 4. The lowest R_a, R_{max}, and R_z values

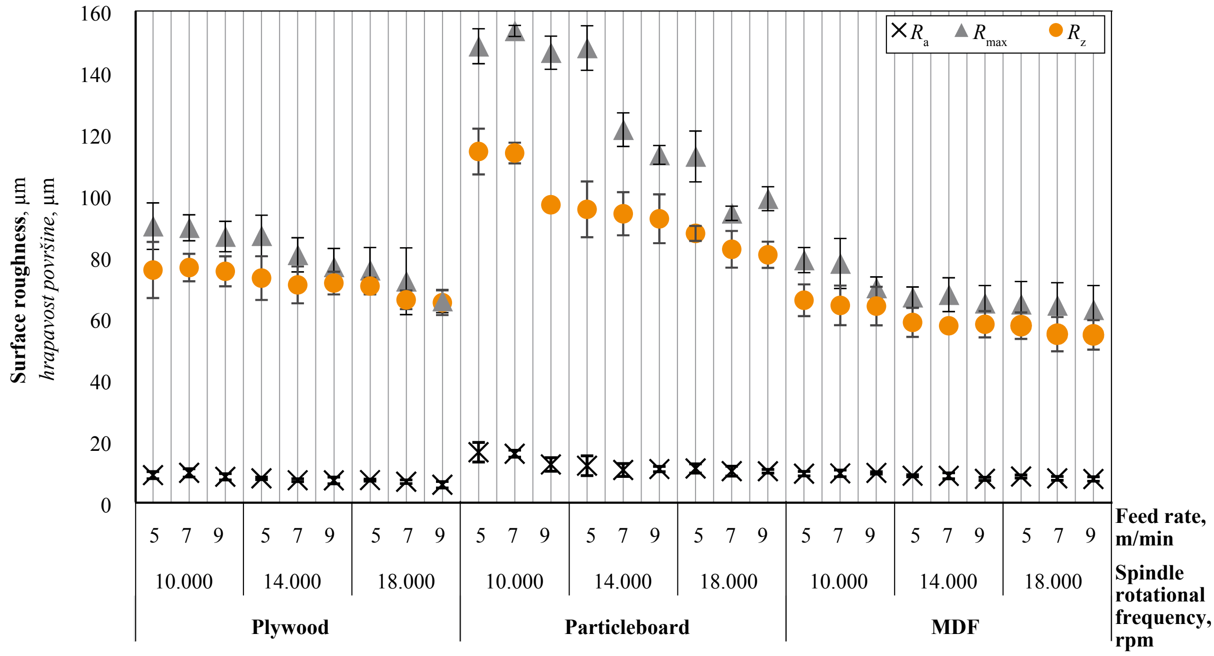

Figure 3 Variation of surface roughness values of wood-based panels processed with 2 mm diameter cutting tool according to CNC cutting parameters
Slika 3. Odstupanja vrijednosti hrapavosti površine ploča na bazi drva obrađenih reznim alatom promjera 2 mm s obzirom na parametre CNC obrade

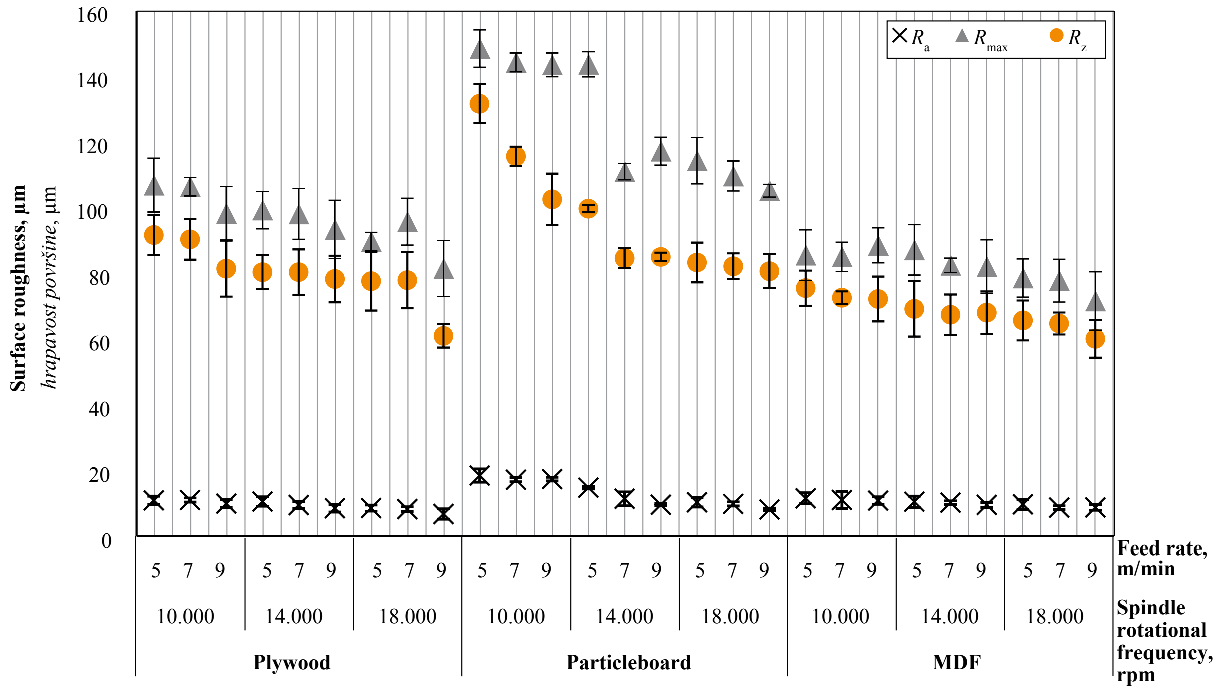

Figure 4 Variation of surface roughness values of wood-based panels processed with 5 mm diameter cutting tool according to CNC cutting parameters
Slika 4. Odstupanja vrijednosti hrapavosti površine ploča na bazi drva obrađenih reznim alatom promjera 5 mm s obzirom na parametre CNC obrade

were obtained with 2 mm diameter cutting tool, 18.000 rpm spindle rotational frequency and 9 m/min feed rate for all three panel samples. However, the highest values were obtained with 5 mm diameter cutting tool, 10.000 rpm spindle rotational frequency and 5 m/min feed rate for all panel groups. When the data of surface roughness were analysed, higher surface roughness values were obtained with 5 mm diameter cutting tool than with 2 mm diameter cutting tool.

ANOVA tables have been compiled with the average surface roughness obtained from the experiments. The effects of related parameters on surface roughness have been determined. The results of F and Significant Levels (Sig.) of variance analysis are given in Table 1. The Sig. values lower than 0.05 show that the factors and interaction of the factors are statistically significant. Multiple comparison test results (Student–Newman–Keuls) for surface roughness parameter values (R_a, R_{max} and R_z) are given in Table 2.

When analysing the results of Table 2, it can be seen that the surface roughness values (R_a, R_{max} and R_z) for all of the panel types decreased with increasing values of spindle rotational frequency. In the literature, the reasons for changes in spindle rotational frequency were determined by some researchers (Pelit *et al.*, 2021; Deus *et al.*, 2015; Valarmathi *et al.*, 2013). Pelit *et al.* (2021) stated that the contact between the cutting edge of the tool and the cut surface increased with the increase of spindle rotation frequency, and hence the surface roughness values of wood materials decreased. Deus *et al.* (2015) explained that each cutting tooth removes less material at high spindle rotational frequency where the feed rate is constant, and thus less rough material surfaces can be obtained. Moreover, Valarmathi *et al.* (2013) stated that, with the increase of spindle rotational frequency, the processed material heats up and its surface softens and becomes smoother. As a result of these effects, surface roughness decreases. Similar results have been obtained in many studies on the effects of spindle rotation frequency on wood and chipboard surface roughness. Similar results were obtained in many studies regarding the effects of spindle rotation frequency on the surface roughness of wood and fiberboard (Sofuoglu, 2017; Hazir and Koc, 2019; Deus *et al.,* 2015; Prakash *et al.,* 2011; Sedlecký *et al.,* 2018; Sutcu and Karagoz, 2012). In this study, the spindle rotational frequency at 18.000 rpm provided the smallest surface roughness values for all wood-based panel types.

Similarly, the surface roughness parameters decreased statistically with increasing feed rate for the plywood and particleboard panels. In the MDF panels, although the effect of feed rate on R_{max} and R_z roughness values was not statistically significant, it was ob-

Table 1 ANOVA test results for R_a, R_{max} and R_z values of wood-based panel types
Tablica 1. Rezultati ANOVA testiranja vrijednosti R_a, R_{max} i R_z za ploče na bazi drva

Panel types *Vrsta ploče*	Source of variance *Izvor odstupanja*	R_a		R_{max}		R_z	
		F	Sig.	*F*	Sig.	*F*	Sig.
Plywood *furnirska ploča*	A: Tool diameter / *promjer alata*, mm	93.477	0.000	129.753	0.000	44.463	0.000
	B: Spindle rotational frequency *frekvencija rotacije vretena*, mm^{-1}	45.903	0.000	40.764	0.000	28.501	0.000
	C: Feed rate / *posmična brzina*, m/min	12.219	0.000	11.716	0.000	8.034	0.001
	A*B	1.265	0.289	0.194	0.824	2.393	0.099
	A*C	0.872	0.423	0.898	0.412	4.236	0.018
	B*C	1.208	0.315	0.910	0.463	1.684	0.163
	A*B*C	0.349	0.844	0.418	0.795	0.987	0.420
Particleboard *ploča iverica*	A: Tool diameter / *promjer alata*, mm	13.671	0.000	0.219	0.641	1.359	0.248
	B: Spindle rotational frequency *frekvencija rotacije vretena*, mm^{-1}	119.143	0.000	568.722	0.000	240.809	0.000
	C: Feed rate / *posmična brzina*, m/min	15.890	0.000	91.551	0.000	39.536	0.000
	A*B	10.280	0.000	14.792	0.000	11.220	0.000
	A*C	0.593	0.555	1.454	0.240	5.013	0.009
	B*C	1.088	0.369	31.039	0.000	9.655	0.000
	A*B*C	3.697	0.009	6.081	0.000	2.974	0.025
MDF	A: Tool diameter / *promjer alata*, mm	67.827	0.000	113.898	0.000	66.810	0.000
	B: Spindle rotational frequency *frekvencija rotacije vretena*, mm^{-1}	19.846	0.000	19.887	0.000	23.156	0.000
	C: Feed rate / *posmična brzina*, m/min	2.922	0.060	2.914	0.061	1.807	0.171
	A*B	0.760	0.471	2.017	0.140	0.337	0.715
	A*C	0.305	0.738	0.142	0.868	0.187	0.830
	B*C	0.676	0.611	0.345	0.847	0.311	0.869
	A*B*C	0.156	0.960	2.175	0.080	0.144	0.965

Table 2 Results of Student-Newman-Keuls test at 95 % confidence level for CNC processing parameters
Tablica 2. Rezultati Student-Newman-Keulsova testa za razinu pouzdanosti od 95 % za parametre CNC obrade

Panel types *Vrsta ploče*	**Factors** / *Čimbenici*	R_a		R_{max}		R_z	
		LS Mean	**HG***	**LS Mean**	**HG**	**LS Mean**	**HG**
Plywood *furnirska ploča*	Tool diameter / *promjer alata,* mm						
	2	8.15	a	81.04	a	72.04	a
	5	10.26	b	97.45	b	80.79	b
	Spindle rotational frequency *frekvencija vrtnje vretena,* min^{-1}						
	10.000	10.50	c	96.92	c	82.49	c
	14.000	9.18	b	89.81	b	76.41	b
	18.000	7.94	a	81.01	a	70.35	a
	Feed rate / *posmična brzina,* m/min						
	5	9.71	b	92.03	b	78.82	b
	7	9.44	b	91.39	b	77.67	b
	9	8.46	a	84.33	a	72.75	a
Particleboard *ploča iverica*	Tool diameter / *promjer alata,* mm						
	2	12.65	a	126.73	a	95.72	a
	5	13.93	b	127.20	a	97.04	a
	Spindle rotational frequency *frekvencija vrtnje vretena,* min^{-1}						
	10.000	16.98	c	148.08	c	113.13	c
	14.000	12.24	b	126.37	b	92.53	b
	18.000	10.65	a	106.46	a	83.48	a
	Feed rate / *posmična brzina,* m/min						
	5	14.53	c	136.57	b	102.67	c
	7	13.21	b	122.97	a	96.11	b
	9	12.13	a	121.37	a	90.36	a
MDF	Tool diameter / *promjer alata,* mm						
	2	9.10	a	69.05	a	59.91	a
	5	11.02	b	83.42	b	69.28	b
	Spindle rotational frequency *frekvencija vrtnje vretena,* min^{-1}						
	10.000	11.02	c	81.63	c	69.72	c
	14.000	9.93	b	75.83	b	63.82	b
	18.000	9.23	a	71.25	a	60.25	a
	Feed rate / *posmična brzina,* m/min						
	5	10.42	b	77.60	a	66.08	a
	7	10.03	ab	77.16	a	64.22	a
	9	9.73	a	73.95	a	63.49	a

*Homogeneity Groups: different letters denote a statistically significant difference. / *Homogene grupe: različita slova označavaju statistički značajnu razliku.*

served that Ra roughness values of the whole group decreased with increasing feed rate except for the samples processed with 2 mm diameter cutting tool at 10.000 rpm spindle rotational frequency. Davim *et al.* (2015) suggested that the last low feed rate values reduce the stress on the tool, contribute to the prevention of grooves on the surface of the wood and increase the processing efficiency. In the literature, there are many studies showing different relationships between the feed rate and the surface roughness values of wood materials. Some researchers found that the surface roughness of processed materials decreased with increasing feed rate (Gawronski, 2013; Davim *et al.,* 2009; Sutcu and Karagoz, 2012), while others found that the surface roughness of processed materials increased with increasing feed rate (Koc *et al.,* 2017; Isleyen and Karamanoglu, 2019; Deus *et al.,* 2015). Sutcu (2013) stated that the effect of feed rate on surface roughness is statistically insignificant. In this study, the same results were obtained for the R_{max} and R_z values of the MDF panels (Table 2). The differences between the results in literature are thought to be due to the selected feed rate values and the differences between them. Generally, 9 m/min feed rate presented the smallest surface rough-

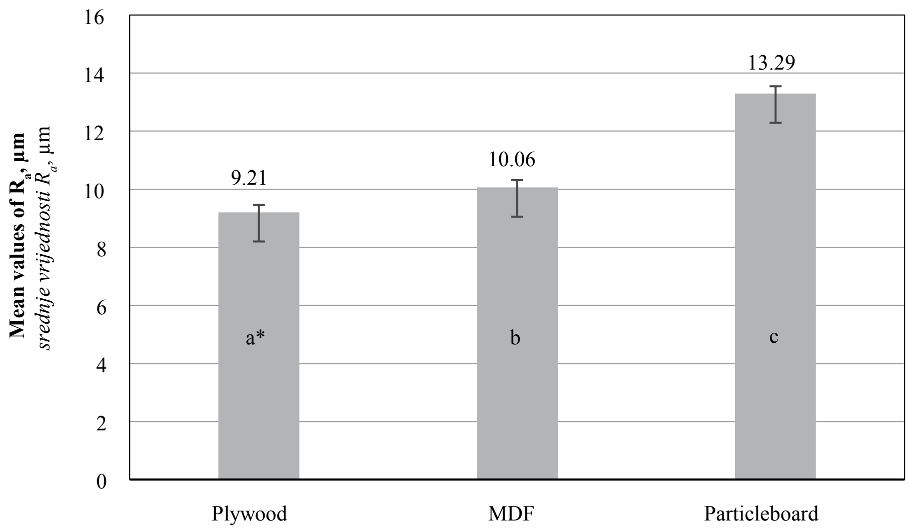

Figure 5 Comparison of wood-based panel types processed with CNC machine (*Different letters denote a statistically significant difference)
Slika 5. Usporedba vrijednosti hrapavosti ploča na bazi drva obrađenih CNC strojem (*različita slova označavaju statistički značajnu razliku)

ness for the wood-based panels in this study. Similarly, the use of high feed rates in wood milling has been strongly recommended by Očkajová *et al.* (2020).

Even though the most influencing CNC processing parameters on the surface roughness are spindle rotational frequency and feed rate, tool geometry and tool diameter also have influence on the processing of composites (Prakash *et al.,* 2011). As can be seen from Table 2 and Figure 4, the surface roughness values increased as the cutting tool diameter increased for the plywood and MDF panel groups. The same results were obtained from the R_a values of particleboard panel groups. However, there was no statistically significant effect of tool diameter on the R_{max} and R_z values of the particleboard panels. İsleyen and Karamanoğlu (2019) stated that the friction area between the tool and the material increased with the increase of the tool diameter, and hence the surface roughness values of the materials increased. Many researchers determined that cutting tool diameter was an effective factor in all the wood machining processes and the smallest roughness values were obtained from lower tool diameter values (Isleyen and Karamanoglu, 2019; Prakash *et al.,* 2011). In this study, it was determined that the plywood and MDF samples processed with 2 mm diameter cutting tool had the lowest surface roughness values.

The average roughness values (R_a) of the panel types processed with CNC machine were statistically analysed according to the panel types. The comparison results of the panel types are shown in Figure 5.

As can be seen from Figure 5, the plywood processed with CNC machine has the smoothest surfaces of all other panel types. The highest surface roughness values were measured at the particleboard samples processed with CNC machine. Since the chips have a rough surface compared to the peeling veneers and fibers, the surface roughness values of the particleboards are also expected to be high. However, Zhong *et al.* (2013) determined surface roughness values of plywood, particleboard, MDF and solid wood manufactured from different wood species in Singapore. According to the results of that study, the R_a values of MDF panel were found to be the highest among the wood-based panels. Moreover, some researchers compared the surface roughness values of particleboard and MDF panels and found that MDF panels had smoother surfaces than particleboard (Hiziroglu *et al.,* 2004; Ulker, 2018). The various factors such as wood species, humidity, wood processing, measurement methods can cause the differences in the results obtained from the studies.

4 CONCLUSIONS
4. ZAKLJUČAK

This study was aimed to determine the effects of process parameters on the surface roughness values after CNC machine processing of plywood, particleboard and MDF panels, which are frequently used in the furniture industry. The findings obtained from this study are given below.

The CNC processing parameters such as tool diameter, spindle rotational frequency and feed rate are very important factors in terms of surface roughness values (R_a, R_{max} and R_z) of plywood and particleboard as well as MDF panels.

The surface roughness values of the plywood and MDF panels processed with 2 mm diameter cutting tool were found to be higher than those processed with 5 mm diameter cutting tool. Although similar results were found for R_a values of particleboard groups, no statistical difference was found in other surface roughness parameter values (R_{max} and R_z) depending on the increase in tool diameter values.

The higher spindle rotational frequency values caused the smoothest surface on all of panel groups. Therefore, the selected spindle rotational frequency of 18.000 rpm was the best for the surface quality of these boards.

The surface roughness values of the plywood and particleboard panel groups decreased with increasing feed rate values. The smoothest surfaces for these panels were obtained at the feed rate of 9 m/min. Although similar results were found for R_a values of MDF panel groups, no statistical difference was found in other surface roughness parameter values (R_{max} and R_z) depending on the increase in feed rate values.

When comparing the R_a values measured on the panel samples after processing with CNC machine, the plywood groups presented the smoothest surfaces. The highest R_a values were obtained for the particleboard samples.

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Safety Stresses of Red Pine Wood According to Site Index Grade

Dopuštena naprezanja drva crvenog bora ovisno o indeksu staništa

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ABSTRACT • *One of the most important indicators for using wooden materials as a building material is safety stress (SS). Site index is also an essential criterion for construction materials. This research was planned with the aim to reveal the relationship between safety stress and site index classes (I, II, III) of red pine wood, which is an important tree species of Turkey and the Mediterranean basin. Also, the safety coefficient was calculated. The safety stress tests for compression, bending, and tensile, tensile perpendicular to fibers, cleavage, and shearing strengths were calculated as 9.2, 9.7, 8.9, 0.35, 0.10, and 1.4 N/mm², respectively. The statistical analyses indicated that the site index difference had a significant effect on the safety stress in red pine wood as mentioned above. Again, the safety coefficient was calculated as 5.27 for red pine wood. In addition, it was determined that the safety stress values of red pine wood provided the desired lower limit values according to the standard (TS 647 and EN 1995-1-1), excluding class I. As a result of the regression and correlation analyses, the presence of a moderately increasing linear relationship (R^2 values equal to 0.41-0.68) was found between the density and safety stress values for all site indexes.*

KEYWORDS: *red pine; site index; mechanical properties; safety stress; safety coefficient*

SAŽETAK • *Jedan od najvažnijih pokazatelja za uporabu drva kao građevnog materijala jest njegovo dopušteno naprezanje. A za drvo kao građevni materijal bitan je i kriterij indeks staništa. Ovo je istraživanje poduzeto radi otkrivanja odnosa između dopuštenog naprezanja i razreda indeksa staništa (I., II., III.) drva crvenog bora, važne vrste drva u Turskoj i na području mediteranskog bazena. Usto je izračunan koeficijent sigurnosti. Ispitivanjem su dobivena dopuštena naprezanja na tlak, savijanje i vlak, dopuštena vlačna naprezanja okomito na vlakanca te naprezanja na cijepanje i smicajna naprezanja, koja su redom iznosila 9,2; 9,7; 8,9; 0,35; 0,10 i 1,4 N/mm². Statistička je analiza pokazala da razlika u indeksu staništa ima znatan učinak na dopušteno naprezanje drva crvenog bora, kao što je i navedeno. Za drvo crvenog bora izračunani koeficijent sigurnosti iznosi 5,27. Osim toga, utvrđeno je da vrijednosti dopuštenog naprezanja drva crvenog bora osiguravaju željene donje granične vrijednosti prema standardu TS 647 odnosno EN 1995-1-1, isključujući razred I. Kao rezultat regresijske i korelacijske analize utvrđeno je postojanje umjereno rastućega linearnog odnosa (vrijednosti R^2 iznose 0,41 – 0,68) između vrijednosti gustoće drva i dopuštenog naprezanja za sve indekse staništa.*

KLJUČNE RIJEČI: *drvo crvenog bora; indeks staništa; mehanička svojstva; dopušteno naprezanje; koeficijent sigurnosti*

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1 INTRODUCTION
1. UVOD

Wood is a natural organic material that has many characteristics that make it suitable for constructing buildings. It has been used for many centuries in construction, bridges, and various other structures (Kržišnik *et al.,* 2020; Kathem *et al*., 2014; Harte, 2009). The wood material may also be particularly efficient in certain structural forms based on its mechanical properties (Yildrim *et al*., 2021; Ramage *et al*., 2016). Although concrete and steel are the most popular materials in construction around the world, lumber was relegated to minor structural uses (Echenique Racero *et al*., 2015). The wood material has very high strength, especially when compared with its low weight. However, it is very anisotropic with different properties in different directions due to its make-up of oriented fibers (Kathem *et al*., 2014). Ramage *et al*. (2016) confirmed that timber excels where strength (or stiffness) to weight is more important than absolute strength (or stiffness). Good quality material is one of the basic requirements to manufacture a strong building (Boen, 2014). Until recently, safety checking requirements for most construction materials have been based on allowable stress design (ASD) concepts (Ellingwood, 1997).

Nowadays, on the one hand, existing wooden structures are strengthened against seismic loadings; on the other hand, it is emphasized that mechanical properties of wood-based materials to be used for new timber structures should have standard values.

In addition, wood is a renewable building material whose structural properties vary by species, natural growth characteristics, and manufacturing practices. At the same time, the quality of the tree and the site index are a part of this definition.

According to the standard methods, the average strength values obtained from the experiments made on the small size and defect-free samples taken from the wood material cannot represent the wooden material with large defects in practice. The solid wood material is not a uniform structure like metals. Various defects such as knots, cracks, spiral fiber, moisture content, temperature grade, loading character, duration, and many other factors negatively affect strength values (Berkel, 1970). The results of mechanical properties are obtained from laboratory tests of straight-grained clear wood samples (without natural defects that would reduce strength, such as knots, checks, splits, etc.) (Winandy, 1994).

Considering that often there is no precise information about these factors, it is inevitable that the loads to be carried in wood material in practice are only a small fraction of the strength obtained in small-sized samples (Bozkurt and Göker, 1996).

Safety stresses are also determined by considering the contained defects that the maximum load can carry when the wood material is used on large products in practice. The safety stress is the ultimate tension limit that a structural element can reliably withstand, depending on the shape, dimensions, and mechanical properties of the material of which a structural part is made. Thus, there is a need to know the safety stresses and use a high safety factor to safely determine the load that the wood material will carry in practice (As, 1992).

The aforementioned ‘safety stress’ placed in TS 647 can be regarded as a concept equivalent to the ‘working stress’ and ‘allowable stress’ expressed in ASTM D2555 (2006) and ASTM D245 (2011). This issue is stated in ASTM D2555-16 (2017) as follows: “This practice covers the determination of strength values for clear wood of different species in the unseasoned condition, unadjusted for end use, applicable to the establishment of working stresses for different solid wood products such as lumber, laminated wood, plywood, and round wood”. Similarly, the allowable stresses are derived from using ASTM D245 to establish acceptable properties for a particular combination of natural growth characteristics (strength-reducing features) in a given timber. ASTM D245 also defines the procedure for determining allowable design stresses for visually graded wood starting from the average breaking strength values given in ASTM D2555 for small clear wood specimens (Anthony and Nehil, 2018).

Eraslan (1982) defined site index as “a term that reveals the productivity of the growth area in which stands grow and develop, yield and power of production”. Yeşil (1993) published a study named “site index research in the natural red pine forest” in Turkey. As a result of this study, the equations essential for the classification of site index have been obtained in pure red pine stands. The development of the measure based on the site index classification of the height and diameter of the same aged stands has been achieved.

Furthermore, the damage could start from the destruction of the wood component on the wooden structures. That is why the basic properties of wood (mechanical and physical) should be known well to be considered in structural design (Yoresta, 2015).

After these explanations in the literature, it can be said that safety stress is a significant factor that should be known in the construction sector, especially in wooden structures. The reduction in the average strength of the wood perfect for achieving safety stress is indispensable for conditions of structural use. In order to better understand the behavior of the wood, such as its mechanical properties and safety stresses, it is necessary to perform experimental tests. This study focused on change of safety stresses and safety coefficients according to the site index classes of red pine, one of the most important tree species of Turkey and the Mediterranean basin. As one of the most important properties of lignocellulosic materials and its effect on strength, performance, and the general quality of final products (Sedlar *et al*., 2021; Sedlar *et al*., 2020; Anjos *et al*., 2014; Priadi and Hızıroğlu, 2013), relationships between the wood density and safety stresses were investigated.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

The material used in the experiment was collected from Turkey pine in accordance with the site index classes (I=SI1, site index II=SI2, and site index III=SI3) and the principles specified in TS 2470:1976 (≡ISO 3129:1975) and TS 4176: 1984 (≡ISO 4471:1982). In order to reveal site index variability, test trees were selected from trial areas with similar characteristics such as soil properties and ecological factors. For the same reason, test trees had ±5 years age, ±100 m height, and ±10 % slopes.

The test specimens were prepared according to the standards related to air-conditioning to reach a humidity level of 12 %.

The tests of the oven dry density (D_0), compression strength parallel to fibers (*CoS*), static bending strength (*BS*), tensile strength parallel to fibers (*TS//*), tensile strength perpendicular to fibers (*TS*⊥), cleavage strength (*CS*) and shear strength parallel to fibers (*SS*) tests were performed on the prepared test specimens in accordance with the relevant standards.

Safety stress was calculated using the following Eq. 1 (Bektaş *et al*., 2018):

$$Safety\,stress = strength\,value\,at\,12\,\% - [25\,\%\ reduction\,due\,to\,defects + 25\,\%\ reduction\ due\,to\,changes + \frac{7}{16}\ reduction\,due\,to\,continuous\ loads + \frac{2}{5}\ reduction\ for\,true\,safety] \quad (1)$$

Where:

- Reduction due to defects: Defects such as cracks, knots, fiber orientation, cork damage significantly reduce the resistance values depending on the usage area of the wood material. Therefore, these must be taken into account when calculating the safety stresses.
- Reduction due to changes: Wood is an anisotropic and hygroscopic material. This leads to significant changes in its properties in areas of use.
- Reduction due to continuous loads: Fatigue loads significantly reduce the resistance of the wood material depending on the usage period.
- Reduction for true safety: The true tolerance of safety is a vital ratio added to preserve life after all other factors are taken into account.

In the same way, the coefficient of safety (safety factor or reduction factor) was calculated using both strength value at 12 % humidity and safety stress value with the help of the following Eq. 2:

Coefficient of safety = (strength value at 12 %) / (safety stress) (2)

Finally, the obtained results were statistically analyzed using one-way ANOVA and Duncan's mean separation test to populate homogeneity groups that showed significant differences at the 95 % confidence level. Again, analyses of the regression and correlation were made to evaluate the relationships between density and safety stresses.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Safety stresses
3.1. Dopuštena naprezanja

The analysis results of the compression strength parallel to fibers test specimens prepared from the site index classes of red pine timber are shown in Table 1.

Table 1 shows that the site index class makes significant differences in the level of $p < 0.001$ on the

Table 1 Safety stresses of compression strength as a function of site index
Tablica 1. Dopuštena naprezanja na tlak drva crvenog bora kao funkcija indeksa staništa

Site index *Indeks staništa*	***N****	**Mean**** *Srednja vrijednost***, N/mm^2	**Standard deviation** *Standardna devijacija*	**Standard error** *Standardna pogreška*	**Coefficient of variation, %** *Koeficijent varijacije, %*	**Coefficient of safety** *Koeficijent sigurnosti*	**Probability** Vjerojatnost
SI1	52	9.92A	3.392	0.470	13.87	5.27	$p < 0.001$
SI2	48	9.03AB	1.310	0.189	14.43	5.27	
SI3	24	8.02B	0.806	0.164	20.41	5.27	
Total	124	9.21	2.459	0.221	8.98	5.27	-

*Number of samples / *broj uzoraka.*
**Means with the same capital letter are not significantly different in Duncan's mean separation test. / *Srednje vrijednosti s jednakim velikim slovom nisu značajno različite prema Duncanovu testu razdvajanja srednjih vrijednosti.*

Table 2 Analyses results of safety stresses for static bending strength experiments
Tablica 2. Rezultati analize dopuštenih naprezanja na savijanje

Site index *Indeks staništa*	***N****	**Mean**** *Srednja vrijednost***, N/mm²	**Standard deviation** *Standardna devijacija*	**Standard error** *Standardna pogreška*	**Coefficient of variation, %** *Koeficijent varijacije, %*	**Coefficient of safety** *Koeficijent sigurnosti*	**Probability** *Vjerojatnost*
SI1	70	11.1A	2.10	0.25	11.95	5.27	$p < 0.001$
SI2	55	8.8B	1.29	0.17	13.48	5.27	
SI3	32	8.0B	2.09	0.37	17.68	5.27	
Total	157	9.7	2.27	0.18	7.98	5.27	-

*Number of samples / *broj uzoraka.*
**Means with the same capital letter are not significantly different in Duncan's mean separation test. / *Srednje vrijednosti s jednakim velikim slovom nisu značajno različite prema Duncanovu testu razdvajanja srednjih vrijednosti.*

compression strength parallel to fibers in the red pine wood. Again, the same table shows that the compression strength values of the samples also decrease statistically as the site index deteriorates (from SI1 to SI3). Since safety stresses are an important factor in using wood as building material, this difference in compression strength due to the site index class should be considered. Considering the effect of the fiber direction on the compression strength, Harte (2009) stated that the compressive strength parallel to the fibers is approximately 5 to 10 times the compression strength perpendicular to the fibers. Here, in compressive failure under pressure, the cells flatten, and the cell walls contact one another, increasing the density and compression strength of the material.

Table 2 shows the relationship between the bending stresses calculated for the static bending strength and the site index. Significant differences ($p < 0.001$) were also determined in safety stresses of bending strength between the first and the other two site index classes. No meaningful separation was observed between SI2 and SI3 classes. This negative relationship between SI2 and SI3 indices may be due to the fact that the prepared bending strength samples have properties close to each other, the location of the samples from the tree is similar to each other and other factors eliminated affecting mechanical performance.

These observations remind us that, in the use of red pine wood timber as a carrier member in timber construction systems, the grade of the site index must be taken into account during the calculation of the safety stresses in the bending strength. It is generally accepted that wood-based main handling elements used in wooden structures have significant roles in building safety. Arroyo (1987) emphasized that the lumber to be used in construction as roof trusses, formworks, beams, scaffolds, and more, must have high rupture strength properties such as static bending strength, modulus of elasticity as well as maximum compression stress.

Table 3 shows that the safety strength values calculated based on the values of tensile strength parallel to the fibers have significant differences in the $p < 0.001$ confidence level among the site indexes. However, this deviation is mainly due to SI1. Table 3 also shows that there is no statistical difference between SI2 and SI3.

As is known, the tensile strength is much higher when loaded in parallel to the fiber direction, while it is deficient when loaded perpendicular to the fiber direction. This low strength perpendicular to the fiber direction needs to be addressed when designing timber structures (Kathem *et al*., 2014).

Another important property of wood is its tensile strength, which is its ability to bend under pressure without breaking. This is one of the main reasons why wood is preferred as a building material; its remarkably strong qualities make it the perfect choice for heavy-duty building materials such as structural beams (AHEC 2017). Also, Raposo *et al*. (2017) reported that the tensile strength parallel to the fibers is superior to

Table 3 Safety stresses of tensile strength parallel to fibers according to site index
Tablica 3. Vrijednosti dopuštenog naprezanja na vlak paralelno s vlakancima ovisno o indeksu staništa

Site index *Indeks staništa*	***N****	**Mean**** *Srednja vrijednost***, N/mm²	**Standard deviation** *Standardna devijacija*	**Standard error** *Standardna pogreška*	**Coefficient of variation, %** *Koeficijent varijacije, %*	**Coefficient of safety** *Koeficijent sigurnosti*	**Probability** *Vjerojatnost*
SI1	35	9.5A	0.818	0.138	11.61	5.27	$p < 0.001$
SI2	37	8.8B	1.329	0.218	11.92	5.27	
SI3	33	8.4B	0.933	0.162	10.88	5.27	
Total	105	8.9	1.128	0.110	11.92	5.27	-

*Number of samples / *broj uzoraka.*
**Means with the same capital letter are not significantly different in Duncan's mean separation test. / *Srednje vrijednosti s jednakim velikim slovom nisu značajno različite prema Duncanovu testu razdvajanja srednjih vrijednosti.*

Table 4 Safety stresses of tensile strength perpendicular to fibers according to site index
Tablica 4. Vrijednosti dopuštenih naprezanja na vlak okomito na vlakanca ovisno o indeksu staništa

Site index *Indeks staništa*	N*	**Mean**** *Srednja vrijednost***, N/mm^2	**Standard deviation** *Standardna devijacija*	**Standard error** *Standardna pogreška*	**Coefficient of variation, %** *Koeficijent varijacije, %*	**Coefficient of safety** *Koeficijent sigurnosti*	**Probability** *Vjerojatnost*
SI1	72	0.37A	0.032	0.004	11.79	5.27	$p < 0.001$
SI2	53	0.35B	0.049	0.007	13.74	5.27	
SI3	38	0.34B	0.037	0.006	16.22	5.27	
Total	163	0.35	0.041	0.003	7.83	5.27	-

*Number of samples / *broj uzoraka.*
**Means with the same capital letter are not significantly different in Duncan's mean separation test. / *Srednje vrijednosti s jednakim velikim slovom nisu značajno različite prema Duncanovu testu razdvajanja srednjih vrijednosti.*

compressive strength parallel to the fibers for specimens made without defects, thanks to the buckling of the fibers under compression.

The analysis of the safety stresses calculated according to the site index in the tensile strength perpendicular to fibers of the samples shows that the site index has a significant effect ($p < 0.001$) on the safety stresses. Moreover, as a result of the Duncan test, it was determined that this effect originated from the difference between SI1 and the others.

The analysis results of the safety stresses in Table 4 calculated according to the site index in the tensile strength perpendicular to fibers of the samples show that the site index has a significant effect ($p < 0.001$) on the safety stresses. Moreover, as a result of the Duncan test, it was determined that this effect originated from the difference between SI1 and the others. While wood has high strength and stiffness in the direction parallel to the fibers, it is an anisotropic material with generally low properties in the direction perpendicular to the fibers. Thus, Harte (2009) emphasized that this characteristic must be taken into account in the design of timber structures where it is important to determine tensile strength perpendicular to the fibers such as in joints, conical or curved members and notched beams, etc. When the data in Table 5 are examined, it can be seen that the analysis results of the ANOVA and Duncan tests of the safety stress calculated for the cleavage strength parallel to fibers were similar to the tensile strength perpendicular to fibers.

The cleavage strength and tensile strength perpendicular to fibers tests are very similar to each other in terms of the test procedure and the shape of the specimens. That is why the two test results are similar to each other. However, cleavage strength is less important in terms of the effect that occurs during use in wood structures than tensile strength perpendicular to fibers. Almeida *et al*. (2015) mentioned that cleavage strength in wood materials is important because it relates to the design of bolted and nailed joints in timber structures. Ferro *et al*. (2013), Segundinho (2010) and Junior *et al*. (2014) stated that wood material can also be applied for structural purposes like bridges, roofs, footbridges, frameworks, and packages if their physical strength and stiffness properties, including cleavage strength, are known. Table 6 depicts that the safety stress values calculated based on shear strength showed a significant difference ($p < 0.043$ level) between SI1 and SI3. In contrast, the SI2 did not show any statistical deviation from other quality classes.

From these data, it can be deduced that the deterioration in the site index classes (from SI1 to SI3) affects the shear resistance safety stress negatively. The shear strength of the wooden material is a factor that plays an important role during the use of wood material in structures. So, the shear strength should always be taken into account when wood is used as a carrier, support and bonding element in wooden structure walls, diaphragms, roofs and floors. Wood (1958) emphasized that most of the reduction in the average

Table 5 Safety stresses of cleavage strength for site indexes
Tablica 5. Dopuštena naprezanja na cijepanje za različite indekse staništa

Site index *Indeks staništa*	N*	**Mean**** *Srednja vrijednost***, N/mm^2	**Standard deviation** *Standardna devijacija*	**Standard error** *Standardna pogreška*	**Coefficient of variation, %** *Koeficijent varijacije, %*	**Coefficient of safety** *Koeficijent sigurnosti*	**Probability** *Vjerojatnost*
SI1	72	0.11A	0.011	0.001	11.79	5.27	$p < 0.001$
SI2	53	0.09B	0.014	0.002	13.74	5.27	
SI3	38	0.09B	0.010	0.002	16.22	5.27	
Total	163	0.10	0.014	0.001	7.83	5.27	-

*Number of samples / *broj uzoraka.*
**Means with the same capital letter are not significantly different in Duncan's mean separation test. / *Srednje vrijednosti s jednakim velikim slovom nisu značajno različite prema Duncanovu testu razdvajanja srednjih vrijednosti.*

Table 6 Calculated safety stress values according to site indexes for shearing strength
Tablica 6. Izračunane vrijednosti dopuštenog naprezanja na smicanje s obzirom na indekse staništa

Site index *Indeks staništa*	*N**	**Mean**** *Srednja vrijednost***, N/mm²	**Standard deviation** *Standardna devijacija*	**Standard error** *Standardna pogreška*	**Coefficient of variation, %** *Koeficijent varijacije, %*	**Coefficient of safety** *Koeficijent sigurnosti*	**Probability** *Vjerojatnost*
SI1	41	1.46A	0.360	0.056	15.62	5.27	$p < 0.043$
SI2	49	1.35AB	0.295	0.042	14.29	5.27	
SI3	24	1.25B	0.174	0.041	23.57	5.27	
Total	114	1.35	0.312	0.030	9.62	5.27	-

*Number of samples / *broj uzoraka.*
**Means with the same capital letter are not significantly different in Duncan's mean separation test. / *Srednje vrijednosti s jednakim velikim slovom nisu značajno različite prema Duncanovu testu razdvajanja srednjih vrijednosti.*

strength of a flawless wood material made to achieve design stress is necessary for structural use conditions because it does not produce a margin for safety. Also, he reported that a simple way to estimate safety is to use near-minimum values for these conversion factors and make a further reduction for unforeseen conditions. Similarly, in a study by Kim *et al.* (2011), the clone and site differences significantly affected fiber length, microfibril angle, and density of Acacia wood. As is known, in wooden structures, the relations between the shear, tensile and compression strengths and the connections (especially the bolt connections) are very tight (Echenique Racero *et al*., 2015).

3.2 Safety coefficient
3.2. Koeficijent sigurnosti

As shown in Table 1-6, the safety coefficient was calculated as an average 5.3 at the 12 % moisture content regardless of site index difference. This value (5.3) is among the recommended limits in the literature. Bozkurt and Göker (1996) accept the safety coefficient for wood material range from 3 to 6. The safety coefficient is the same for all strength values in the same tree type, and in practice, safety stresses are commonly calculated over the safety coefficient. On the other hand, Usta (2007) notes that safety factors for timber construction materials are calculated due to the variability of wood material strength, while safety factors much larger than those of other construction materials should be selected. Indeed, a "main stress" value is determined by considering the differences in each tree type, loading time, safety factor, and other factors suitable for the use of structural lumber and its nature (Wood, 1960).

3.3 Comparison of site indexes safety stresses with standard values
3.3. Usporedba dopuštenih naprezanja drva za različite indekse staništa sa standardnim vrijednostima

The results obtained are compared with the values presented in the Turkish National Codes of Eurocode 5 for the quality classes assigned by Turkish Standard TS 647. Table 7 shows the comparison of the requested safety stress values for coniferous timber quality classes according to TS 647 with calculated safety stress values for site indexes in red pine. The standard TS 647 proposes reference values for the me-

Table 7 Comparison of site indexes safety stresses with standard (TS 647)
Tablica 7. Usporedba dopuštenih naprezanja drva za različite indekse staništa sa standardom TS 647

Safety stress *Dopušteno naprezanje*	**Standard values (TS 647)** *Standardne vrijednosti (TS 647)*			**Values of red pine** *Vrijednosti za drvo crvenog bora*		
	Timber classes, N/mm²* *Klase kvalitete drva,* N/mm²*			**Site indexes, N/mm²** *Indeks staništa,* N/mm²		
	T1	T2	T3	SI1	SI2	SI3
Static bending strength *statička čvrstoća na savijanje*	13	10	7	11.1	8.8	8.0
Compression strength (parallel) *čvrstoća na tlak (paralelno)*	11	8.5	6	9.9	9.0	8.0
Tensile strength (parallel) *čvrstoća na vlak (paralelno)*	10.5	8.5	0	9.5	8.8	8.4
Shearing strength (parallel) *čvrstoća na smicanje (paralelno)*	0.9	0.9	0.9	1.7	1.4	1.3
Modulus of elasticity *modul elastičnosti*	10 000			9 651**		

*Timber classes I, II, and III expressed in TS 647 are marked as T1, T2, and T3, respectively. / *Klase kvalitete drva I., II. i III. izražene u TS 647 označene su kao T1, T2 i T3.*
**Özkaya, 2013.

chanical properties of coniferous trees. If we compare them with those obtained experimentally for red pine grown in Turkey, it will be seen that most of these were higher than those proposed by the standard. At the same time, Turkey Wood Building Regulations divide the wood into three strength level classes for coniferous woods (class I, II, and III) (Table 7). In this classification, as the strength and quality of the wood increases, the class level also increases from 3 towards 1.

Table 7 shows that the safety stress values of the red pine wood calculated for the site index SI1 cannot provide the required standard values for grade T1 timber, except for the shear strength. Again, the same table shows that the safety stresses determined for the other site indexes (SI2 and SI3) meet the standard (for T1 and T2). It has also been found that the elasticity modulus value (9 651 N/mm^2) is very close to the requested standard value (10 000 N/mm^2) for all timber classes. For all classes, the results obtained from the statistical analysis of the *CoSS* and *TS* values were in total 4.7 % and 25.7 % higher than those proposed by TS 647, respectively. In contrast, the calculated BSS values are 7 % lower than the recommended standard values.

However, it should not be ruled out that the site index and the timber class values have not the same meaning. Despite all these, it is possible to say that the safety stresses calculated in the red pine wood provide the required safety stress values for wood materials to be used in wooden structures according to TS 647, excluding T1. Therefore, we could propose that the reference values established by the TS 647 standard be considered valid, especially for classes outside class T1. After all these evaluations, it can be said that the structural elements to be made with red pine wood are suitable for structural use according to the Turkey Wood Building Regulations. The National Design Specification (NDS) has, on the other hand, recognized the importance of these system effects by permitting an increase of 15 % in the allowable bending stress used to design assemblies where three or more members are used repetitively (Bezaleel, 2004). In the end, wood construction manufacturers will consider these standard and wood safety stress values for project calculations in structures.

3.4 Variation of safety stress and density with site index
3.4. Varijacija dopuštenog naprezanja i gustoće drva s promjenom indeksa staništa

Density values were also calculated on the sample where each mechanical resistance value was measured. Then, with the help of these calculated density values, graphs (Figures 1-3) showing relationships between the safety stress and density were obtained. In these charts, for a healthy display, the *TSS* (safety stress at tensile stress) and *CSS* (safety stress at cleavage stress) values were multiplied by 10, while the *SSS* (safety stress at shear stress) values (for SI3) were multiplied by 2.

As shown in Figure 1, there is a generally positive increasing relationship between safety stress and density for the first site index. In Table 8, the correlation coefficients (R^2) safety stress values were calculated between 0.49 and 0.63 for the first site index. The strongest correlation (R^2=0.63) was determined in the *SSS*, while the weakest (R^2=0.49) was found in the CSS.

Figure 2 shows the interaction between density and safety stress values measured for SI2. When the graph in Figure 2 is evaluated together with the data in Table 8, it can be said that the correlation coefficients calculated according to the relationship between the safety stress of the mechanical properties and densities are R^2>48. This means that the density can explain more than 50 % of the comparative safety stress values.

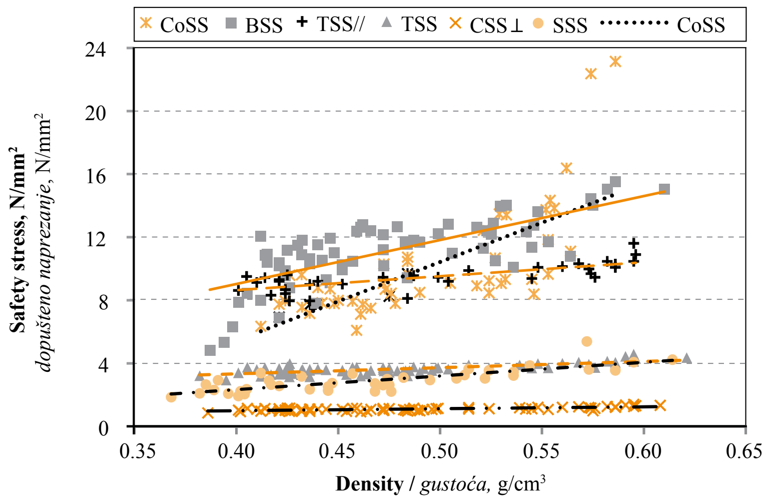

Figure 1 Slope lines of relations between safety stress and density for SI1
Slika 1. Nagibi odnosa između dopuštenog naprezanja i gustoće drva za SI1

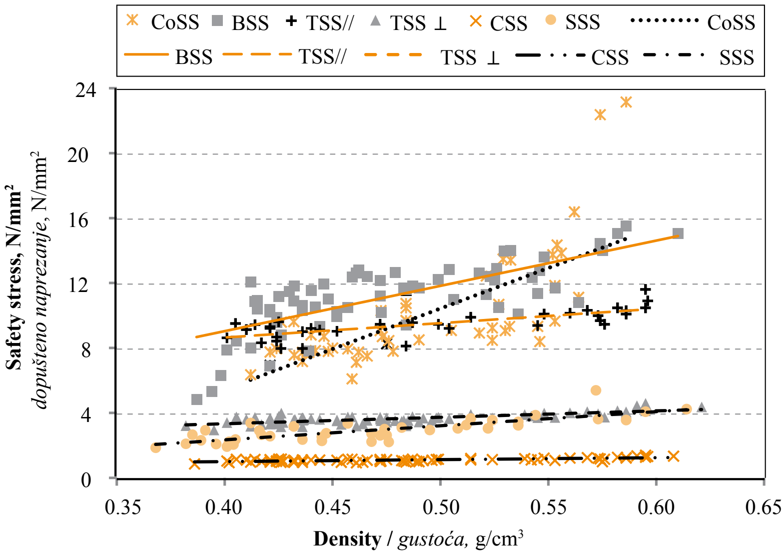

Figure 2 Relationships between safety stress and density for SI2
Slika 2. Odnos između dopuštenog naprezanja i gustoće drva za SI2

In general, it can be said that there is a moderate correlation between density and safety stresses for SI2.

For the SI3, it is seen that R^2 values calculated in Table 8 through the slope lines formed in the graph (Figure 3) are ranging between 0.41-0.68. Again, it is understood from Figure 3 and Table 8 that the relationship of the weakest correlation is between density and TSS// (R^2 = 0.41), and the most substantial relationship is between density and CoSS (safety stress at compression stress) (R^2 = 0.68).

When the correlation coefficients calculated in Table 8 were evaluated according to site index classes, the highest R^2 values were calculated in TSS and SSS for SI1. Also, CSS was obtained for SI2 and determined in CoSS and BSS for SI3. The same table shows close correlation coefficient averages (R^2= 0.55, 0.54, and 0.55) calculated for the site indexes. Machado and Cruz (2005) reported that the relationship between wood density and strength properties is acknowledged in part because density is a measure of the relative amount of solid cell wall. In several researches (Bektaş *et al.*, 2020; Sedlar *et al.*, 2019; Güler, 2004; Yang and Evans, 2003; Evans and Ilic, 2001; Rozenberg *et al.*, 1999; Cave and Walker, 1994), various factors, such as cell wall thickness, wood component ratio, microfibril angle, and fiber angle, are reported to affect the relationship between wood density and mechanical properties. In general, the presence of a linear relationship between density and strength values is agreed to a large extent. It can be said that this determination is generally consistent with the results to be deduced from the slope lines drawn in Figures 1-3 for density and safety stresses. It is also a fact that the wood quality assessment involves considering wood density and mechanical properties (Anoop, 2014).

Again, when the regression equations given in Table 8 are examined in terms of site index classes, it will be easily seen that the sign of b values is "positive"

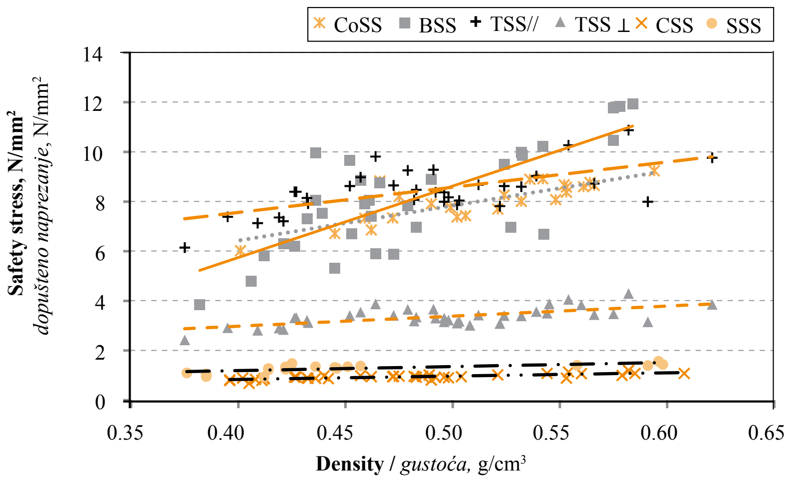

Figure 3 Relationships between safety stress and density for SI3
Slika 3. Odnos između dopuštenog naprezanja i gustoće drva za SI3

Table 8 Equations derived from regression analyses for safety stresses
Tablica 8. Jednadžbe izvedene iz regresijske analize za vrijednosti dopuštenih naprezanja

Parameter *Parametar*	**SI1**		**SI2**		**SI3**	
	Equation / *Jednadžba*	R^2	**Equation** / *Jednadžba*	R^2	**Equation** / *Jednadžba*	R^2
CoSS	$y = -14.5+49.9 \cdot x$	0.51	$y = 0.51+8 \cdot x$	0.53	$y = 0.8+14.1 \cdot x$	0.68
BSS	$y = -2.1+27.9 \cdot x$	0.54	$y = 0.7+16.4 \cdot x$	0.57	$y = -5.8+28.9 \cdot x$	0.58
TSS//	$y = 5.1+8.8 \cdot x$	0.59	$y = 1.7+14.2 \cdot x$	0.55	$y = 3.5+10.2 \cdot x$	0.41
TSS⊥	$y = 1.8 +3.9 \cdot x$	0.59	$y = 0.7+5.6 \cdot x$	0.50	$y = 1.4+4.1 \cdot x$	0.42
CSS	$y = 0.5+ 1.2 \cdot x$	0.49	$y = 0.1+1.9 \cdot x$	0.62	$y = 0.3+1.3 \cdot x$	0.61
SSS	$y = -1.1+8.7 \cdot 6x$	0.63	$y = -0.2+3.3 \cdot x$	0.48	$y = 0.5+1.6 \cdot x$	0.48
Average	-	0.55	-	0.54	-	0.55

for all site index classes. As is known, in the equations ($a + b \cdot x$) obtained by the regression analysis, if the sign of b is positive, then the variables of the compared societies either increase or decrease together (Başar and Oktay, 2007).

4 CONCLUSIONS
4. ZAKLJUČAK

In the scope of the study, safety stress and safety coefficients for the compression strength, bending strength, tensile strength parallel to fibers, tensile strength perpendicular to fibers, cleavage strength, and shearing strength were calculated for the red pine wood. As a result of the analyses, the values of CoSS, BSS (safety stress at bending stress), TSS//, TSS⊥, CSS and SSS were determined as average 9.2 N/mm^2, 9.7 N/mm^2, 8.9 N/mm^2, 0.35 N/mm^2, 0.10 N/mm^2 and 1.4 N/mm^2, respectively.

Again, the results of statistical analyses (ANOVA and Duncan's mean separation test) showed that site index had a significant effect (p <0.05-0.001 levels) on the safety stresses.

When the data obtained in the study are evaluated in total, it can be said that the safety stress values of red pine wood provided other lower limit standard values (For SI2 and SI3) according to TS 647 excluding T1.

However, when the indexes and classes were compared one to one, it was determined that site index 1 satisfied only SSS of class 1, site index 2 satisfied CoSS, TSS //, TS⊥ , and SSS of class 2, and site index 3 satisfied all values of class 3. On the other hand, the safety coefficient was calculated as 5.27 for red pine wood.

The results of regression and correlation analyses revealed a relationship between density and safety stresses that varies according to site index and strength values. Calculated R^2 values were between 0.49-0.63 for SI1, 0.48-0.62 for SI2, and 0.41-0.68 for SI3. It can be assumed that there is a medium degree-strong relationship for all site indexes between density and safety stress in red pine wood.

In the guidance of these results and evaluations, it can be said that red pine wood can be used safely in construction and especially in wood structures where mechanical strength is important. Thus, this wood can be recommended for use in wood building structures (columns, beams, and floor).

In the future, as emphasized in a study (Heräjärvi, 2004), the building industries will need predictable, homogeneous, and cost-competitive wood products with structural safety in increasing quantity and quality.

The final word of this research is the necessity of dealing with new species-origin combinations in studies to determine the properties of the wood material used, which is one of the essential issues of timber structures. And also, it is necessary to know the standard values in practice for each wood element of wood construction, the safety stresses as calculated by standard methods of engineering mechanics.

After all, it is recommended that further studies be carried out under the guidance of the developing technology for the use of wood materials for structural purposes, as this issue is critically important and has not been thoroughly investigated so for.

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Prediction of Optimum Expanded Polystyrene Densities for Best Thermal Insulation Performances of Polystyrene Composite Particleboards by Using Artificial Neural Network

Predviđanje optimalne gustoće ekspandiranog polistirena za najbolje performanse toplinske izolacije polistirenske kompozitne iverice primjenom umjetne neuronske mreže

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ABSTRACT • *The objective of this study is to predict the optimum expanded polystyrene (EPS) densities for the best insulation properties of the particleboards manufactured with waste EPS instead of formaldehyde-based adhesives used in particleboard production with artificial neural network (ANN). For this purpose, the waste EPS particles of five different densities were used in the production of composite particleboards. The experimental data used in the study were obtained from the previous study. Half of the beech, poplar, alder, pine and spruce chips were dried in a drying oven and the other half were naturally conditioned at room temperature, and then 18 mm thick three-layer composite particleboards were produced. The thermal conductivity of panels was determined according to ASTM C 518. The prediction model with the best performance and acceptable deviations was determined by using statistical and graphical comparisons between the experimental data and the prediction values obtained as a result of ANN analysis. Then, using this prediction model, the thermal conductivity coefficient values were estimated for the intermediate EPS densities that were not experimentally tested. According to the analysis findings, the thermal insulation performance for both beech and spruce polystyrene composite particleboards (PCP) panels increased with using of waste EPS foams with a density of 30 kg/m^3. The lowest thermal conductivity values were obtained from the EPS waste foams with the density of 18, 13 and 22 kg/m^3 for the PCP panels produced with poplar, alder and pine in the natural drying, respectively. In the technical drying, these values were found to be 15, 14 and 11-13 kg/m^3, respectively. Technical drying showed much better thermal performance than natural drying while poplar indicated the best performance among the wood species.*

KEYWORDS: *polystyrene composite particleboard; thermal insulation; thermal conductivity; EPS; ANN*

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SAŽETAK • *Cilj je ove studije primjenom umjetne neuronske mreže (ANN) predvidjeti optimalne gustoće ekspandiranog polistirena (EPS) radi postizanja najboljih izolacijskih svojstava iverice proizvedene s otpadnim EPS-om umjesto s ljepilom na bazi formaldehida, kakvo se rabi u proizvodnji iverice. Stoga je za proizvodnju iverice upotrijebljen otpadni EPS pet različitih gustoća. Eksperimentalni podatci primijenjeni u studiji dobiveni su prijašnjim istraživanjem. Jedna je polovica iverja bukovine, topolovine, johovine, borovine i smrekovine osušena u sušioniku, a druga je polovica iverja kondicionirana na sobnoj temperaturi. Od osušenoga i kondicioniranog iverja proizvedene su troslojne kompozitne iverice debljine 18 mm. Toplinska vodljivost ploča određena je metodom ASTM C 518. Predikcijski model najboljih svojstava i prihvatljivih devijacija određen je statističkom i grafičkom usporedbom eksperimentalnih podataka s vrijednostima predviđenima ANN analizom. Potom su primjenom predikcijskog modela procijenjeni koeficijenti toplinske vodljivosti za one gustoće ekspandiranog polistirena koje nisu eksperimentalno ispitane. Prema toj analizi, termoizolacijska svojstva polistirenske kompozitne iverice (PCP) od bukovine i smrekovine poboljšana su pjenom od otpadnog EPS-a gustoće 30 kg/m³. Najniže vrijednosti toplinske vodljivosti za polistirensku kompozitnu ivericu od prirodno osušene topolovine, johovine i borovine dobivene su uz uporabu pjene otpadnog ESP-a gustoće 18, 13 i 22 kg/m³. Za polistirensku kompozitnu ivericu od tehnički osušenog iverja te su vrijednosti bile 15, 14 i 11-13 kg/m³. Tehničkim sušenjem iverja postignuta su znatno bolja toplinska svojstva polistirenske kompozitne iverice nego prirodnim sušenjem, a topolovina je pokazala najbolja svojstva od svih ispitivanih vrsta drva.*

KLJUČNE RIJEČI: *polistirenska kompozitna iverica; termoizolacija; toplinska vodljivost; ESP; ANN*

1 INTRODUCTION
1. UVOD

Thermal insulation is one of the most effective measures to increase energy efficiency by improving the thermal properties of building envelopes (Cetiner and Shea, 2018). At first, it was tried to provide thermal insulation in buildings by using natural materials such as rice straw, rice husk, sawdust, animal fur and wood (Wei *et al.,* 2015). After the industrialization process, chemicals were used in the production of insulation materials to improve their performance (Khoukhi, 2018), and foam insulation materials based on polymer compounds such as polyurethane, polystyrene, and polyethylene have been recently developed (Wi *et al.,* 2021). Expanded polystyrene (EPS) foam, usually called Styrofoam, is widely used in packaging, appliances, building insulations, and decorations because of its outstanding properties, such as superior thermal insulation, excellent dimensional stability, low cost, low density, and low sensitivity to moisture, and it occupies the largest market share in building insulation materials (Li *et al.,* 2020). However, one of the most abundant plastics waste sources comes from various products of EPS foam (Rajak *et al.,* 2020). Global plastic production has exponentially increased in the past decades, and reached 359 million tons in 2018 (Plastics Europe, 2019). Although the amount of plastic waste sent to recycling has doubled in Europe and major industrial countries since 2006, plastic pollution is still a major environmental concern (Geyer *et al.,* 2017; Plastics Europe, 2019; Tokiwa *et al.,* 2009; Xu *et al.,* 2020; Song *et al.,* 2020). The most of EPS wastes come from food packaging and storage, and therefore studies on the recycling of EPS waste have increased recently in more than 30 countries (Koksal *et al.,* 2020). The toxic gases such as carcinogenic polycyclic aromatic hydrocarbons (PAHs) and dioxin, which are released by burning waste EPS foams, cause serious environmental problems (Chaukura *et al.,* 2016; Uttaravalli *et al.,* 2020). In addition, the low density of EPS can rapidly consume the loading capacity of waste storage areas (Chaukura *et al.,* 2016). Due to these problems, recycling methods of EPS wastes need to be both eco-friendly and economical. Demirkir *et al.* (2013a) stated that the production of wood and polystyrene materials as a composite could be an effective solution both in reducing the environmental pollution caused by polystyrene waste and in reducing the emission of formaldehyde released from wood-based panels.

In the previous study, it was determined that the thermal insulation properties of the particleboard panels bonded with different densities of EPS foam wastes were much better than those of the control groups produced with urea formaldehyde (Demirkir *et al.,* 2019). It was observed that the densities of EPS foams used in the production of these panels influenced the thermal conductivity values. Based on this, it is crucial to determine the optimum EPS densities to get the best thermal insulation performance for the building industry. Therefore, it is very important to use the right methods that do not require further experimentation, labour, time, energy and high costs (Demirkir *et al.,* 2013b). Researchers used artificial neural networks (ANN), which are more adaptable than traditional methods, for optimization of wood and wood-based materials because they were faster and more economical (Esteban *et al.,* 2011; Demirkir *et al.,* 2013b; Ozsahin and Aydin, 2014; Tiryaki *et al.,* 2017; Ozsahin and Murat, 2018). Even if the relationships between the experimentally obtained input and output data were complex and meaningless, ANN modelling could be successfully

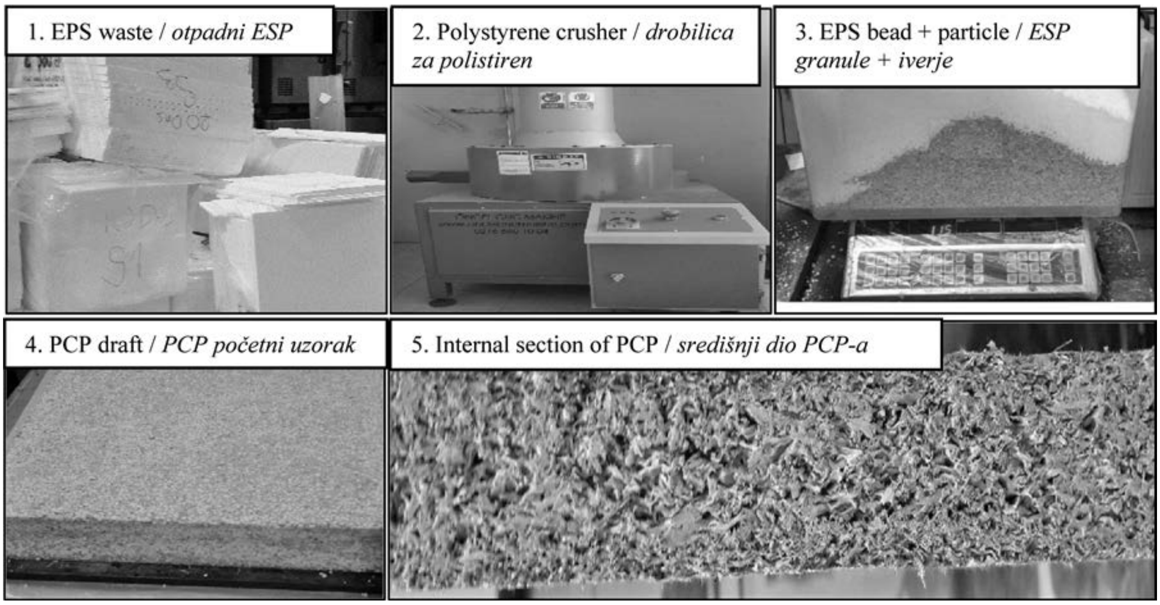

Figure 1 Polystyrene composite particleboards (PCP) production process
Slika 1. Proces proizvodnje polistirenske kompozitne iverice (PCP)

performed to obtain the desired optimum values (Fernandez *et al.,* 2008).

The objective of this study is to predict the optimum EPS density values for the best thermal insulation properties of the particleboards produced with EPS waste instead of formaldehyde-based adhesives via artificial neural network (ANN). For this purpose, the thermal conductivity values of intermediate EPS density values, which were not used in experimental studies, were also predicted by ANN model and the effects of EPS densities for each polystyrene composite particleboard (PCP) group were revealed.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Data collection
2.1. Prikupljanje podataka

The experimental data used in this study were obtained from the previous study by Demirkir *et al.* (2019). Beech (*Fagus orientalis* Lipsky), poplar (*Populus deltoides* I-77/51), alder (*Alnus glutinosa* subsp. *barbata*), pine (*Pinus Sylvestris*) and spruce (*Picea orientalis* L.) woods, widely preferred in the particleboard industry, were used in the production of composites by chipping. Half of these chips were dried in a drying machine at 90 °C until reachng 3 % moisture content (technical drying). The other half were conditioned at room temperature until reaching 12 % moisture content (natural drying). The waste EPS particles of five different densities (10, 16, 20, 24 and 30 kg/m^3) were used as bonding material in the production of composite particleboards. The waste EPS particles, crushed in a polystyrene crusher with a size of 1.5 - 3 mm, were used in the production of polystyrene composite particleboards (PCP). The EPS particles were homogeneously mixed at a rate of 10 % for the surface layer and 8 % for the core layer based on the particle weight (dry weight for technical drying and wet weight for natural drying).

The panel drafts with dimensions of 55 cm × 55 cm × 1.8 cm were pressed for 10 minutes at 150 °C at a pressure of 23-25 kg/cm^2. The ratio of the face thickness to the total thickness of a panel, known as the shelling ratio, was 0.35 for all samples, while the target density was 0.68 g/cm^3. After the panels were conditioned for three weeks, the thermal conductivity test measurements were carried out to determine the thermal properties of the panels. The thermal conductivity coefficients of the panels were determined according to the ASTM C 518 (2004) standard using the Lasercomp Fox-314 thermal conductivity device (Figure 2). Moreover, the thermal conductivity values of waste EPS foams of 10 cm thickness, with a cell content of 98 %

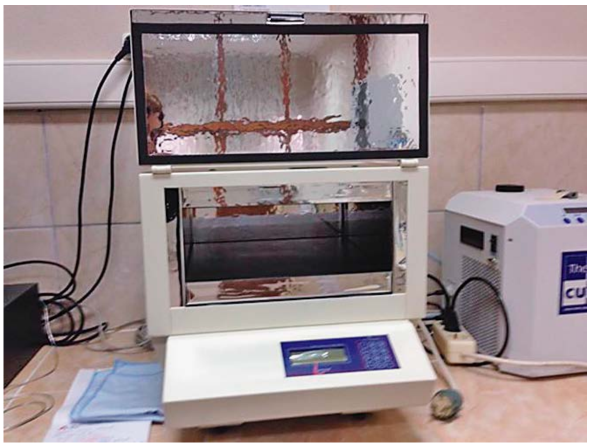

Figure 2 Lasercomp Fox-314 thermal conductivity device
Slika 2. Uređaj za toplinsku vodljivost Lasercomp Fox-314

air and 2 % polystyrene, were calculated as 0.040, 0.039, 0.036, 0.035 and 0.034 W/mK according to their density, respectively.

2.2 Artificial neural network (ANN) analysis
2.2. Analiza umjetnom neuronskom mrežom

ANN analysis, which is one of the algorithms used in nonlinear input and output variables modelling, has started to be preferred in recent years instead of logistic regression analysis, which is one of the most widely used methods in the development of predictive models. ANN, which can successfully model the complex relationships of input and output variables without the need for further statistical training, easily reveals all possible interactions between these variables and enables multiple training algorithms (Tu, 1996; Dumitru and Maria, 2013). ANN was used to predict thermal conductivity values of the EPS densities not used in experimental studies and to predict the optimum EPS densities that gave the best thermal insulating performance for each PCP group. Drying types, wood species and EPS densities were the main variables in ANN modelling of this study. The data obtained from experimental studies were modelled with the MATLAB Neural Network Toolbox. The experimental data were grouped as training data and testing data to determine

Table 1 Training data set used for thermal conductivity prediction model results
Tablica 1. Skup primjera za učenje primijenjen za predikcijski model toplinske vodljivosti

Drying type *Tip sušenja*	**Wood species** *Vrsta drva*	**EPS density, kg/m³** *Gustoća EPS-a,* kg/m³	**Thermal conductivity, W/mK** *Toplinska vodljivost,* W/mK		
			Actual *Stvarna*	**Predicted** *Predviđena*	**Error, %** *Greška,* %
Natural drying *prirodno sušenje*	Beech / *bukovina*	16	0.09891	0.09891	-0.004
	Beech / *bukovina*	24	0.09467	0.09466	0.008
	Beech / *bukovina*	30	0.09245	0.09245	-0.002
	Poplar / *topolovina*	10	0.10040	0.10040	0.005
	Poplar / *topolovina*	16	0.09266	0.09267	-0.011
	Poplar / *topolovina*	20	0.09276	0.09277	-0.010
	Poplar / *topolovina*	30	0.09529	0.09531	-0.025
	Alder / *johovina*	10	0.09674	0.09674	0.005
	Alder / *johovina*	20	0.09668	0.09641	0.282
	Alder / *johovina*	24	0.09664	0.09665	-0.008
	Pine / *borovina*	10	0.09705	0.09705	0.002
	Pine / *borovina*	16	0.09578	0.09661	-0.865
	Pine / *borovina*	24	0.09802	0.09670	1.343
	Pine / *borovina*	30	0.09428	0.09495	-0.714
	Spruce / *smrekovina*	10	0.10110	0.10064	0.452
	Spruce / *smrekovina*	20	0.09794	0.09831	-0.381
	Spruce / *smrekovina*	30	0.09692	0.09705	-0.138
Technical drying *tehničko sušenje*	Beech / *bukovina*	10	0.08995	0.09000	-0.050
	Beech / *bukovina*	20	0.09167	0.09103	0.701
	Beech / *bukovina*	24	0.08866	0.08938	-0.817
	Beech / *bukovina*	30	0.08621	0.08627	-0.074
	Poplar / *topolovina*	16	0.08316	0.08310	0.070
	Poplar / *topolovina*	20	0.08443	0.08604	-1.910
	Poplar / *topolovina*	30	0.08461	0.08414	0.554
	Alder / *johovina*	10	0.08642	0.08357	3.296
	Alder / *johovina*	16	0.08662	0.08665	-0.031
	Alder / *johovina*	20	0.08957	0.08670	3.202
	Alder / *johovina*	24	0.08783	0.08644	1.581
	Pine / *borovina*	10	0.08128	0.08366	-2.928
	Pine / *borovina*	20	0.08602	0.08700	-1.134
	Pine / *borovina*	24	0.08441	0.08691	-2.963
	Spruce / *smrekovina*	16	0.09075	0.09071	0.044
	Spruce / *smrekovina*	20	0.08788	0.08811	-0.263
	Spruce / *smrekovina*	24	0.08783	0.08730	0.604
	Spruce / *smrekovina*	30	0.08673	0.08701	-0.326
Mean absolute percent error (MAPE) training *Srednja apsolutna postotna pogreška (MAPE) na skupu za učenje*			0.70870		
Root Mean Square Error (RMSE) Training *Korijen srednje kvadratne pogreške (RMSE) na skupu za učenje*			0.00110		

Table 2 Testing data set used for thermal conductivity prediction model results
Tablica 2. Testni skup podataka upotrijebljen za predikcijski model toplinske vodljivosti

Drying type Tip sušenja	**Wood species** Vrsta drva	**EPS density,** kg/m³ *Gustoća EPS-a,* kg/m³	**Thermal conductivity, W/mK** *Toplinska vodljivost*, W/mK		
			Actual *Stvarna*	**Predicted** *Predviđena*	**Error, %** *Greška*, %
Natural drying *prirodno sušenje*	Beech / *bukovina*	10	0.10080	0.10071	0.089
	Beech / *bukovina*	20	0.09413	0.09685	-2.884
	Poplar / *topolovina*	24	0.09307	0.09305	0.021
	Alder / *johovina*	16	0.09700	0.09781	-0.836
	Alder / *johovina*	30	0.09385	0.09647	-2.792
	Pine / *borovina*	20	0.09482	0.09491	-0.096
	Spruce / *smrekovina*	16	0.10230	0.09951	2.730
	Spruce / *smrekovina*	24	0.09739	0.10013	-2.816
Technical drying *tehničko sušenje*	Beech / *bukovina*	16	0.08742	0.08882	-1.605
	Poplar / *topolovina*	10	0.08250	0.08335	-1.032
	Poplar / *topolovina*	24	0.08239	0.08546	-3.730
	Alder / *johovina*	30	0.08398	0.08575	-2.106
	Alder / *johovina*	16	0.08679	0.08705	-0.296
	Pine / *borovina*	30	0.08557	0.08668	-1.295
	Spruce / *smrekovina*	10	0.09318	0.09345	-0.289
MAPE testing / *MAPE na testnom skupu*				1.50790	
RMSER testing / *MSE na testnom skupu*				0.00180	

the effects of drying types, wood species and EPS densities on the thermal insulating performance. In order to successfully obtain the ANN prediction model from the experimentally determined thermal conductivity coefficient values, 35 data containing 70 % of the total experimental data were separated for the training set, while the remaining 15 data containing 30 % of the whole data were separated for the test set by considering the homogeneity of the groups. The thermal conductivity coefficient values obtained from both experiments and ANN analyses and the error percentages are shown in Tables 1 and 2 as training and test data sets.

The reliability of a predictive model whose training process has been completed successfully is determined by commonly used performance functions. The root mean square error (RMSE), mean absolute percent error (MAPE) and coefficient of determination (R^2), which are the most important of these functions, were preferred in this study. The equations formed depending on differences between the actual values obtained from the experimental data (t_i) and the predicted values obtained from the ANN model (td_i) in the testing data set are given in Eqs. 1, 2 and 3, respectively (N is the number of objects).

$$RMSE = \sqrt{\frac{1}{N}\sum_{i=1}^{N}\left(t_i - td_i\right)^2} \tag{1}$$

$$MAPE = \frac{1}{N}\left(\sum_{i=1}^{N}\left[\left|\frac{t_i - td_i}{t_i}\right|\right]\right)\cdot 100 \tag{2}$$

$$R^2 = 1 - \frac{\sum_{i=1}^{N}(t_i - td_i)^2}{\sum_{i=1}^{N}(t_i - \bar{t})^2} \tag{3}$$

The network structure of the most reliable prediction model determined using performance functions after numerous trials is shown in Figure 3. This model consists

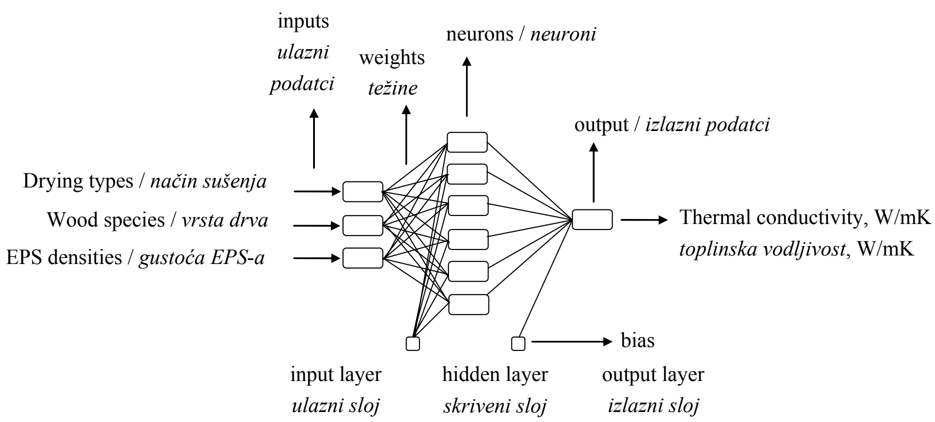

Figure 3 ANN architecture selected as prediction model
Slika 3. Arhiktektura ANN modela odabranoga za predikcijski model

Table 3 Connection weights and biases of thermal conductivity prediction model
Tablica 3. Težine i sistemske pogreške predikcijskog modela toplinske vodljivosti

Hidden layer / *Skriveni sloj*						**Output layer** / *Izlazni sloj*		
Neuron1	Neuron2	Neuron3	Neuron4	Neuron5	Neuron6	Bias1	Neuron1	Bias2
-6.33217	6.21830	12.41869	11.37884	1.17178	-3.39807	6.90932	-0.28800	0.39836
10.34989	10.70420	-14.02956	6.09988	1.16163	-3.28623	0.72941	0.16322	-
-6.15530	25.22297	-1.90658	-0.69203	-0.61331	-1.40521	0.42025	0.57538	-
-	-	-	-	-	-	1.32899	-0.35932	-
-	-	-	-	-	-	0.70403	0.86996	-
-	-	-	-	-	-	-5.91313	0.65224	-

of an input layer containing drying types, wood species and EPS densities, a hidden layer containing 6 neurons, and an output layer containing thermal conductivity coefficients. The connection weights and biases of the thermal conductivity prediction model are given in Table 3.

The feed forward and backpropagation multilayer ANN were used to determine the prediction model. In ANN analysis trials, the transfer (activation) function used the hyperbolic tangent sigmoid function (tansig) in the hidden layer, while the linear transfer function (purelin) was used in the output layer. The Levenberg marquardt algorithm (trainlm) and the momentum gradient reduction backpropagation algorithm (traingdm) were chosen as the training algorithm and the learning rule. The mean square error (MSE) was used for stopping the training phase. The MSE equation, which can be calculated based on the difference between the actual values obtained from the experimental data (t_i) and the predicted values obtained from the ANN model (td_i) in the training data sets, is given in Eq. 4 (N is the total number of training patterns).

$$MSE = \frac{1}{N}\sum_{i=1}^{N}\left(t_{\mathrm{i}} - td_{\mathrm{i}}\right)^2 \quad (4)$$

The data in the training and testing set were normalized (-1, 1 range) since the hyperbolic tangent sigmoid (tansig) function was used to contribute equally to the model for each parameter in the prediction model and then the data were transformed to their original values by reverse normalization so that the results could be evaluated. The processing of normalization was performed by using Eq. 5.

$$X_{\mathrm{norm}} = 2 \cdot \frac{X - X_{\mathrm{min}}}{X_{\mathrm{max}} - X_{\mathrm{min}}} - 1 \quad (5)$$

Where, X_{norm} is the normalized value of a variable X (real value of the variable), and X_{max} and X_{min} are the maximum and minimum values of X, respectively.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

Figure 4 presents the MSE value that gives the best training performance of 0.0099939 in the 500th iteration when the MSE changes based on iteration of the prediction model determined as a result of ANN analysis.

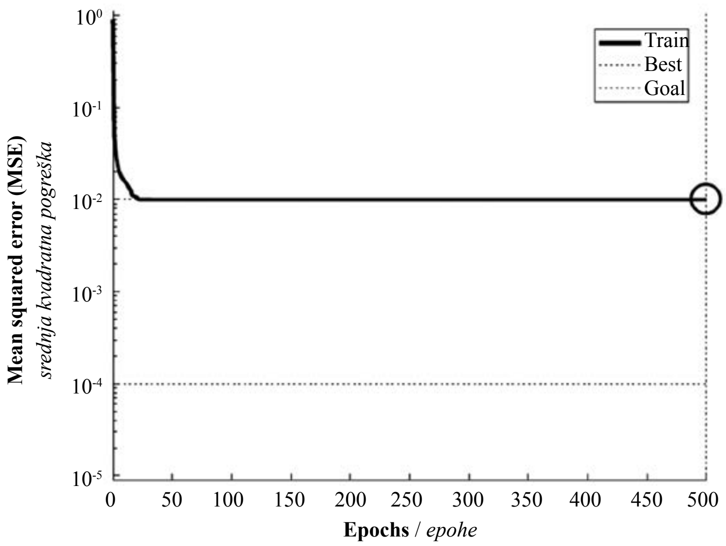

Figure 4 MSE changes at each iteration for training data set of ANN model
Slika 4. Promjene u srednjoj kvadratnoj pogrešci za svaku iteraciju na skupu podataka za učenje modela umjetne neuronske mreže

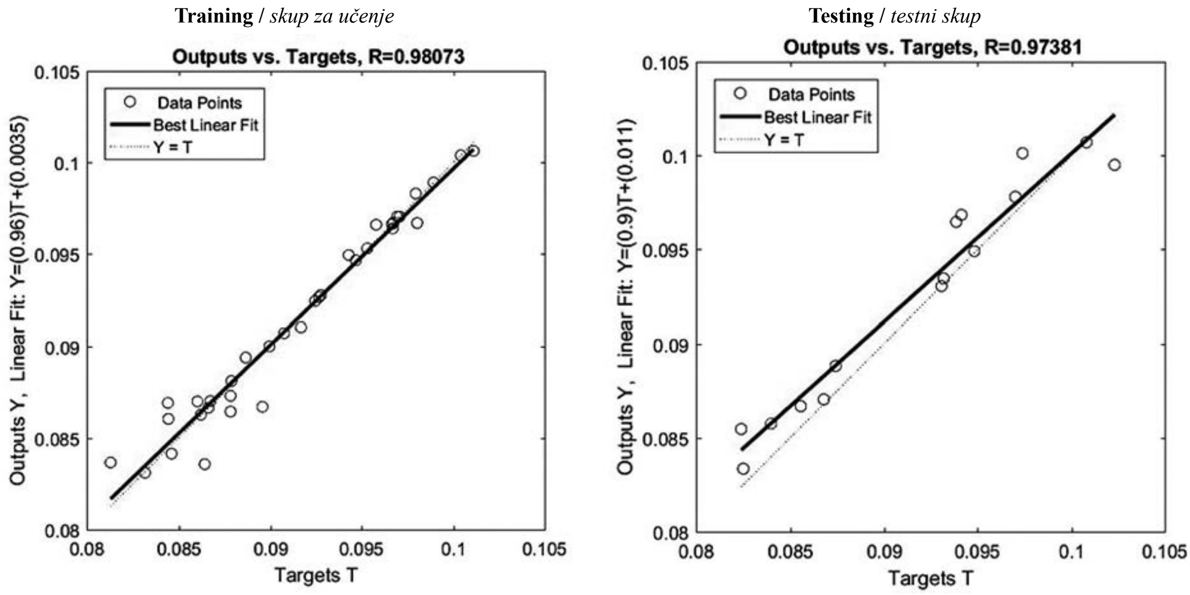

Figure 5 Regression models of thermal conductivity prediction model
Slika 5. Regresijski modeli predikcijskog modela toplinske vodljivosti

The regression analysis results of the thermal conductivity prediction model are given in Figure 5. The correlation coefficient values for training and testing data sets were calculated as 0.98073 and 0.97381, respectively. These values proved the accuracy and validity of the prediction model obtained. Since the reliability of prediction models increases as the correlation coefficients approach 1, a perfect fit between the actual and predicted values can be achieved (Ozsahin, 2012).

The comparison of the thermal conductivity coefficient values obtained from the experiments and the predicted values obtained from the ANN analysis is given graphically in Figure 6. It is seen that the actual values and the predicted values are quite close to each other, and this proves that the predictive ability of the model is quite high.

The MAPE values in the training data sets and testing data sets were calculated as 0.71 % and 1.51 %, respectively (Table 1 and 2). The MAPE value, which is frequently used by researchers to evaluate ANN model performance, is expected to be below 10 % (Antanasijević *et al*., 2013; Tiryaki *et al*., 2016). It is proved that the prediction performance of ANN models is high with these values lower than 10 % (Yadav and Nath, 2017). It is stated in the literature that it is extremely important to calculate RMSE values as well as MAPE values in order to determine the performance of prediction models (Kucukonder *et al*., 2016). In this study, the RMSE values of the thermal conductivity prediction model for training and testing phase were calculated as 0.0011 and 0.0018, respectively. (Table 1 and 2). Taspınar and Bozkurt (2014) stated that the low RMSE values obtained from the ANN analyses are an indicator of the successful performance of the prediction model. The MAPE and RMSE values obtained from the study proved that the ANN model used for prediction and optimization are reliable and can give satisfactory accurate results.

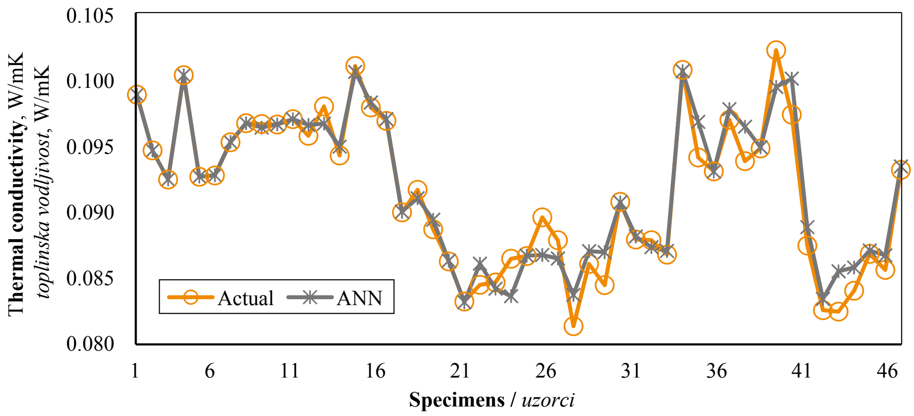

Figure 6 Comparison of measured and predicted values
Slika 6. Usporedba izmjerenih i predviđenih vrijednosti

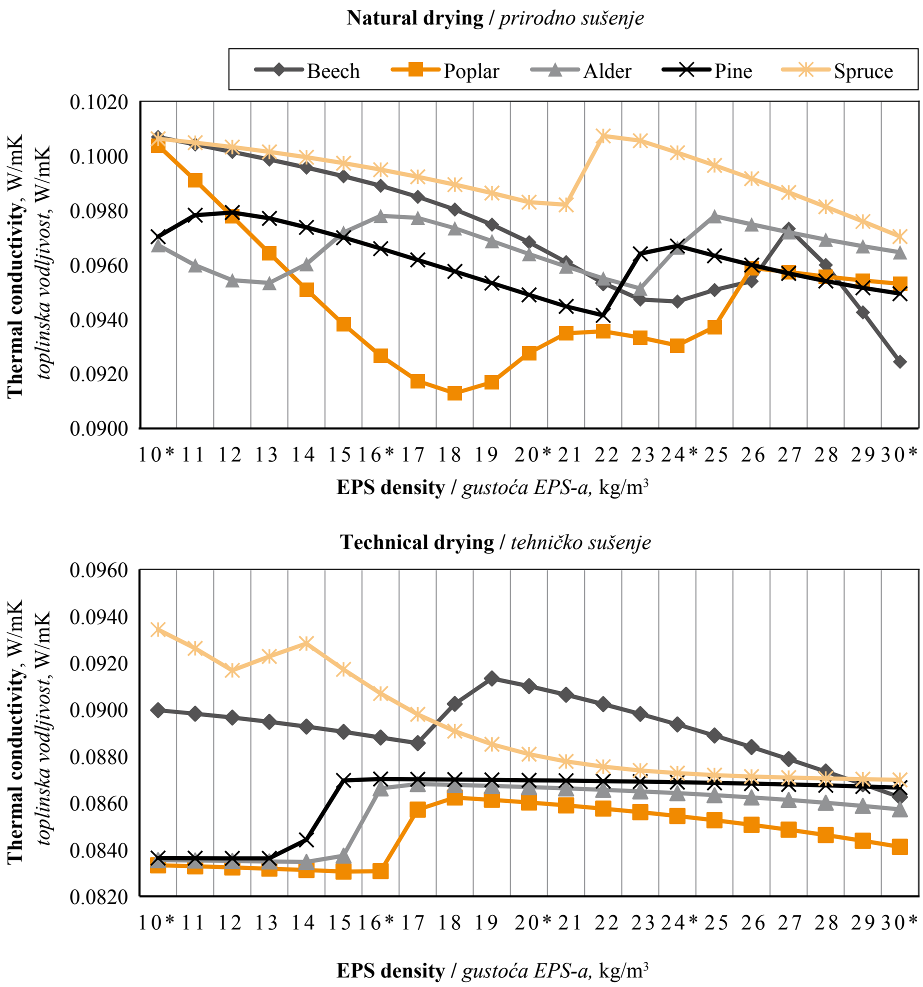

Figure 7 Changes in thermal conductivity values of PCP panels according to increasing EPS densities (*These density values were used both in experiments and ANN analysis. Thermal conductivity results related to these in graphics were obtained from ANN analysis.)
Slika 7. Promjene u toplinskoj vodljivosti PCP ploča s obzirom na povećavanje gustoće EPS-a (*Označene su vrijednosti gustoće korištene i u eksperimentima i u ANN analizi. Rezultati toplinske vodljivosti na grafikonima dobiveni su ANN analizom.)

Thanks to ANN models with low error values (MAPE and RMSE) and high performance, output values can be predicted with high accuracy for intermediate input values that are not used in experiments (Varol *et al*., 2018). In this study, the thermal conductivity coefficient values were determined by the ANN prediction model for different EPS densities; they are shown in Figure 7 according to the wood species and drying types.

Figure 7 shows the differences in thermal conductivity values among wood species according to the drying type, depending on the density of EPS waste. In the literature, it was stated that the thermal conductivity coefficient values of wooden materials might differ depending on the wood species (Kol and Sefil, 2011; Rice and Shepard, 2004). Furthermore, thermal conductivity values in wood-based panels changed depending on the types and amounts of various binders, fillers and additives (Demir *et al.,* 2016). The lowest thermal conductivity coefficient values of the beech and spruce PCP panels produced by both natural and technical drying were obtained from EPS waste foams with a density of 30 kg/m^3. In the natural drying, the thermal conductivity values of PCP panels produced with poplar, alder and pine gave the lowest values by using EPS waste foams with the density of 18, 13 and 22 kg/m^3, respectively. In the technical drying, the lowest thermal conductivity values were found in EPS waste boards with the density of 15, 14 and 11-13 kg/m^3 for PCP panels produced with poplar, alder and pine, respectively. Density, moisture content, the ratio of early and late wood zones, temperature and heat flow direction of composite materials are some factors that significantly affect the thermal conductivity of the wood-based panels (Suleiman *et al.,* 1999; Bader *et al.,* 2007; Sonderegger and Niemz, 2009; Demirkir *et al.,* 2013a).

While the PCP panels produced with poplar for both types of drying were the group that gave the lowest thermal conductivity values among wood species, spruce and beech PCP panels generally gave the highest values. It has been stated in many studies in the literature that the thermal conductivity coefficient values of wood and wood-based composites are strongly dependent on the density, and in general, the thermal conductivity values increase with the increase in density (Kamke and Zylkowski, 1989; Kol and Altun, 2009; Aydin *et al.,* 2015). Furthermore, the extractive contents in the spruce wood could be shown as a reason for the increase in the thermal conductivity of spruce composite panels. As stated in the literature, the extractive content and a number of checks and knots were other important factors affecting the thermal conductivity (Simpson and Tenwolde, 2007).

The thermal conductivity coefficient values of the composite panels produced with technically dried chips were found to be lower than the boards produced with naturally dried chips. It was known in the literature that the thermal conductivity of the panels varied depending on the temperature changes. Zhou *et al.* (2013) investigated the effect of temperature changes on the thermal conductivity of MDF panels and stated that the thermal conductivity increased with temperature up to 50 ºC and then decreased with increasing temperature between 50 ºC and 100 ºC. The density of the air in the cavities of the wood decreases depending on the increase in temperature, and therefore the heat conduction decreases (Suleiman *et al.,* 1999; Aydin *et al.,* 2015). Moreover, the thermal conductivity values also increased with increasing the moisture content of wood-based panels (Sonderegger and Niemz, 2009). Consequently, it was expected that PCP panels produced from particles dried at 90 ºC to 3 % moisture content gave lower thermal conductivity values than naturally air-conditioned PCP panels at 12 % moisture content.

4 CONCLUSIONS
4. ZAKLJUČAK

In this study, the optimum EPS densities giving the best thermal insulation performances of PCP panels produced with EPS waste foams were determined with ANN analysis. As a results of the ANN analysis, the MAPE and RMSE values of thermal conductivity prediction model in the testing data sets were determined as 1.51 % and 0.0018, respectively. The coefficient of determination (R^2) value was calculated as 0.9483 in the testing data sets. Although there was a complex and non-linear relationship between the input and output variables in the study, the performance of the model was proven with diagnostic tools and accurate, encouraging, and satisfactory results were obtained.

According to the ANN analysis results of the study, the use of EPS waste foams with a density of 30 kg/m³ increased the thermal insulation performance for both beech and spruce PCP panels. Furthermore, the lowest thermal conductivity values were obtained from the EPS waste foams with the density of 18, 13 and 22 kg/m³ for the PCP panels produced with poplar, alder and pine in the natural drying, respectively. In the technical drying, these values were found to be 15, 14 and 11-13 kg/m³, respectively. Poplar species is the wood species that gave the best thermal insulation performance. Technical drying performed much better than natural drying in terms of thermal conductivity. By utilizing the results of this study, PCP panels with the best thermal insulation performance will be produced quickly at low costs, and this will contribute to the recycling of waste EPS foams.

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Combustion and Decay Resistance Performance of Scots Pine Treated with Boron and Copper Based Wood Preservatives

Otpornost prema gorenju i propadanju borovine zaštićene sredstvima na bazi bora i bakra

ORIGINAL SCIENTIFIC PAPER
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ABSTRACT • *Boron compounds in the form of boric acid, borax or disodiumoctaborate tetrahydrate have been used as insecticide, fungicide and fire retardant in wood preservatives industry for decades. Also, copper is the most commonly used component in most of relatively modern preservatives as it is highly effective against fungi. The objective of this study was to investigate the combustion and decay resistance of boron-copper based solutions which were developed by our group. These solutions contain boric acid, sodium borate decahydrate, copper hydroxy carbonate, ethanolamine, quaternary ammonium compound (benzalkonium chloride), and/or organic acid (octanoic acid). Scots Pine (Pinus sylvestris) woods were treated with the preservatives according to ASTM D1413-07 standard by vacuum-pressure impregnation system, which was developed by our group. Decay resistance performances against white and brown rot fungi were determined according to EN 113 standard and combustion tests were performed with respect to ASTM E160-50 standard. All the impregnated wood samples were found highly resistant to both white (Trametes versicolor L.) and brown (Coniophora puteana L.) fungi. Besides, they gave better results than the control samples in terms of combustion tests.*

KEYWORDS*: wood preservatives; decay resistance; boron compounds; combustion resistance filler*

SAŽETAK • *Spojevi bora poput borne kiseline, boraksa ili dinatrijeva oktaborata tetrahidrata već se desetljećima u industriji sredstava za zaštitu drva proizvode kao insekticidi, fungicidi i usporivači gorenja. Usto, bakar je najčešća komponenta u većini relativno modernih zaštitnih sredstava jer je visoko učinkovit u zaštiti od gljiva. Cilj ove studije bio je istražiti otpornost prema gorenju i propadanju drva bora zaštićenog otopinama na bazi bora i bakra koje je razvila naša grupa. Te otopine sadržavaju bornu kiselinu, boraks, bakrov(II) karbonatni hidroksid karbonat, etanolamin, kvaterni amonijev spoj (benzalkonijev klorid) i/ili organsku kiselinu (oktansku kiselinu). Uzorci borovine (Pinus sylvestris) obrađeni su zaštitnim sredstvima prema standardu ASTM D1413-07 postupkom impregnacije pod vakuumom koji je razvila naša grupa. Otpornost prema gljivama bijele i smeđe truleži određena*

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je prema standardu EN 113, a ispitivanje gorenja provedeno je prema standardu ASTM E160-50. Svi impregnirani uzorci drva pokazali su se vrlo otpornima i na bijele (Trametes versicolor L.) i na smeđe (Coniophora puteana L.) gljive. Osim toga, pri ispitivanju gorenja pokazali su bolje rezultate od kontrolnih uzoraka.

KLJUČNE RIJEČI: *sredstva za zaštitu drva; otpornost prema propadanju; spojevi bora; otpornost prema gorenju*

1 INTRODUCTION
1. UVOD

Wood is both an ecological and sustainable natural resource. It is frequently preferred both indoors and outdoors because it is easy to maintain and repair, aesthetically pleasing, durable and has a wide range of use. In addition to its light and durable structure, it provides good thermal insulation and is therefore an ideal construction material. However, if the necessary care and conditions are not provided, deterioration occurs in wood. Materials that are in direct contact with weather conditions in the external environment are subject to more deformation. Biological pests such as bacteria, fungi, insects, termites, adverse weather conditions, exposure to humidity and sunlight are among the factors that accelerate the deterioration of wood. Thermal stability and durability are significant for the wood material. Therefore, wood preservatives are used to kill fungi, bacteria or insects directly, or to provide higher thermal stability (Ramage *et al.,* 2001; Bekhta and Niemz 2003).

Wood preservation is based on the impregnation of wood with biocides such as creosote, arsenic, zinc, copper, boron, chromium, etc. to prevent degradation of wood and to eliminate the appropriate nutrient environment for the growth of microorganisms. In the wood preservation industry, the main change in the process that has been developing since the beginning of the twentieth century has occurred in wood preservatives rather than in wood preservation methods. In recent years, growing environmental concerns have led to drastic modifications in the active ingredients of wood preservatives in many countries, such as eliminating or restricting the use of creosote and chromate copper arsenate (CCA). Alternatively, wood preservatives containing copper, boron and organic biocides as active ingredients are used in many countries. In this context, recent prohibitions on the use of these toxic impregnants have led the wood protection industry to use and develop wood preservatives based on organic or inorganic compounds such as alkali copper quaternary ammonium compounds (ACQ-1, ACQ-2), copper azole and copper-HDO. ACQ wood preservatives are classified into two types based on their composition: ACQ-1 contains copper and benzalkonium chloride (BAC) and ACQ-2 contains copper and didecyldimethyl ammonium chloride (DDAC) (Koski, 2008; Humar *et al*., 2005; Tomak, 2011).

Copper is the most commonly used component in most of the relatively modern preservatives. The copper cation has been reported to be adsorbed or to form complexes with phenolic groups of lignin or cellulose (Richardson, 2003; Lebow, 1996). Recently, amines have been frequently used to prevent the leaching of copper from wood. Therefore, amines that act like a ligand and thus affect the stability, polarity and solubility of copper amine complexes appear very efficient in fixing copper into wood (Humar *et al.,* 2001).

The use of boron compounds, known as environmentally friendly impregnating agents, has an important place in this respect and their importance is increasing day by day. Borates (borax, boric acid, disodium octaborate tetrahydrate) are inorganic, colorless and odorless boron-based biocides that are noncorrosive to metal fasteners and readily soluble in water. Boron compounds exhibit both fungicidal and insecticidal properties against wood destroying insects and fungi (Tomak *et al.,* 2011; Freeman, 2008; Terzi *et al.,* 2017). In addition, when boron compounds are exposed to heat, they form a glassy structure in the wood, reducing the rate and spread of flammable gases and preventing the movement of thermal decomposition products. Boric acid reduces combustion in the form of ember, but does not completely prevent the spread of the flame (Freeman, 2008; Townsend and Solo-Gabriele, 2006; Yamaguchi, 2003). Borax prevents the spread of flame. In different studies, boric acid and borax were used together and it was determined that wood material had higher burning resistance (Baysal *et al.,* 2003).

Boron compounds are considered to be more effective preservatives than copper and zinc compounds due to their wide fungicidal and insecticidal effects. The reason why copper and zinc perform better is not their natural fungicidal activities, but their fixation in wood (Obanda *et al.,* 2008). However, the boron is susceptible to rapid leaching particularly in outdoor exposure. The boron compounds are well diffusible substances to the wood and as a result, they can be leached easily when in contact with water. The reason for easy diffusion and leaching of boron in wood is that the molecules cannot be fixed to the cell wall. Boron does not react with the cell wall, but can form complexes with hydroxyl groups. A significant potential site for the absorption of boron is the hydroxyls of the carboxylic acids and phenolic groups. In recent years, a lot of research has been done on delaying or preventing leaching of boron compounds from wood and increasing the potential use of boron compounds. Nevertheless, the leaching resistant results reported so far appear generally poor (Townsend and Solo-Gabriele, 2006; Yamaguchi, 2003; Baysal *et al.,* 2003; Obanda *et al.,* 2008; Yalinkilic *et al.,* 1999). In addition, in our previous study, in which the retention and leaching properties of boron and copper were determined, it was observed that the leaching of boron and copper was significantly reduced, especially with the use of octanoic acid (Yildiz *et al.,* 2019).

The objective of this study was to investigate decay and fire resistance of the wood samples impregnated with boron-copper based solutions. Boric acid, borax, copper hydroxy carbonate, ethanolamine, octanoic acid and benzalkonium chloride were used as chemical substances of 4 different preservative solutions. Scots Pine (*Pinus sylvestris*) wood was treated with the preservatives by vacuum-pressure impregnation system according to Bethell method. Decay resistance performances against white and brown rot fungi were determined and combustion tests were performed for the impregnated wood samples as well as for one unimpregnated control sample. Consequently, the effects of the substances in decay resistance and combustion tests were investigated. The aim of impregnation with developed chemicals was to protect the wood against fungi, to increase its resistance to flame and, accordingly, to extend its life.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

The sapwood samples used in the study were obtained from the Scots pine (*Pinus sylvestris*) trees of Bolu Dörtdivan region. The logs of wood were selected as healthy, smooth fibrous, knotless, exhibiting normal growth, undamaged by fungus and insect pest. Sapwood blocks were cut in dimensions of 5 mm × 15 mm × 30 mm for fungal decay-resistance tests, and 13 mm × 13 mm × 76 mm for combustion tests. Before treatment, all wood blocks were conditioned at (20±2) °C and (65±5) % relative humidity for two weeks.

The impregnation chemicals used in the study were water based and the contents were boric acid (Eti Mining Corp.), borax decahydrate (Eti Mining Corp.), copper hydroxy carbonate (Tekkim), ethanolamine (Merck), benzalkonium chloride (Tekkim) and octanoic acid (Merck). Four different impregnation chemicals coded as BC1-BC4 were prepared at the molar ratios shown in Table 1 for 1000 mL of concentrated solution.

Figure 1 Vacuum-pressure wood impregnation system
Slika 1. Vakuumsko-tlačni sustav za impregnaciju drva

Concentrated impregnation chemicals obtained as a result of all processes were mixed in concentrations of 3 % in water and impregnated into the wood placed in the vacuum-pressure tank by Bethell Method according to ASTM D1413-07 (ASTM 2007). To accomplish this, specimens were vacuumed under pressure of 60 mmHg for 30 min and then placed in a solution under 10 bar pressure for 60 min. The impregnation processes of the solutions into the wood were carried out in a vacuum-pressure wood impregnation system within the Boron Research Institute (Figure 1). The samples were left to dry at room temperature after impregnation.

Table 1 Molar concentration (mol/L) of concentrated solution
Tablica 1. Molarna koncentracija (mol/L) koncentrirane otopine

Component / *Komponenta*	**BC1, mol/L**	**BC2, mol/L**	**BC3, mol/L**	**BC4, mol/L**
Boric acid / *borna kiselina*	0.60	0.60	0.68	0.60
Borax / *boraks*	0.06	0.06	0.08	0.06
Copper hydroxy carbonate *bakrov(II) karbonatni hidroksid*	0.47	0.47	0.34	0.47
Ethanolamine / *etanolamin*	3.76	3.76	2.82	3.76
Octanoic acid / *oktanska kiselina*	-	0.34	0.34	0.34
Benzalkonium chloride *benzalkonijev klorid*	-	-	0.08	0.12

2.1 Retention and Leaching Tests
2.1. Ispitivanje retencije i ispiranja

Sapwood blocks were cut in dimensions of 20 mm × 20 mm × 20 mm from Scots pine logs. Before treatment, all wood blocks were conditioned at (20±2) °C and (65±5) % relative humidity for two weeks. Each sample group was subjected to leaching procedure separately. Five cubes of treated wood specimens were submerged in 400 mL of distilled water. Afterwards, the leachate was removed and replaced with fresh distilled water after 6, 24, 48, 72, 96, and 120 hours in a sequence. Each leachate sample was collected and stored for copper and boron analyses. Vibratory Disc Mill was used to grind the impregnated wood specimens for 1.5 minutes, a sample of each variation was taken in a beaker and nitric acid (65 % Merck) was added. The solutions were filtered using filter paper and then diluted with distilled water after acid treatment. Inductively Coupled Plasma-Mass Spectrometer (ICP-MS Perkin Elmer) was used to determine the amount of boron and copper in impregnated and leachate samples.

2.2 Decay resistance tests
2.2. Ispitivanje otpornosti prema propadanju

Decay resistance performances against white and brown rot fungi were determined according to modified EN 113 (EN 1996) standard. For the Scots pine, experiments were carried out on two different fungus species and four different solution variations. The sample sizes of wood were modified to 5 mm x 15 mm x 30 mm dimensions specified in the standard and then oven dry weights of the prepared samples were measured. After the impregnation process, the wood samples were dried in the air-conditioning chamber at (20±2) °C and (65±5) % relative humidity (Figure 2a) and then placed in each petri dish as one test and one control sample, with 20 replicates for each variation. 48 % malt-agar mixture was used for the growth medium of the fungi. In order to sterilize the prepared solution, the flasks were covered with aluminum foil and kept in an autoclave at 121 °C for 20 minutes and allowed to cool in the inoculation cabinet. After cooling well, approximately 23 mL was poured into each petri dish. After the white (*Trametes versicolor* L.) and brown (*Coniophora puteana* L.) rot fungi were inoculated into the nutrient media, the petri dishes were kept in the air conditioning chamber until the fungal growth was completed. At the end of the period, the test and control samples were placed in the petri dishes before the decay and were placed in the incubator at (22±1) °C and (70±5) % relative humidity and out of light for 8 weeks (Figure 2b). Then, the samples were taken from petri dishes and kept in the oven at (103±2) °C until they reached constant weight and their weights were recorded as full dry weight after fungi attack. Mass loss (*ML*) was calculated according to the following Eq.

$$ML(\%) = \frac{M_0 - M_1}{M_0} \cdot 100 \qquad (1)$$

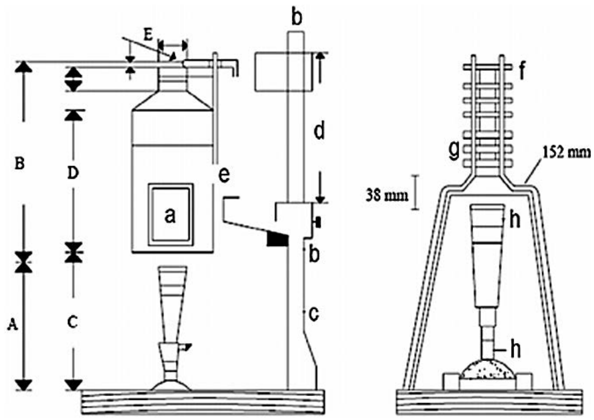
Where, M_1 (g) is the final dry weight of specimens after fungal exposure and M_0 (g) is the initial dry weight of samples.

Also, in this study, the strengths of Scots pine samples impregnated with preservatives according to EN 350 (EN 2016) standard were classified. It accepts a maximum of 3 % mass loss in standard test specimens and five strength classes are formed based on *X* value (Test *ML* (%) / Control *ML* (%)). These strength classes are: very durable $X \leq 0.15$; $0.30 \geq$ durable > 0.15; $0.60 \geq$ moderately durable > 0.30; $0.90 \geq$ light resistant > 0.60; undurable > 0.90.

2.3 Combustion tests
2.3. Ispitivanje gorenja

Combustion tests were performed according to ASTM E160-50 standard (ASTM 1975). The impregnated and control specimens were adjusted to (27±2) °C temperature and 30 % - 35 % relative humidity in the air-conditioning cabinet prior to the combustion process. Each stand, with 24 samples, was placed verti-

Figure 2 Test specimens (a) in air-conditioning chamber, (b) in petri dishes
Slika 2. Ispitni uzorci: a) u klimatizacijskoj komori, b) u Petrijevim zdjelicama



a. Mica glass
b. End of slide
c. Flame burner guide
d. Slide
e. Millivolt meter inlet
f. Wood specimens
g. Wire cage
h. Flame burner

Figure 3 Combustion test device
Slika 3. Uređaj za ispitivanje gorenja

cally, 2 samples on each floor. In each experiment, one control and impregnated wood samples with four different impregnation chemicals were combusted. Tests for each parameter were performed in triplicate. Samples were exposed to a (25±2) cm flame, under a gas pressure of 0.5 kg/cm^2 for 180 seconds. Following the combustion with fire, the flame was extinguished and the samples were allowed to combust autogenously until they collapsed. Temperature changes during combustion were determined using a thermometer. During the combustion process, the gas pressure was kept constant at the level specified in the standard, and the combustion test parameters were measured for three combustion stages as flame source combustion, non-flame source combustion and glowing combustion. Combustion test device is shown in Figure 3.

In order to determine the effect of flame source combustion, non-flame source combustion and glowing combustion on the impregnated wood samples, the variance analysis was applied to the groups. According to the analysis of variance, the importance levels of the significant factors were determined by Duncan test.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

The amount of boron-copper retention and leaching rates are shown in Table 2. BC1 samples showed lower retention of boron and copper. Also, almost all boron was leached from the impregnated wood in BC1. As can be seen from the results of BC2, the relative increase in boron and copper fixation is considered to be due to the addition of octanoic acid. However, when BC3 and BC4 samples were examined, it was observed that the addition of benzalkonium chloride to the solution slightly reduced the retention and leaching properties of boron. As seen from all the results in Table 2, octanoic acid and especially benzalkonium chloride enhanced copper retention, and BC4 solution had the maximum copper retention level.

Decay resistance performances against white *(Trametes versicolor)* and brown *(Coniophora puteana)* rot fungi and combustion test results have been analyzed. Decay resistance tests ended after malt-agar mixture had been completely dry in 4 months. Mass loss of the untreated control samples was between 8 - 10 %. Mass loss of control samples and impregnated wood materials and chemical mass loss prevention ratios were calculated and it was determined that all impregnation chemicals protect wood against fungi. The results of the decay test are given in Figure 4.

The highest mass loss of 1.01 % was observed in BC1 sample for *Trametes versicolor* fungi. The whole mass loss of other samples was below this value. According to the test results, mass loss of BC2, BC3 and BC4 samples was 0.75 %, 0.50 % and 0.58 %, respectively. The success of the solutions used in the experiments in preventing mass loss was found to be statisti-

Table 2 Boron copper retention and percentage released from wood samples treated with various solutions
Tablica 2. Retencija bora i bakra te postotak otpuštanja bora i bakra iz uzoraka drva zaštićenog različitim otopinama

Solution types *Vrsta otopine*	**Avg. boron retention** *Srednja vrijednost retencije bora,* kg/m^3	**Avg. copper retention** *Srednja vrijednost retencije bakra,* kg/m^3	**Avg. boron released** *Srednja vrijednost otpuštanja bora,* %	**Avg. copper released** Srednja vrijednost otpuštanja bakra, %
BC1	0.203±0.04	0.834±0.03	99.52±0.4	10.49±0.3
BC2	0.279±0.02	0.909±0.04	74.34±0.6	5.98±0.2
BC3	0.258±0.01	0.905±0.03	75.88±0.5	4.79±0.2
BC4	0.233± 0.02	1.290 ± 0.05	81.44±0.7	4.43±0.2

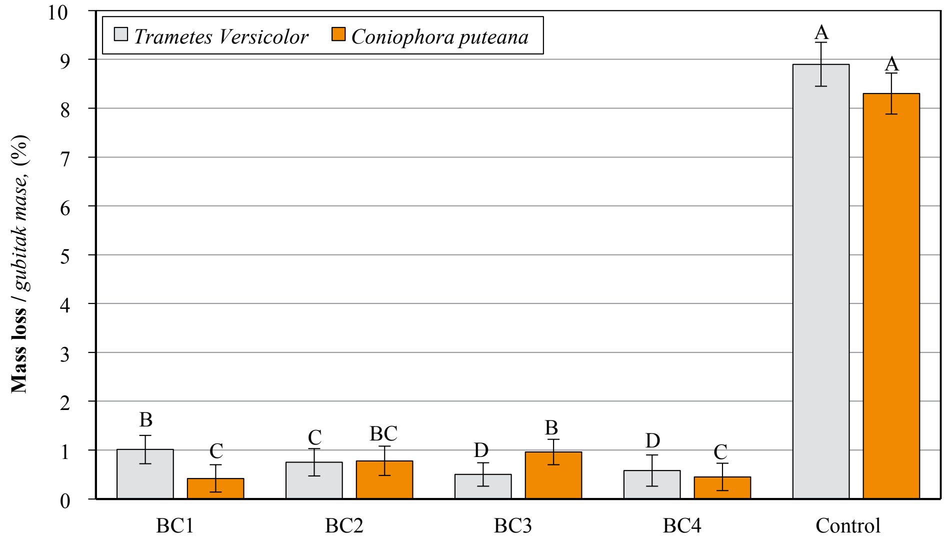


Figure 4 Mass loss (%) of samples exposed to *Trametes versicolor* and *Coniophora puteana* (Number followed by the same letter indicates no statistically significant differences according to the Least-Significant-Difference Test with 0.95 confidence)
Slika 4. Gubitak mase (u %) uzoraka izloženih gljivama *Trametes versicolor* i *Coniophora puteana* (broj iza kojeg slijedi isto slovo znači da nema statistički značajnih razlika prema testu najmanje značajne razlike s pouzdanošću od 0,95)

cally significant; however, the difference between the BC3 and BC4 solutions was found to be statistically insignificant. Also, *Coniophora puteana* fungi caused the mass loss of 0.96 % on BC3 as the highest value. Mass loss of BC1, BC2 and BC4 samples was 0.42 %, 0.78 % and 0.45 %, respectively. Considering the mass losses, in terms of *Trametes versicolor* fungus, its success in preventing mass loss was statistically significant, but the difference between the BC1 and BC4 solutions was found to be statistically insignificant.

Test results have shown that all prepared chemicals are highly resistant to both *Trametes versicolor* and *Coniophora puteana* fungi. The effects of copper and boron compounds mentioned previously in various studies on fungi were found to be compatible with the test results of this study (Kartal *et al.,* 2019; Humar *et al.,* 2007; Cao and Kamdem, 2004; Mourant *et al.,* 2009; Thevenon *et al.,* 2009; Kartal *et al.,* 2004).

According to the test results, it was determined that octanoic acid increased the resistance of impregnated wood against *Trametes versicolor*, while the resistance of wood against *Coniophora puteana* fungi was slightly decreased. The efficacy of octanoic acid against fungi has also been demonstrated in different studies (Humar *et al*., 2007; Schmidt, 1985). By addition of benzalkonium chloride, fungal effect has been more significant against *Trametes versicolor* and *Coniophora puteana*. Benzalkonium chloride is a salt of quaternary ammonia compounds and known as disinfectant and fungicidal activity in the literature (Terzi *et al*., 2011; Pernak *et al*., 2004; Preston and Nicholas, 1982) which corresponds to the results of this study.

The results of the combustion tests are given in Table 3. It was observed that the temperatures formed in the combustion with flame source, non-flame combustion and glowing combustion were in the range of 150-185 °C, 460-530 °C and 200-235 °C, respectively. These temperatures have delayed the ignition. For the Scots pine control samples, temperatures of 227.3 °C in the combustion with flame source, 532.7 °C in the non-flame combustion and 237.0 °C in the glowing combustion were measured. These results show that impregnated wood samples have better flame resistance than control samples.

BC1 samples (150.1 °C) gave the most positive results in terms of temperature during the combustion with flame source, while BC4 samples (184.7 °C) gave the most negative results except for the control samples. However, depending on the temperature of the control samples, this value can still be considered positive.

The lowest temperature values were obtained in the samples impregnated with BC3 (461.3 °C) and BC4 (495.7 °C) chemicals during combustion in non-flame source stage. The temperature of wood impregnated with chemical BC2 reached the highest value (530.3 °C) and a result similar to the control group was encountered. The lowest temperature value of the core during the glowing combustion stage was observed in BC1 (201.0 °C) and BC3 (202.3 °C) samples. The temperature of wood impregnated with chemical BC2 reached the highest value (232.0 °C) and a result close to the control group was obtained.

When taking into consideration the results of the combustion tests, it is seen that the wood samples impregnated with boron compounds known to increase the flame resistance give better results than the control samples. The fire-retardant effect of boron compounds

Table 3 Results of Duncan tests for combustion properties
Tablica 3. Rezultati Duncanova testa za svojstva gorivosti

Solution types *Vrsta otopine*	**Combustion properties** / *Svojstva gorivosti*		
	Combustion with flame source *Gorenje s izvorom plamena,* °C	**Non-flame source combustion** *Gorenje bez izvora plamena,* °C	**Glowing combustion** *Gorenje žarenjem,* °C
BC1	150.1 ± 6.2^{A}	509.7 ± 14.6^{BC}	201.0 ± 4.4^{A}
BC2	154.3 ± 7.1^{AB}	530.3 ± 16.4^{C}	232.0 ± 13.6^{C}
BC3	168.1 ± 7.8^{ABC}	461.3 ± 18.7^{A}	202.3 ± 4.7^{A}
BC4	184.7 ± 3.5^{C}	495.7 ± 19.1^{B}	219.3 ± 9.9^{B}
Control / *kontrolni uzorak*	227.3 ± 10.6^{D}	532.7 ± 13.9^{CD}	237.0 ± 16.1^{C}

Number followed by the same letter indicates no statistically significant differences according to the Least-Significant-Difference Test with 0.95 confidence. / *Broj iza kojeg slijedi isto slovo znači da nema statistički značajnih razlika prema testu najmanje značajne razlike s pouzdanošću od 0,95.*

has already been proven by different studies (Örs *et al.,* 1999; Baysal *et al.,* 2003; Temiz *et al.,* 2008). However, it can be seen that the increase in the amount of organic matter in the impregnation chemical is an important factor in the results obtained near the temperature of the control samples.

The addition of octanoic acid to the impregnation chemical did not cause a significant change in combustion with flame temperatures, but caused a significant increase in temperature compared to the results of non-flame combustion and glowing combustion. This result showed that octanoic acid in general decreased the flame resistance partially. Benzalkonium chloride, on the other hand, caused a notable increase in the combustion with flame source temperature and caused lower combustion temperatures in case of non-flame combustion and glowing combustion.

4 CONCLUSIONS
4. ZAKLJUČAK

This study investigates the combustion and decay resistance properties of boron-copper based solutions which were developed by our group. Scots pine (*Pinus sylvestris*) wood was treated with the preservatives by vacuum-pressure impregnation system. According to the decay tests, all prepared chemicals are highly resistant to both *Trametes Versicolor* and *Coniophora puteana* fungi. In addition to boron and copper, octanoic acid and benzalkonium chloride to some degree also provide fungicidal efficacy in the wood. The combustion test results demonstrate that, while boron compounds have a significant fire-retardant impact on the wood, octanoic acid decreases the flame resistance feebly. Furthermore, benzalkonium chloride causes a slight increase in the combustion with flame source temperature and lower combustion temperatures in case of non-flame combustion and glowing combustion.

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Characterization and Separation of Lignin from Kraft Black Liquor with Different Alcohols

Karakterizacija i odvajanje lignina iz crnog luga dobivenog kraft metodom uz primjenu različitih alkohola

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ABSTRACT • *The aim of this study was to investigate the properties of lignin obtained from black liquors of Scots pine (Pinus sylvestris L.) and European aspen (Populus tremula L.) woods cooked by Kraft method. In the study, the cooking process was carried out according to parameters such as: 1/4 of wood/liquor ratio, cooking temperature at (170±2) °C, and cooking time of 90 min. After the cooking process, the black liquor was taken to a beaker from the digester, and lignin was recovered in different ways from the black liquor with methyl alcohol (MeOH) and ethyl alcohol (EtOH). The material properties of the recovered lignin were analyzed by scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FT-IR), x-ray diffraction (XRD), and thermogravimetric analysis (TGA). Commercial lignin (cml-lignin) was used to compare the properties of lignin obtained from black liquor. SEM images showed that the particle sizes of lignin obtained with EtOH and MeOH were similar. In FT-IR, it was detected that the recovered lignin types have similar functional groups, while some differences were observed in intensities of the peaks. XRD results revealed that all lignin types were found to have a similar structure with the cml-lignin, and that alcohol types used during the recovery process of lignin do not have an important effect on the structural properties of lignin. The TGA results indicated that the thermal stability of recovered lignin has better thermal stability than cml-lignin. The results showed that the recovered lignin was similar to commercial lignin and that it can be recovered from the liquor leftover from Kraft pulping by the method described and used in industry.*

KEYWORDS: *Kraft pulping; black liquor; lignin, recycling; lignin characterization*

SAŽETAK • *Cilj rada bio je istražiti svojstva lignina dobivenog od crnog luga drva bijelog bora (Pinus sylvestris L.) i jasike (Populus tremula L.), kuhanoga kraft metodom. Proces kuhanja u ovom je istraživanju proveden uz ove parametre: omjer drva i luga 1 : 4, temperatura kuhanja 170 ± 2 °C, vrijeme kuhanja 90 min. Nakon procesa kuhanja crni je lug premješten iz digestora u čašu te je lignin metilnim alkoholom (MeOH) i etilnim alkoholom (EtOH) na različite načine izdvojen iz crnog luga. Svojstva izdvojenog lignina analizirana su skenirajućim elektronskim mikroskopom (SEM), infracrvenom spektroskopijom s Fourierovom transformacijom (FT-IR), difrakcijom X-zraka*

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(XRD) i termogravimetrijskom analizom (TGA). Za usporedbu svojstava lignina dobivenog iz crnog luga poslužio je komercijalni lignin (cml-lignin). SEM slike pokazale su da su veličine čestica lignina dobivenog uz pomoć EtOH i MeOH bile slične. FT-IR analizom utvrđeno je da izdvojeni tipovi lignina imaju slične funkcionalne grupe te da su se pojavile neke razlike u intenzitetu pikova. Rezultati XRD-a otkrili su da svi tipovi lignina imaju sličnu strukturu kao i cml-lignin i da vrsta alkohola upotrijebljenog tijekom procesa izdvajanja lignina nema bitan učinak na strukturna svojstva lignina. Rezultati TGA analize pokazali su da je toplinska stabilnost izdvojenog lignina bolja od toplinske stabilnosti cml-lignina. Prema rezultatima istraživanja može se zaključiti da je izdvojeni lignin sličan komercijalnom ligninu, da može biti izdvojen iz tekućine koja je preostala od proizvodnje celuloze opisanom kraft metodom te da se može rabiti u industriji.

KLJUČNE RIJEČI: *proizvodnja celuloze kraft metodom; crni lug; lignin; recikliranje; karakterizacija lignina*

1 INTRODUCTION
1. UVOD

The pulp and paper industry is an important sector that produces paper and paper-based materials from biomaterials such as wood, vegetable plants, and other biomass. In the world, many paper mills have manufactured paper products with a chemical pulping technique in huge amounts (Sasaki and Goto, 2008; Zhang *et al.,* 2019; Do *et al.,* 2020). Large amounts of energy, water, and biomass have been used to obtain the pulp/paper, generally without recoverying the chemicals after the production of pulping or using recovering systems that are not sufficient to clean the chemical wastes such as black liquor, other cooking solvents, etc. After the pulping process, the chemical wastes are generally discharged to water resources such as rivers, seas, oceans, etc. In order to reduce the waste, they were burned for heat because of the great amount of lignin in chemical wastes (Lateef *et al.*, 2009; Zainab *et al.,* 2018) causing major environmental pollution in many developing countries. The wastewater contains several biomaterials such as lignin, hemicellulose, etc. and efforts have been made to recover the biomaterials in different ways; lignin, as one of the biomaterials, is a commercially important biopolymer (Rydholm, 1965; Sun *et al.,* 1999; Lake and Blackburn 2014).

Lignin is an aromatic amorphous polymer, which is composed of 5 – 500 phenylpropan units and it has a cross-linked structure both with itself and with other elements such as hemicellulose and cellulose. Lignin has several monomers such as sinapyl, coniferyl and coumaryl alcohols according to the wood species (Tomani, 2010; Gordobil *et al.,* 2016; Hubbe *et al.,* 2019). Previous research has been conducted on the recovery of lignin from black liquor with different methods such as alkaline or acidic methods by Sun *et al.* (1999), Kouisni *et al.* (2011), Lubis *et al.* (2012), Kouisni *et al.* (2012), Ragauskas *et al.* (2014). Kamble and Bhattacharyulu (2015) investigated the separation and characterization of lignin from black liquor waste by inorganic and organic acids. The obtained results showed that the mixture of phosphoric acid and acetic acids at the reaction temperature of 50 °C was determined as the best chemical for lignin separation. Thermogravimetric analysis results of the obtained lignin showed that lignin from different acids differs significantly in thermal decomposition. In another study, Maitz *et al.* (2020) studied the preparation and characterization of lignin obtained from black liquor by two methods such as water-washed and acid-washed process. According to the results obtained in previous studies, it was found that acid-washed lignin has a higher molecular weight than water-washed lignin; however, the content of hydroxyl and methyl groups of each lignin was similar to each other. In another study, Sameni et al. (2016) investigated the characterization of lignin isolated from kraft black liquor. The obtained results showed that the amount of lignin isolated was 27 % of black liquor solids and all types of lignin obtained in different ways were found to exhibit similar physicochemical properties. As a result, the literature review showed that the recovery of lignin, as by-products to value-added materials, is an important issue in obtaining eco-friendly materials and reducing the global warming problems. Consequently, the aim of this research is to isolate the lignin from chemical wastes (named black liquor) of two types of biomass, Scots pine and European aspen woods, with two different solvents such as EtOH and MeOH. The material properties such as thermogravimetric analysis (TGA), Fourier transform infrared spectroscopy (FT-IR), x-ray diffraction analysis (XRD), and scanning electron microscopy (SEM) of the recovered lignin were investigated.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

Scots pine (*Pinus sylvestris* L.) and European aspen (*Populus tremula* L.) woods were supplied from a local mill in Bartin City, Turkey. Wood materials were chipped by hand using a hammer and knife. The obtained chips of about 4 cm × 2.5 cm × 1 mm were dried under room conditions (about 25 °C). The cooking process was done in an electrically heated rotary digester. The cooking parameters are given in Table 1.

Table 1 Cooking parameters
Tablica 1. Parametri kuhanja

Wood Type *Vrsta drva*	**Wood / liquor** *Drvo / lug*	**Active alkali (NaOH), %** *Aktivna lužina* (NaOH), %	**Sulfidity (Na_2S), %** *Sulfidnost* (Na_2S), %	**Cooking temperature, °C** *Temperatura kuhanja*, °C	**Reach time to cooking temperature, min** *Vrijeme dosezanja temperature kuhanja*, min	**Cooking time, min** *Vrijeme kuhanja*, min
Pine / *borovina*	4/1	18	25	170±2	90	75
Aspen / *jasikovina*	4/1	18	25	170±2	90	75

A B

Figure 1 Obtaining (A) and recovery process (B) of lignin from black liquor
Slika 1. Dobivanje (A) i proces izdvajanja (B) lignina iz crnog luga

After kraft pulping, the black liquor was taken from the digester with a beaker as visually presented in Figure 1(A). After Kraft pulping process, the black liquor was taken with a beaker from the digester as shown in Figure 1(A). The top surface of the beaker was covered with a paraffin film and stored in a dark cabinet.

Commercial lignin (cml-lignin) was supplied from Canadian Lignin Inc. for comparison with lignin from black liquor.

2.2 Recovery process of lignin
2.2. Proces izdvajanja lignina

In this study, lignin was obtained by precipitating with MeOH and EtOH. 10 mL black liquor was added to 50 ml beaker, and four different beakers were prepared to isolate the lignin from Kraft black liquor of pine and aspen wood with MeOH and EtOH. The solutions were put on a heater plate at 100 °C for 30 min. All water content was meant to evaporate, and the solid residue of black liquor was obtained for the next processing. The solid residues of black liquor were solved with 60 % MeOH and EtOH in 50 mL beakers, respectively, and then three drops of hexane were added to the solutions. The resulting solution was kept under laboratory conditions for one day, and then it was filtered with a special filter paper for the analysis as given in Figure 1(B).

2.3 Characterization technics
2.3. Tehnike karakterizacije

2.3.1 Morphological analysis
2.3.1. Morfološka analiza

The samples were morphologically characterized with TESCAN MAIA3 scanning electron microscopy (SEM). First, the samples were covered with gold nanoparticles by using a coater, and SEM analysis was conducted at an accelerating voltage of 15 kV.

2.3.2 Fourier Transform Infrared Spectroscopy (FT-IR)
2.3.2. Infracrvena spektroskopija s Fourierovom transformacijom (FT-IR)

A Shimadzu IRA Affinity-1 spectrometer was used for the chemical characterization of the samples. The device included a single-reflection ATR pike MIRacle sampling attachment. Spectra of FT-IR was between 800 cm^{-1} and 4000 cm^{-1} wavenumbers at a resolution of 4 cm^{-1}. Three specimens for each formulation were scanned and the average spectra were used.

2.3.3 X-Ray Diffraction Analysis (XRD)
2.3.3. Analiza difrakcije X-zraka (XRD)

A high-resolution X-ray diffractometer (Model Model Rigaku SmartLab, PAN Analytical, Netherlands) with Ni-filtered Cu Ka (1.540562 Å) radiation source operated at 45 kV voltage and 40 mA electric current was used in the structural characterization. The specimens were scanned in the range from 5° to 90° 2θ with 0.02° steps. A silicon zero-background plate was used to make sure that there was no peak associated with the sample holder. The same sample holder and the same position of the holder were used for all tests. The crystallinity index (CI) of the powdered samples was calculated as the ratio of the total area under the resolved crystalline peaks to the total area under the unresolved X-ray scattering curve (Rabiej, 2003). CI values were found using the formulation given below:

$$CI = \frac{\Sigma A_c}{\Sigma(A_c + A_a)} (\%) \tag{1}$$

Where A_c is the integrated area under the respective crystalline peaks, and A_a is the integrated area of the amorphous halo.

2.3.4 Thermogravimetric analysis
2.3.4. Termogravimetrijska analiza

The thermal stability of lignin is an important issue in many applications; therefore, the thermal properties of the lignin particles were investigated using a thermogravimetric analyzer (TGA/Q6 DTG) (Perkin

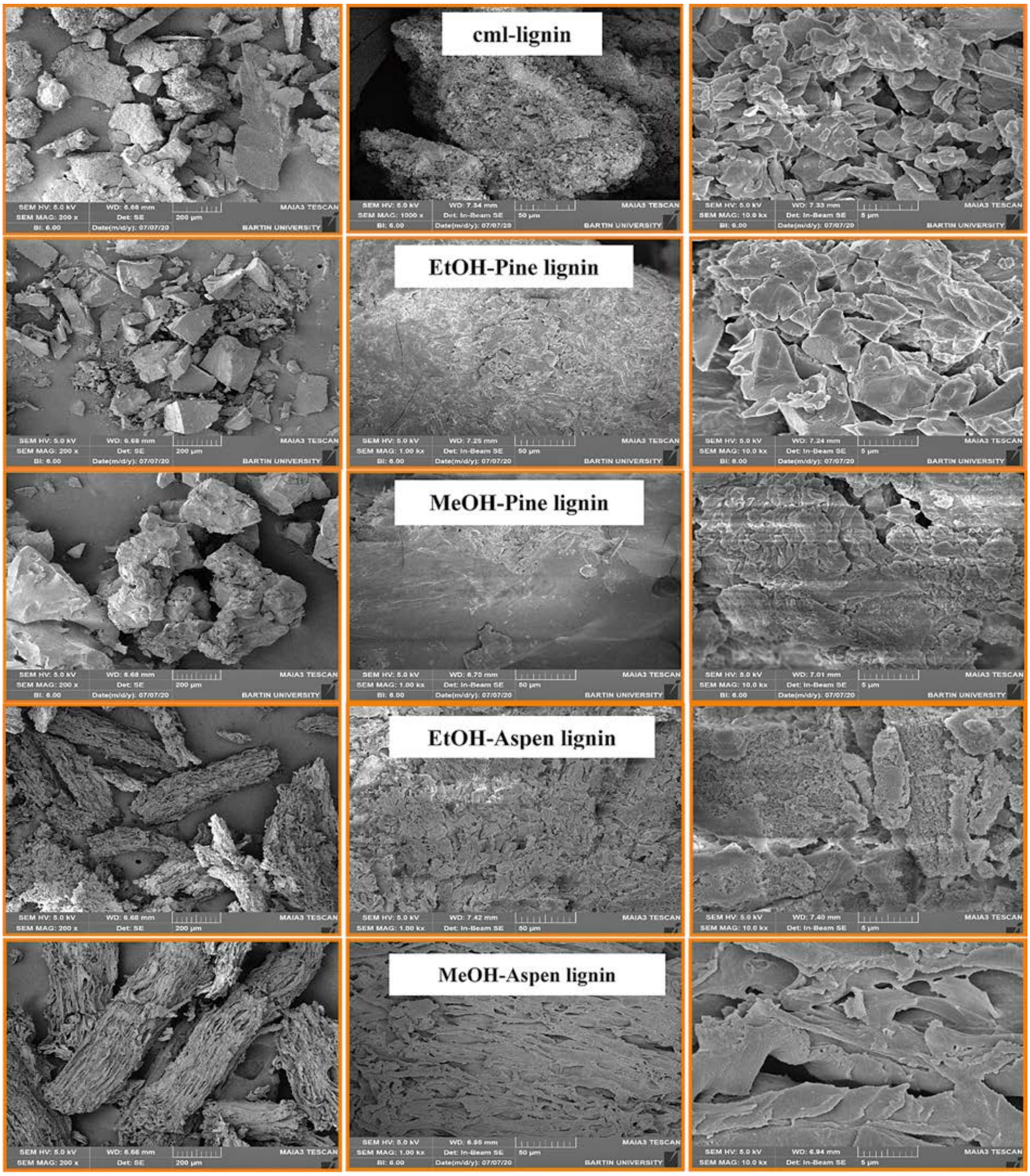

Figure 2 SEM images of lignin particles recovered from black liquor and cml-lignin
Slika 2. SEM slike čestica lignina izdvojenoga iz crnog luga i cml-lignina

Elmer, TA Instruments). The particles were heated from 25 °C to 1000 °C at a heating rate of 20 °C/min and a nitrogen flow of 20 ml/min. After TG analysis, the degradation temperatures of the samples at 10 % weight loss ($T_{10\%}$), 50 % weight loss ($T_{50\%}$), and 75 % weight loss ($T_{75\%}$) were determined, as well as the maximum degradation temperature in the derivative thermogravimetric peaks (DTG_{max}). Finally, the mass loss of the samples was calculated.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

After the recovery process, the morphological analysis of commercial and recovered types of lignin including cml-lignin, lignin recovered with MeOH and EtOH from the black liquor of both pine and aspen wood, was conducted with SEM. The obtained SEM images are given in Figure 2.

The morphological structure and particle size of both MeOH-pine and EtOH-Pine lignin were similar compared to Cml-lignin, and the particle size was generally determined as a circle, sphere, or polygon. However, MeOH-aspen and EtOH-aspen lignin had layered structures and it was determined that they were similar to a long and wide bar. As a result, it can be said that the structure of lignin depends on the wood type, and that the particle sizes of lignin obtained with EtOH and MeOH alcohols were generally similar.

In a study, Haz *et al.* (2015) investigated the size distribution of lignin precipitated with sulphuric acid and the obtained results showed that lignin has a bimodal distribution, and contains nano- and micro-sized particles. The average diameter of the particles was found to range from 353 nm to 5.5 µm. In another study, Hermiati *et al.* (2017) studied the morphological structure of lignin obtained from Kraft pulping black liquor with 2 different hydrochloric acid solutions/ethanol and SEM images showed that the surface morphology of lignin was generally similar and lignin surface was determined to have some impurity and pores. Ibrahim *et al*. (2011) found similar results.

The chemical structure of lignin was investigated with FT-IR and the obtained spectra are given in Figure 3.

The FT-IR spectra of the lignin types obtained in two different ways from the black liquor of pine and aspen chips are given in Table 2. The spectra of the recovered lignin appear to be generally similar to cml-lignin. However, some spectra shifted to lower or higher wavenumbers. Fifteen peaks of commercial lignin and other lignin types were clearly detected as given in Figure 3 and Table 2. The FT-IR spectra revealed that the recovered lignin types had similar functional groups but some differences in intensities of the peaks were observed. It has also been stated that the FT-IR spectra of the four lignin fractions from the black liquor appear to be rather similar to typical lignins, indicating that the "core" of the lignin does not change dramatically and that the recovered lignin isolates had similar functional groups (Sun *et al*., 1999; Hermiati *et al.,* 2016; Sameni *et al.,* 2016; Santos *et al.,* 2015). As a result, it can be said that the recovered lignin types were similar to the cml-lignin

XRD was used for the structural analysis of the recovered lignin and the obtained results are given in Figure 4.

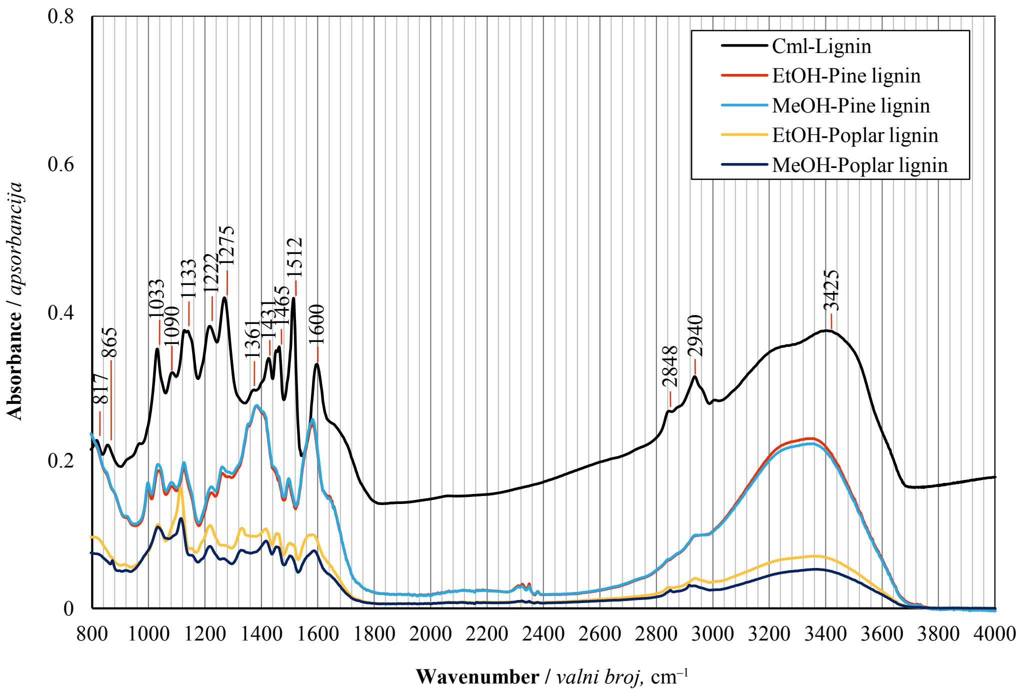

Figure 3 FT-IR spectra of lignin particles recovered from black liquor
Slika 3. FT-IR spektar čestica lignina izdvojenoga iz crnog luga

Table 2 FT-IR spectra of different lignin samples (Sun *et al*., 1999; Zhao *et al*., 2009; Hermiati *et al*., 2017; Hidayati *et al*., 2018)
Tablica 2. FT-IR spektar različitih uzoraka lignina (Sun *et al*., 1999.; Zhao *et al*., 2009.; Hermiati *et al*., 2017.; Hidayati *et al*., 2018.)

cml-L, cm^{-1}	Pine / *Borovina*, cm^{-1}		Aspen / *Drvo jasike*, cm^{-1}		Band position / *Pozicija vrpce*, cm^{-1}	Assignments / *Vrsta grupe i vibracije*
	EtOH	MeOH	EtOH	MeOH		
3425	3400		2950	2940	3450-3400	O-H stretching (phenolic OH and aliphatic OH)
2940	2933	2928	2830	2833	2940-2820	C-H stretching (CH_3 and CH_2)
2848	-		2835	2840	2850-2840	C-H stretching (OCH_3)
					1715-1710	C=O stretching (unconjugated ketone, carbonyl and ester groups)
	1655	1655			1675-1660	The C = O range is unconjugated with an aromatic ring
1600	1595	1595	1589	1593	1605-1595	C-C stretching (aromatic ring)
1512	1505	1502	1508	1510	1515-1505	C-C stretching (aromatic ring)
1465	1450	1456	1464	1462	1470-1461	C-H deformation (asymmetric in $–CH_3$ and $–CH_2$)
1431			1421	1422	1430-1422	C-C stretching (aromatic ring) with C-H in-plane deformation
1361	1375	1368			1370-1365	In-plane deformation vibration of phenolic OH
	1335	1333	1335	1338	1330-1325	C-O stretching (syringil)
					1328 and 1215	C_{aryl}-O vibration in syringyl derivatives
1275	1273	1271	1280	1278	1270-1275	Vibration of guaiacyl rings
1222	1225	1227	1222	1224	1220	C-O(H) + C-O(Ar) (phenolic OH and ether in syringil and guaiacyl)
			1116	1118	1115	Ar-CH in-plane deformation (syringil)
1133	1132	1130			1123 and 1028	Aromatic C-H inplane deformations in syringyl-type and guaiacyl-type lignin
1090	1095	1095	1035	1040	1085-1030	C-O(H) and C-O(C) (first order aliphatic OH and ether)
1033	1035	1035			1030	C-O of syringyl and guaiacyl ring, C-H bond in guaiacyl ring
930	931	931	920	922	915	C-H out of plane (aromatic ring)
865, 825				875	838-875	aromatic C-H out-of-plane bending

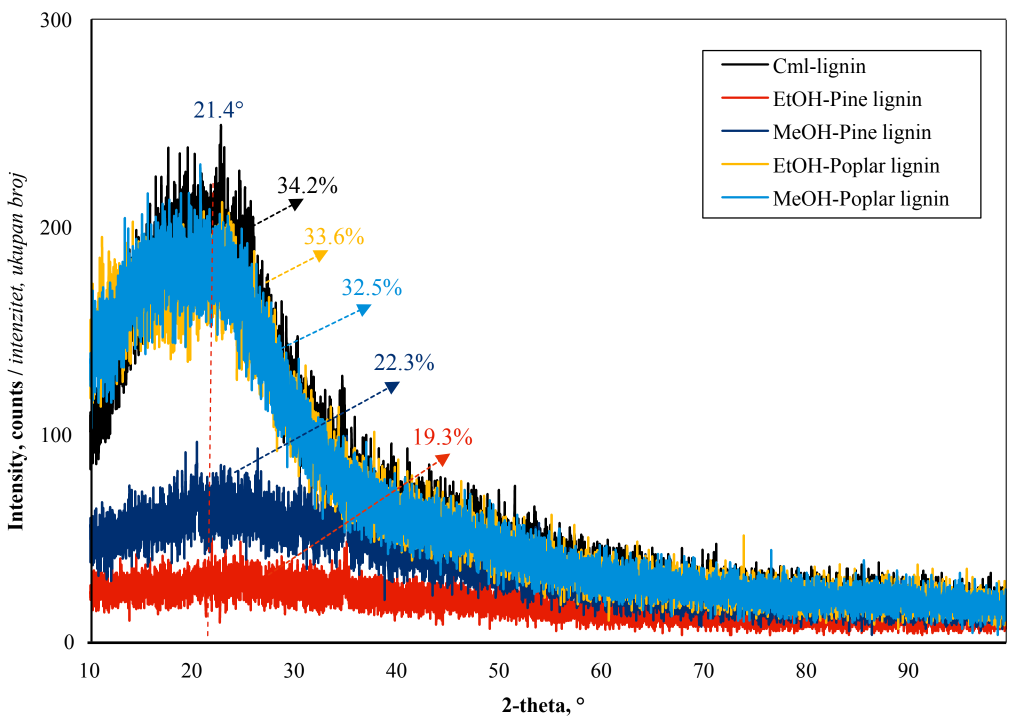

Figure 4 XRD pattern of lignin particles recovered from black liquor
Slika 4. XRD uzorak čestica lignina izdvojenoga iz crnog luga

According to Figure 4, the peak point for the recovered lignin and control lignin was detected at 21.4°. While the peak for the EtOH-aspen and MeOH-aspen has high intensity, the intensity of the EtOH-pine and MeOH-pine was detected to be lower. In CI, the highest and lowest CI for the samples was calculated as 34.2 % and 19.3 % for control lignin and for EtOH-pine, respectively. As a result, the lignin recovered from pine was found to have a similar structure as the control lignin and it can be said that the lignin structure changed according to the wood type, while the alcohol type used during the lignin recovery process had no significant effect on the structural properties of lignin.

The thermal analysis of the recovered lignin was conducted from 25 °C to 1000 °C. The obtained TG curves are given in Figure 5 - 7, and Table 3 shows the summary of TG curves of the recovered lignin and cml-lignin. The thermal decomposition of the recovered lignin and the cml-lignin consists of degradation peaks at different areas occurring between 75 °C and 900 °C (Figure 5). The DTG curves showed many decomposition peaks of lignin at the same temperatures (Figure 6) due to hydrolysis, decomposition (at temperatures between 200 °C and 500 °C) and oxidative chain scission (at temperatures above 600 °C) of lignin as shown by the DTG curves. According to Figure 5 and 6, it can be said that pine lignin exhibited a different behavior than aspen lignin.

The thermal stability of the recovered lignin was found to be generally similar compared to cml-lignin as shown in Table 3. $T_{10\%}$ of the cml-lignin and lignin from aspen were similar to each other. However, $T_{10\%}$ of lignin

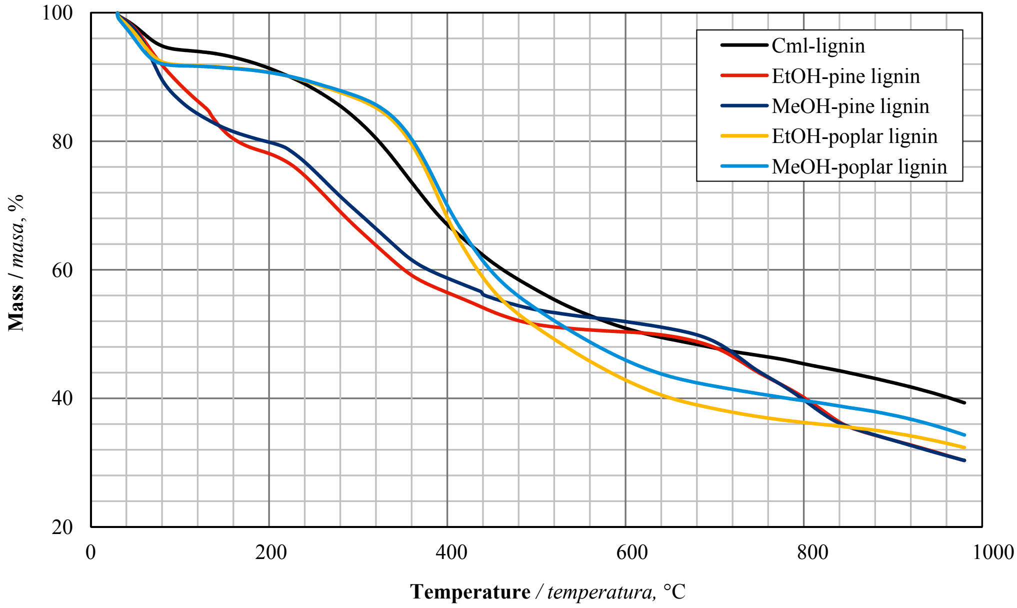

Figure 5 TGA curves of lignin particles recovered from black liquor
Slika 5. TGA krivulje čestica lignina izdvojenoga iz crnog luga

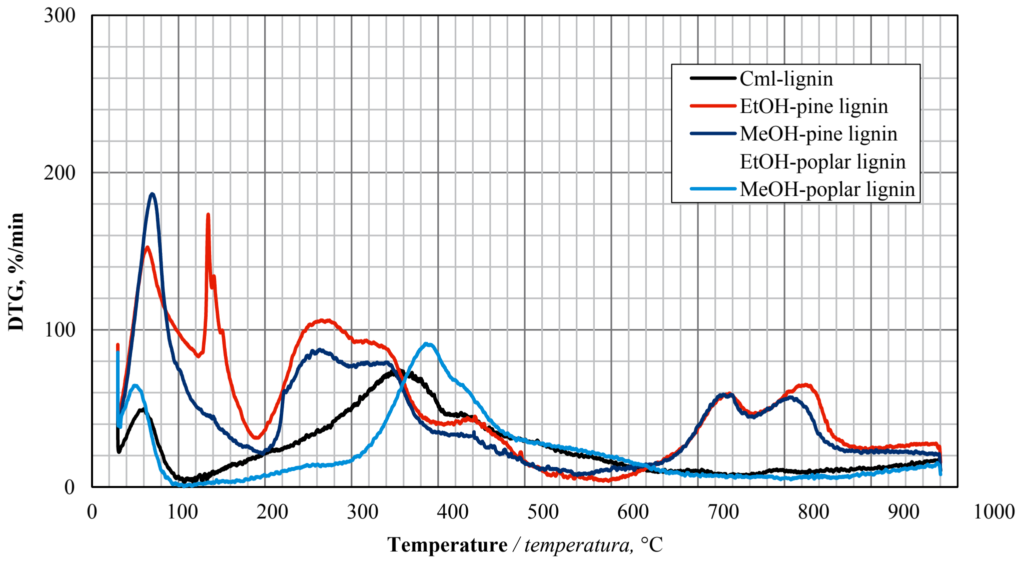

Figure 6 DTG curves of lignin particles recovered from black liquor
Slika 6. DTG krivulje čestica lignina izdvojenoga iz crnog luga

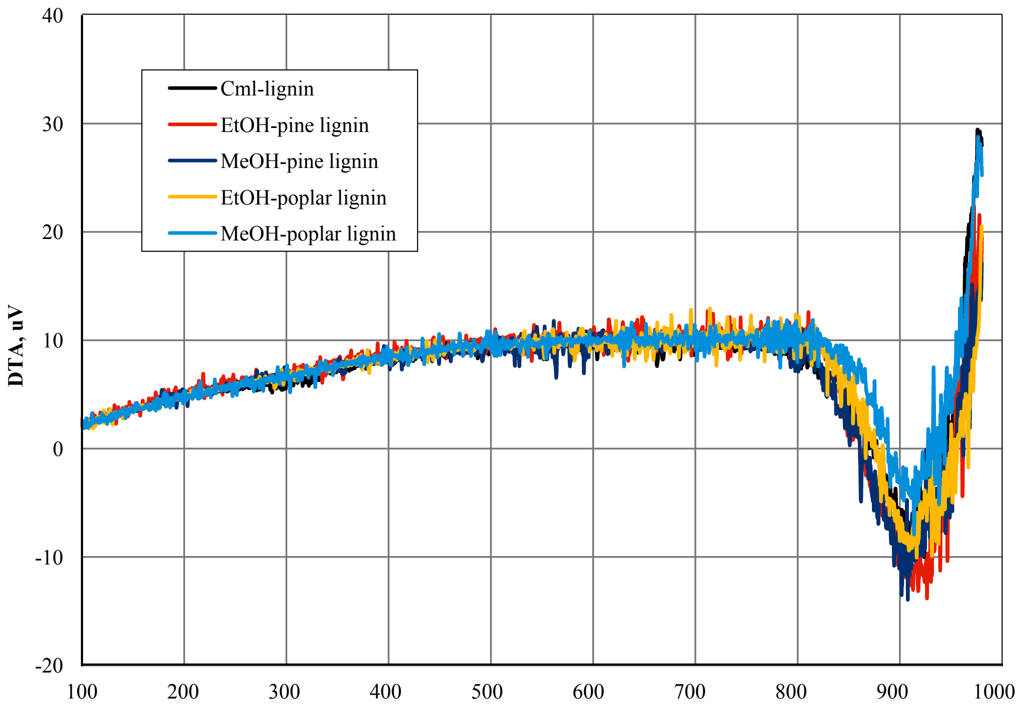

Figure 7 DTA curves of lignin particles recovered from black liquor
Slika 7. DTA krivulje čestica lignina izdvojenoga iz crnog luga

Table 3 Summary of TGA/DTG curves of lignin particles recovered from black liquor
Tablica 3. Sažetak za TGA/DTG krivulje čestica lignina izdvojenoga iz crnog luga

Samples / *Uzorci*	$T_{10\%}$, °C	$T_{50\%}$,°C	DTG_{max}, °C	T_d, °C	**Weight loss, %** *Gubitak mase, %*
cml-lignin	223.7	623.7	354.5	923.7	60.7
EtOH-pine lignin	91.7	632	134.4	922.5	69.7
MeOH-pine lignin	78.1	676.2	59.5	907.7	69.7
EtOH-aspen lignin	224.9	511.1	388.9	916.4	67.6
MeOH-aspen lignin	226.6	544.3	385.4	913.8	65.7

from pine was found very low due to possible impurities. $T_{50\%}$ of the recovered lignin was found to change from 511.1 °C to 676.2 °C. The highest and lowest weight loss was calculated as 69.7 % and 60.7 % for lignin from pine and for cml-lignin, respectively. DTG_{max} of all the recovered lignin was found to be lower than that of cml-lignin and the highest and lowest DTG_{max} was found as 388.9 °C and 59.5 °C for EtOH-aspen lignin and for MeOH-pine lignin, respectively. Td of lignin was determined to range from 907.7°C (MeOH-pine lignin) to 923.7 °C (cml-lignin). As a result, it can be concluded that the recovered lignin generally has similar thermal stability as cml-lignin.

4 CONCLUSIONS
4. ZAKLJUČAK

In this study, lignin from black liquors obtained from Scots pine (*Pinus sylvestris* L.) and European aspen (*Populus tremula* L.) wood cooked by the Kraft method was recovered with methyl and ethyl alcohols, and the material properties of the recovered lignin were analyzed by SEM, FT-IR, XRD, and TGA. According to the obtained results, SEM images showed that the particle sizes of lignin obtained with ethyl and methyl alcohols were generally similar to each other. FTIR showed that the recovered lignin types had similar functional groups, while some differences were observed in intensities of the peaks. XRD results revealed that the lignin recovered from pine was found to have a similar structure as the control lignin and that the alcohol type used during the lignin recovery process had no significant effect on the structural properties of lignin.

TGA results show that the recovered lignin generally has thermal stability comparable to cml-lignin. The results indicated that the lignin structure change according to the wood type but the structure of lignin is not significantly changed by the recovery process described. The results revealed that the recovered lignin was similar to commercial lignin. Based on the obtained results, it can be concluded that lignin can be recovered from the liquor leftover from pulping and/or paper production by the method described and that it can be used in industry.

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Effects of Exposure Time and Temperature on Screw Driving Torques in Heat-Treated Anatolian Black Pine and Sessile Oak Wood

Učinci vremena izlaganja i temperature na zakretne momente uvrtanja vijaka u toplinski obrađeno drvo anadolskoga crnog bora i hrasta kitnjaka

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ABSTRACT • *This study aimed to investigate the characteristics of screw driving torques in heat-treated Anatolian black pine and sessile oak wood. The wood samples were subjected to heat under atmospheric pressure at three different temperatures (130, 180, and 230 °C) and two different exposure time levels (2 and 8 h). Screw driving torques of seating and stripping torque (SET and STT) was performed on all samples. The process of screw driving had two main torques, one of which was the seating torque defined as the torque required to clamp parts and the other one was the stripping torque defined as the maximum torque right before the screw strips in the material and the torque drops suddenly because of the formed screw threads being stripped in wood material. Results show that, in both wood species, the SET and STT values decreased due to the increase in heat treatment temperature and exposure time compared to the control groups.*

KEYWORDS: *screw; pilot hole; seating torque; heat-treated wood*

SAŽETAK • *Cilj ovog istraživanja bilo je ispitivanje svojstava zakretnih momenata zavrtanja vijaka u toplinski obrađeno drvo anadolskoga crnog bora i hrasta kitnjaka. Uzorci drva izloženi su toplini u atmosferskom tlaku pri trima različitim temperaturama (130, 180 i 230 °C) i tijekom dva različita vremena izlaganja (2 i 8 h). Na svim su uzorcima određeni zakretni momenti uvrtanja i odvrtanja vijaka (SET i STT). Riječ je o dvama glavnim zakretnim momentima u procesu spajanja vijcima, od kojih je jedan zakretni moment zatezanja, a drugi je zakretni moment odvrtanja, definiran kao maksimalni zakretni moment neposredno prije nego što se vijak izvuče iz materijala i*

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moment se naglo smanji zbog guljenja navoja formiranih u drvnome materijalu. Rezultati pokazuju da se vrijednosti SET i STT za obje vrste drva smanjuju s povećanjem temperature toplinske obrade i produljenjem vremena izlaganja u usporedbi s kontrolnim skupinama.

KLJUČNE RIJEČI: *vijak; pilot-rupa; zakretni moment zatezanja; toplinski obrađeno drvo*

1 INTRODUCTION
1. UVOD

Thermal modification (heat treatment) is one of the environmentally friendly methods used to improve the properties of wood materials. The heat treatment of wood is defined as the application of heat to the wood to bring about the desired improvement in the performance of the material without using any type of chemicals. The method has shown that, by exposing the wood material to temperatures between 170 °C and 220 °C in an oxygen-free environment, it is possible to modify wood components that are sensitive to moisture absorption and biodegradation. After thermal modification, if it does not directly contact the ground in use, dimensional stability and durability of the wood material increase substantially. Heat treatment serves to improve the natural quality properties of the wood, such as dimensional stability and resistance to bio-corrosion and resistance to outdoor weather conditions, biological resistance against fungi and insects, decorative color variation, and equip the wood material with new properties (Hill, 2006; Gündüz *et al*., 2007; Korkut and Kocaefe, 2009; Dagbro *et al*., 2010; Karamanoğlu and Akyıldız, 2013; Demirel and Temiz, 2015; Dagbro, 2016; Perçin *et al*., 2017; Karamanoğlu, 2020; Holy *et al*., 2020). Increasing environmental pressure has appeared in recent years in many countries, leading to important changes in the field of wood preservation. Heat treatment of wood is an eco-friendly method to modify wood without the use of any toxic chemicals (Kol, 2010; Todorović *et al*., 2020). Heat-treated timber is used in the construction of building cladding, interior paneling, parquet, and plank flooring, park and garden furniture, garden fencing, children's playground equipment, window and window shutters, interior and exterior doors, sauna and sauna elements, interior furniture, and musical instruments. Heat-treated timber surfaces naturally tend to age by the effect of sunlight, rain, and wind. This condition does not affect the durability of the heat-treated timber but the wood surface turns grey after a certain period of time (Korkut and Kocaefe, 2009).

The rigidity and durability of the furniture and other wooden furniture accessories depended on the type and thickness of the wood, as well as on how these pieces were joined together (Aytekin, 2008; Perçin *et al*., 2017). To connect wood and wood-based composites to any wooden material or any other materials such as metal or plastics, fasteners such as nails, bolts, dowels, screws, and nail plates are used (Tor *et al*., 2016; Akrami and Laleicke, 2017; Yorur *et al*., 2017; Brandner, 2019). The screws are commonly used as joint components of the wood construction in engineered wood structures, furniture construction for the attachment of corner blocks to rails in chairs and tables, fastening tops to tables, cabinets, and bases, attachment of shelves to end members frames, and trims to cabinets, and installing hardware and since each wood species has its properties, they also have different direct screw withdrawal (DSW) resistance (Perçin *et al*., 2017; Uysal and Haviarova, 2019; Yorur *et al*., 2020).

There are some studies about the factors affecting DSW resistance in the literature. Perçin *et al*. (2017) reported that screw-withdrawal resistance in heat-treated wood decreased with increasing temperature in Hornbeam (*Carpinus betulu*s L.), black pine (*Pinus nigra* Arnold), and Uludağ fir (*Abies bornmuellerinana* Mattf.) wood species. Kariz *et al*. (2013) reported that the heat treatment reduces the withdrawal capacity of screws in Spruce (*Picea abies* Karst.) wood lamellas. The withdrawal capacity of screws from spruce wood that was heat-treated at temperatures of 210 °C and 230 °C decreased significantly compared to the control wood. Gašparík *et al*. (2015) reported that a significant decreasing effect of thermal modification on screw direct withdrawal load resistance was observed in European spruce trees (*Picea abies* L.).

In addition to these studies, a recent study on driving torques in wood and wood-based composites defined the driving torques as the torque required to drive the screw into a pilot hole. In the study, maximum drive torque (MDT) was defined as the torque at the first turning point on the torque curve where the clamping starts. The term of seating torque (SET) was the torque necessary to clamp parts. Stripping torque (STT) was the torque sufficient to cause the screw to fail in shear. Destruction torque is the torque that causes failure of the screw fastening system, and the term is more general than STT, covering other modes of failure, such as screw torsional failure and material splitting problem (Tor *et al*., 2015).

Therefore, the specific objectives of this study on heat-treated wood were to 1) determine seating and stripping torques in the heat-treated Anatolian black pine and sessile oak wood; 2) investigate the effects of heat treatment and exposure time on the screw driving torques.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Wood material
2.1. Drvo

The Anatolian black pine (*Pinus nigra* J.F. Arnold subsp. *nigra* var. *caramenica* (Loudon) Rehder) and sessile oak (*Quercus petraea* Lieb.) were randomly selected and 3 stems at 5 meters, with a breast height diameter (DBH) of 30 – 40 cm, were obtained from forestlands in Kastamonu, Turkey. The lumber was cut from the logs in the sawmill, in parallel to the grain direction according to the Turkish standard, TS 4176. Experimental materials were selected randomly from mixed core and sapwood, with smooth fibers, no knots, no cracks, no difference in color and density, and annual rings perpendicular to the surfaces according to ASTM D7787 / D7787M (2013). Afterward, the lumbers were air-dried until they reached approximately 12 % MC. Then, the lumber was planed and cut into small clear specimens for heat treatment in the dimensions of 50 mm × 50 mm × 10 mm.

2.2 Heat treatment
2.2. Toplinska obrada

Heat treatment was applied in a small heating unit, temperature-controlled to ±1 °C. The temperature of the oven was increased to the temperature at which the actual heat treatment occurs. Heating time was taken as 1 hour in all groups. Then, the samples at ambient temperature were placed in the oven and heat treated at 3 different temperatures (130, 180, and 230 °C) and for 2 different durations (2 and 8h) under atmospheric pressure and in the presence of an inert (nitrogen) environment (Figure 1). After heat treatment, treated and untreated samples were kept in the conditioning room with a temperature of (20 ±2) °C and relative humidity of (65±5) % until they reached a constant rate (TS 642 ISO 554, 1997).

2.3 Measurement of screw driving torques
2.3. Mjerenje zakretnog momenta uvrtanja

All control and heat-treated test samples were cut to 50 mm in width × 50 mm in length × 10 mm in thickness and conditioned at (20 ±2) °C and (65 ± 5) % relative humidity (RH) (ASTM D 618-13, 2013). Before driving a screw into the samples, pilot holes of 3.2 mm were drilled through the thickness of the samples at the center of each sample face. The torque measurements were carried out by the Kraftform torque screwdriver set that included two different screwdrivers based on the torque range (Figure 2). The torque of the first screwdriver ranged from 0.3 to 1.2 N·m and the second one ranged from 1.2 to 3.0 N·m. The measurement accuracy was ± 6 %, which complies with the requirements of ISO 6789-2 (2017). A 10 mm metal plate was used to be consistent with the screw penetration depths in the testing block. The test setup for evaluating SET and STT is shown in Figure 2.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Comparisons of mean screw driving torques
3.1. Usporedbe srednjih zakretnih momenata uvrtanja i odvrtanja vijaka

Table 1 summarizes mean SET and STT values in heat-treated pine and oak wood. In general, the mean

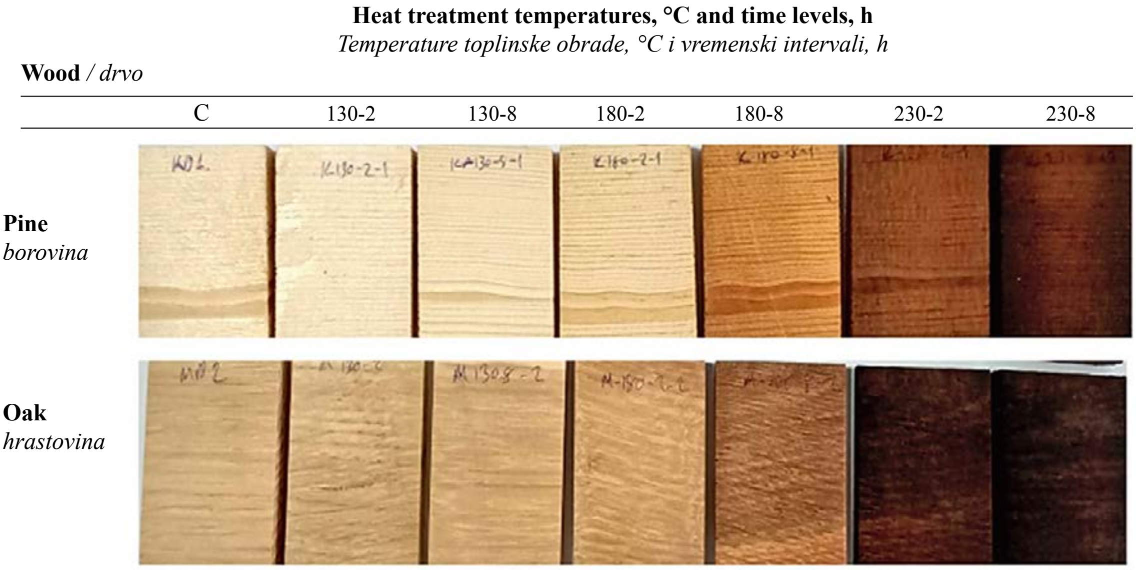

Figure 1 Control samples of pine and oak (C) and heat treated samples
Slika 1. Kontrolni uzorci borovine i hrastovine (C) te toplinski obrađeni uzorci

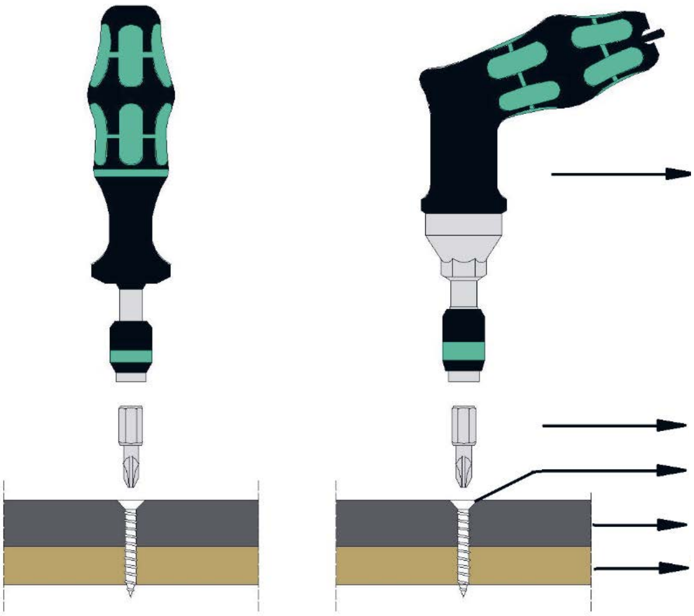

Figure 2 Test setup for evaluating SET and STT of driving screw
Slika 2. Postavi ispitivanja za određivanje SET-a i STT-a pri uvrtanju vijka

SETs ranged from 0.306 to 0.375 N·m and the mean STT ranged from 0.481 to 1.156 N·m for heat-treated pine samples, while the mean SETs ranged from 0.719 to 0.881 N·m and the mean STT ranged from 1.513 to 2.860 N·m as shown in Figure 3.

The ratios of STT/SET for each combination were calculated as shown in Table 1. In general, the ratios decreased when the heat treatment temperature was increased from 130 to 210 °C for both wood species. In a study about screw driving torques obtained in plywood face, the ratios of STT/SET ranged from 2 to 7 with seven layers and 4 to 7 with nine layers (Tor *et al.*, 2020). This ratio is important to keep the torque applied under control when screw driving. Robert (2010) suggested that the ratio should be greater than 3 for high-volume production with power tools. Therefore, the operators should be more careful when driving screws into the pine and oak wood with and without the heat treatment because of the sensitivity level between the SETs and STTs.

A three-factor ANOVA was performed using the GLM procedure for each data set of SET and STT separately at the 5 % significance level to analyze the interaction and the main effects in pine and oak wood. The ANOVA results indicated that three-way interaction was statistically significant with the p-value < 0.0001.

Table 1 Summary of mean SET and STT values and their ratios
Tablica 1. Srednje vrijednosti SET-a i STT-a i njihovih omjera

Wood species *Vrsta drva*	**Heat treatment temperature, °C** *Temperatura toplinske obrade*, °C	**Exposure time, h** *Vrijeme izlaganja*, h	**Mean** *Srednja vrijednost*		**Ratio STT/SET** *Omjer* STT/SET
			SET	**STT**	
Pine *borovina*	Control / *kontrolni uzorak*	-	0.444 (7)	1.213 (13)	2.7
	130	2	0.375 (12)	1.156 (16)	3.1
		8	0.375 (7)	1.056 (13)	2.8
	180	2	0.369 (7)	1.069 (11)	2.9
		8	0.325 (8)	0.894 (9)	2.8
	210	2	0.306 (5)	0.769 (13)	2.5
		8	0.319 (8)	0.481 (5)	1.5
Oak *hrastovina*	Control / *kontrolni uzorak*	-	0.906 (4)	2.863 (5)	3.2
	130	2	0.869 (5)	2.869 (6)	3.3
		8	0.881 (4)	2.775 (5)	3.1
	180	2	0.856 (4)	3.106 (15)	3.6
		8	0.806 (2)	2.813 (5)	3.5
	210	2	0.725 (7)	1.863 (11)	2.6
		8	0.719 (8)	1.563 (11)	2.2

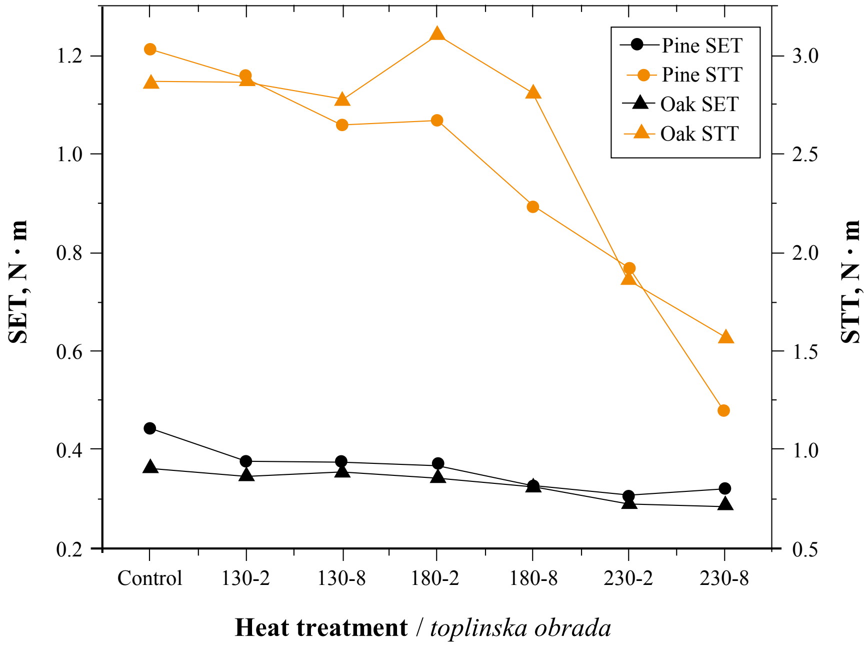

Figure 3 Trend of mean SET and STT for both wood species
Slika 3. Trend srednjh vrijednosti SET-a i STT-a za obje vrste drva

3.2 Treatment temperature effects
3.2. Utjecaj toplinske obrade

According to Table 2, in general, mean SET and STT values decreased due to the increasing heat treatment temperature in both pine and oak samples. The mean SET values in the temperature level of heat treatment were the highest in control samples of both pine and oak, while the mean STT values were the highest in control samples of pine while the values of oak samples did not indicate a proportional decreasing trend.

3.3 Exposure time effects
3.3. Utjecaj vremena izlaganja

Table 3 demonstrates that the mean SET and STT of the samples heat-treated in the long-term (8 hours) were mostly lower than the corresponding ones in the short-term (2 hours) compared to the control samples. There was no significant difference between the mean SETs in the exposure time for the heat treatment at the temperature of 130 and 230 °C in both wood samples. This can be explained by the structural changes in wood materials such as the chemical structure of lignin, cellulose, and hemicellulose, all of which prevent reabsorbing of water molecules. Correlatively, the mechanical resistance properties of wood materials decrease in the heat treatment of wood samples (Hill, 2006; Korkut and Kocaefe, 2009; Perçin and Ayan, 2012).

3.4 Wood species effects
3.4. Utjecaj vrste drva

Table 4 shows that the mean SET and STT values obtained in oak samples were significantly higher than the corresponding ones in pine samples in all treatment combinations. This may be due to the different anatomical properties of wood materials - oak wood is denser, its cell walls are thicker, its lumen is narrower than that in pine wood (need reference). Studies on wood-based composites such as oriented strandboard (OSB), medium-density fiberboard (MDF), particleboard (PB), and wood-plastic composites (WPC) re-

Table 2 Mean comparison of SET and STT values in terms of temperature within exposure time by wood species
Tablica 2. Usporedba srednjih vrijednosti SET-a i STT-a obiju vrsta drva s obzirom na temperaturu unutar vremena izlaganja

Wood species *Vrsta drva*	**Exposure time, h** *Vrijeme izlaganja,* h	**Treatment temperature** / *Temperatura obrade,* **°C**							
		SET, N·m				**STT, N·m**			
		Control *Kontrolni uzorak*	**130**	**180**	**230**	**Control** *Kontrolni uzorak*	**130**	**180**	**230**
Pine *borovina*	2	0.444 (A)	0.375 (B)	0.369 (B)	0.306 (C)	1.212 (A)	1.156 (A)	1.069 (A)	0.769 (B)
	8		0.375 (B)	0.325 (C)	0.319 (C)		1.056 (BA)	0.894 (B)	0.481 (C)
Oak *hrastovina*	2	0.906 (A)	0.869 (B)	0.856 (B)	0.725 (D)	2.862 (B)	2.869 (B)	3.106 (A)	1.862 (C)
	8		0.881 (BA)	0.806 (B)	0.719 (C)	2.862 (A)	2.775 (A)	2.812 (A)	1.562 (B)
		LSD±0.0374				LSD±0.1955			

Table 3 Mean comparison of SET and STT values in terms of exposure time within set temperature range by wood species
Tablica 3. Usporedba srednjih vrijednosti SET-a i STT-a obiju vrsta drva s obzirom na vrijeme izlaganja unutar temperature obrade

Wood species *Vrsta drva*	**Heat treatment temperature, °C** *Temperatura toplinske obrade, °C*	**Exposure time, h / *Vrijeme izlaganja,* h**					
		SET, N·m			**STT, N·m**		
		Control *Kontrolni uzorak*	**2**	**8**	**Control** *Kontrolni uzorak*	**2**	**8**
Pine *borovina*	130	0.444 (A)	0.375 (B)	0.375 (B)	1.212 (A)	1.156 (A)	1.056 (A)
	180		0.369 (B)	0.325 (C)		1.069 (AB)	0.894 (B)
	230		0.306 (B)	0.319 (B)		0.769 (B)	0.481 (C)
Oak *hrastovina*	130	0.906 (A)	0.869 (A)	0.881 (A)	2.862 (A)	2.869 (A)	2.775 (A)
	180		0.856 (B)	0.806 (C)		3.106 (A)	2.812 (B)
	230		0.725 (B)	0.719 (B)		1.862 (B)	1.562 (C)
			LSD±0.0374			LSD±0.1955	

ported in the literature that the density directly affected the mean SET and STT values (Yu *et al.*, 2015; Tor *et al.*, 2015; Tor, 2019; Kuang, 2016).

4 CONCLUSIONS
4. ZAKLJUČAK

The effects of heat treatment on SET and STT were investigated in two different commonly used wood species. Results revealed that mean SETs ranged from 0.306 to 0.444 N·m for pine samples and from 0.719 to 0.906 N·m for oak wood samples whereas the mean STT ranged from 0.481 to 1.213 N·m for pine wood samples and from 1.513 to 3.106 N·m for oak samples. The ratio of STT/SET ranged from 1.5 to 3.1 N·m for pine samples, and from 2.2 to 3.6 N·m for oak samples. In the study, the highest mean SET and STT values were obtained for oak wood in all cases. In both wood species, the mean SET and STT values decreased because of the increase in the temperature and exposure time compared with the control samples. These results will be interesting for manufacturers of furniture and decoration equipment who use heat-treated wood. With this study, operators will understand the process of screw driving torques and know how much torque they will need to drive screws in the samples that were heat-treated at different exposure times. Therefore, knowing the limitations of screw driving torques will possibly reduce work accidents and material consumption and ease assembly in furniture construction.

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Table 4 Mean comparison of SET and STT values in terms of wood species within set temperature range
Tablica 4. Usporedba srednjih vrijednosti SET-a i STT-a s obzirom na vrstu drva pri jednakoj temperaturi obrade

Treatment temperature, °C *Temperatura toplinske obrade, °C*	**Exposure time, h** *Vrijeme izlaganja*, h	**Wood species** / *Vrsta drva*			
		SET, N·m		**STT, N·m**	
		Pine *borovina*	**Oak** *hrastovina*	**Pine** *borovina*	**Oak** *hrastovina*
Control / *kontrolni uzorak*	-	0.444 (B)	0.906 (A)	1.212 (B)	2.862 (A)
130	2	0.375 (B)	0.869 (A)	1.156 (B)	2.869 (A)
180		0.369 (B)	0.856 (A)	1.069 (B)	3.106 (A)
230		0.306 (B)	0.725 (A)	0.769 (B)	1.862 (A)
130	8	0.375 (B)	0.881 (A)	1.056 (B)	2.775 (A)
180		0.325 (B)	0.806 (A)	0.894 (B)	2.812 (A)
230		0.319 (B)	0.719 (A)	0.481 (B)	1.562 (A)
		LSD±0.0374		LSD±0.1955	

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Characterization of Pulp and Paper Properties Produced from Okra (*Abelmoschus esculentus* Stalks

Karakterizacija pulpe i papira izrađenih od stabljika bamije (*Abelmoschus esculentus*

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ABSTRACT • *In this study, the usability of okra (Abelmoschus esculentus) stalks in pulp and paper production was investigated. Firstly, chemical components and fiber morphological properties were determined to investigate the suitability of okra stalks for pulp and paper production. Holocellulose, cellulose and lignin contents were found as 76.1 %, 46.4 % and 16.0 %, respectively and the fiber length, fiber width and cell wall thickness were measured as 0.64 mm, 16.97 µm and 3.66 µm, respectively. According to these data, it has been determined that okra stalks are suitable for pulp and paper production. Four different cooking experiments were carried out by adding sodium borohydride ($NaBH_4$) in different proportions (0 %, 0.3 %, 0.5 %, 0.7 %) to okra stalks by the alkali sulfite method. The yield, chemical, physical and optical properties of the pulps were determined and optimum properties were obtained from the cooking experiment by adding up to 0.7 % $NaBH_4$ into cooking liquor. With the addition of 0.7 % $NaBH_4$, pulp yield, breaking length, and burst index increased about 7.73 %, 3.84 %, and 11 %, respectively. Consequently, it has been concluded that pulp produced from okra stalks can be used in the paper industry by blending with long or recycled fibers in certain proportions.*

KEYWORDS: *okra stalk; pulp; paper; $NaBH_4$*

SAŽETAK • *U ovom je radu istraživana upotrebljivost stabljika bamije (Abelmoschus esculentus) za proizvodnju celuloze i papira. Radi ispitivanja prikladnosti stabljika bamije za proizvodnju celuloze i papira, najprije su određeni udjeli kemijskih komponenata i morfološka svojstva vlakana. Utvrđeni udjeli holoceluloze, celuloze i lignina iznosili su 76,1 %, 46,4 % i 16,0 %. Duljina vlakana, širina vlakana i debljina stanične stijenke bili su 0,64 mm, 16,97 µm i 3,66 µm. Prema tim podatcima utvrđeno je da je stabljika bamije pogodna za proizvodnju celuloze i papira. Zatim su provedena četiri različita pokusa kuhanja alkalno sulfitnim postupkom, uz varijabilni dodatak natrijeva borhidrida ($NaBH_4$): 0 %; 0,3 %; 0,5 %; 0,7 % s obzirom na količinu stabljika bamije. Određeni su prinos pulpe, njezina kemijska, fizikalna i optička svojstva te su postignuta optimalna svojstva pulpe u procesu kuhanja uz dodatak 0,7 % $NaBH_4$ smjesi kemikalija za kuhanje (bijeli lug). Uz dodatak 0,7 % $NaBH_4$, prinos pulpe, duljina loma i indeks pucanja papira izrađenoga od vlakana bamije povećali su se za oko 7,73 %, 3,84 %, odnosno 11 %. Posljedično, zaključeno je da se pulpa proizvedena od stabljika bamije može upotrebljavati u industriji papira u određenim omjerima pomiješana s dugim ili recikliranim vlaknima.*

KLJUČNE RIJEČI: *stabljika bamije; pulpa; papir; $NaBH_4$*

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1 INTRODUCTION
1. UVOD

The decline in forest areas led the forest products industry to find new raw materials in the second half of the 20th century, particularly with the increase in global population and the increase in demand for forest products due to rapid technological developments (Oner and Aslan, 2002; Tutuş *et al*., 2011; Birinci *et al*., 2020). There are various cellulosic raw materials in the pulp and paper production. The most important of these is the wood. If the insufficiency in raw materials is considered to be a major problem of the present and the future, the focus should be placed on annual plants for the production of pulp and paper (Kaldor, 1992; Comlekcioglu *et al*., 2016; Tutuş *et al.,* 2016a).

Flax, cotton, bamboo and cereal stalks have been widely used in the pulp and paper production as non-wood plants and agricultural residues. Okra (*Abelmoschus esculentus*) stalks might be one of the promising plants for the production of non-woody paper (Jimenez *et al*., 2008; Rodriquez *et al*., 2010). The okra (*Abelmoschus esculentus*) and cotton (*Gossypium hirsutum*) families are Malvaceae. Okra is one of the earliest cultivated crops, recorded by the Egyptians in 1216 AD, but strong evidence has been obtained that okra was grown earlier in Ethiopia and some other reports considered India to be the center of its origin (Lamont, 1999; Singha *et al*., 2014; Tong, 2016). According to the Food and Agriculture Organization of the United Nations (FAO), approximately 9.95 million tons of okra were produced in the world in 2019 (Faostat, 2021). As a result of negotiations with okra farmers, 2 kg of okra stalk is obtained in the production of 1 kg of okra and this amount will have an important place in the wood products industries, especially in the pulp and paper industry where an alternative to wood is sought. Atik has utilized okra stalks to produce pulp and paper using the soda-AQ (anthraquinone) method and reported that good pulp could be produced from okra stalks (Atik, 2002). It has previously been observed that pulps produced from okra stalks are suitable for the production of writing and printing paper and that different types of paper can be produced by blending with other pulps (Omer *et al.,* 2019). Comparing soda-AQ and kraft pulping methods for obtaining okra stalk pulp showed that the kraft process is suitable for okra stalk pulping and that kraft pulp showed better bleachability than soda-AQ pulp (Jahan *et al.,* 2012).

Various studies have been conducted to improve the performance, yield and strength of the pulp by adding different chemicals to the conventional cooking liquor (Akgül and Temiz, 2006; Istek and Özkan, 2008; Gülsoy and Eroglu, 2011; Tutuş *et al*., 2011; Tutuş *et al*., 2015; Tutuş and Çiçekler, 2016; Akgül *et al*., 2018). These studies were generally done to stop or reduce the peeling reaction that occurs during cooking. Anthraquinone and boron compounds are commonly used for this purpose. Many studies have been conducted investigating the effect of sodium borohydride ($NaBH_4$) additive, one of the boron compounds, on the pulp and it has been reported that it has a positive effect on the pulp (Pettersson and Rydholm, 1961; Meller, 1963; Khaustova *et al*., 1971; Akgül and Temiz, 2006; Istek and Özkan, 2008; Tutuş *et al*., 2012). Since $NaBH_4$ is a strong reducer, it prevents yield losses in the cooking environment. During the cooking process, $NaBH_4$ reduces the carbonyl group at the reducing ends of the cellulose chain to a hydroxyl group and prevents the peeling reaction that may occur. This reaction occurs not only in cellulose, but also in hemicelluloses. Therefore, the yield loss caused by the peeling reaction is prevented and the yield of the pulp obtained is increased (Akgül and Temiz, 2006; Istek and Özkan, 2008).

Due to the shortage of raw materials in the paper industry, it has been important in recent years to achieve maximum efficiency and quality with new raw materials in pulp production. This study focused on investigating the usability of okra stalks as an alternative raw material to wood and other non-wood lignocellulosic materials in pulp and paper production.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Material
2.1. Materijali

The okra stalks used in the study were obtained from Kahramanmaraş-Turkey after harvest. The stalks were removed from their shells and impurities and chipped at a size of about 4-6 cm, suitable for pulp production.

2.2 Chemical components and fiber morphology determination
2.2. Određivanje udjela kemijskih komponenata i morfologije vlakana

A sufficient amount of air-dried samples to be used in the chemical analysis was ground in a laboratory type Wiley mill according to the TAPPI T257 standard method and sieved in 40 mesh (425 μm) and 60 mesh (250 μm) vibrating sieves. The part that passed through a 40 mesh sieve and remained on the 60 mesh sieve was taken and put into glass jars with lids and prepared to be used in chemical analysis. Then, the samples were subjected to the following chemical analysis: holocellulose (Wise and Karl, 1962), cellulose (Kürschner and Hoffer, 1969), alpha cellulose (TAPPI T203), lignin (TAPPI T222), ethanol-toluene solubility (ASTM D1107), ash content (TAPPI T211), water (TAPPI T207) and 1 % NaOH (TAPPI T212) solubilities. For each experiment, three repetitions were performed, and mean data were used.

The length, lumen diameter, and width of the fibers included in the structure of the morphological parts of the okra stalks are important in terms of paper production. In the preparation of the slides, the chloride maceration method was used to individualize the fibers. Permanent slides were then prepared from the macerated fibers using a glycerin-gelatin solution. In the measurements of fiber morphology; fiber length, lumen diameter and fiber widths were determined by using Nikon FS1 photo microscope (Kılıç Ak *et al.,* 2021). At least 100 measurements were made for each fiber dimension. Thereupon, the fiber parameters were calculated using a previously described procedure (Comlekcioglu *et al*., 2016) by Eqs. 1-4 given below.

$$Felting\ ratio = \frac{Fiber\ length}{Fiber\ diameter} \quad (1)$$

$$Elasticity\ coefficient\ (\%) = \frac{Lumen\ diameter}{Fiber\ diameter} \cdot 100 \quad (2)$$

$$Rigidity\ coefficient\ (\%) = \frac{Cell\ wall\ thickness}{Fiber\ diameter} \cdot 100 \quad (3)$$

$$Runkel\ ratio = \frac{Cell\ wall\ thickness \cdot 2}{Lumen\ diameter} \quad (4)$$

2.3 Pulp production from okra stalks
2.3. Proizvodnja pulpe od stabljika bamije

Cooking processes were carried out in a laboratory type rotary cylindrical digester with a capacity of 15 liters, electrically heated, resistant to a pressure of 25 bar, capable of 4 revolutions per minute, whose temperature can be controlled by thermostat with an automatic control table. The filling of the raw material and unloading of the pulps were done by hand, and 500 grams oven-dried dry okra stalks chips were used in each cooking. Four cooking experiments were carried out under the cooking conditions listed in Table 1.

The pulps obtained at the end of each cooking were washed on a 200 mesh screen with plenty of tap water until the black liquor was removed. After the chemical substances were removed by washing, it was disintegrated in a laboratory type pulp disintegrator for 10 minutes at a certain concentration, and the uncooked parts were separated by a vibrating screen with a slit opening of 0.15 mm. The kappa numbers and viscosity values of the screened pulps were determined according to TAPPI T236 and TAPPI T230, respectively.

2.4 Paper production and tests
2.4. Izrada papira i ispitivanje njegovih svojstava

The pulp obtained from okra stalks was beaten gradually in a Hollander device up to (35±5) SR° degree. The moisture content of the beaten pulps was determined and the pulp was converted into paper weighting approximately (70±2) g/m^2 in a semi-automatic Regmed RK-21 device. The test papers were conditioned in a conditioning room at (23±1) °C and a relative humidity of (50±2) % for 24 h according to TAPPI T402, after which they were physically and optically tested. The brightness was measured according to ISO 2470-1 as the optical property of the papers. The breaking length and burst index were determined in accordance with the standards TAPPI T494 and TAPPI T403, respectively, as the physical properties. Variance and Duncan tests have also been used to show the influence of $NaBH_4$ on the physical and optical characteristics of the paper.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Chemical components and fiber morphology properties of okra stalks
3.1. Udjeli kemijskih komponenata i morfološka svojstva vlakana od stabljika bamije

The chemical components of okra stalks and some annual plants and types of wood are given for comparison in Table 2 below.

The holocellulose and cellulose contents of okra stalks were determined as 76.1 % and 46.4 %, respectively. These analyzed components have a similar val-

Table 1 Cooking conditions applied to okra stalks
Tablica 1. Parametri kuhanja stabljika bamije

Pulping condition / *Uvjeti kuhanja*	**Unit** / *Jedinica*	**Value** / *Vrijednost*
NaOH charge / *udio NaOH*	%	20
Na_2SO_3 charge / *udio Na_2SO_3*	%	15
$NaBH_4$ charge / *udio $NaBH_4$*	%	0, 0.3, 0.5, 0.7
Cooking temperature / *temperatura kuhanja*	°C	160
Time to maximum temperature / *vrijeme potrebno za postizanje maksimalne temperature*	min	140
Time at maximum temperature / *vrijeme kuhanja na maksimalnoj temperaturi*	min	110
Liquor to raw material ratio / *odnos tekućih komponenata i krute sirovine*	l/kg	5/1

Table 2 Main chemical components of okra stalks, annual plants and types of wood
Tablica 2. Glavne kemijske komponente stabljika bamije, jednogodišnjih biljaka i drva

Annual plants and wood species *Drvenasta vrsta*	**Holocellulose** *Holoceluloza,* %	**Cellulose** *Celuloza,* %	**Alpha cellulose** *Alfa-celuloza,* %	**Lignin** *Lignin,* %	**Ash** *Pepeo,* %	**Ethanol-toluene solubility** *Topljivost u smjesi etanola i toluena,* %	**1% NaOH solubility** *Topljivost u 1 %-tnoj otopini NaOH,* %	**Hot water solubility** *Topljivost u vrućoj vodi* %	**Cold water solubility** *Topljivost u hladnoj vodi* %	**Literature** *Literatura*
Okra stalks (Std. Deviation) *stabljike bamije (stand. devijacija)*	76.1 (0.37)	46.4 (0.16)	37.8 (0.12)	16.0 (0.27)	4.90 (0.12)	6.40 (0.02)	37.1 (0.40)	17.5 (0.03)	17.9 (0.39)	Current study *Ovo istraživanje*
Poppy stalks *stabljike maka*	79.8	40.9	51.7	19.2	4.70	-	30.4	10.4	5.10	Tutuş *et al.,* 2011
Cotton stalks *stabljike pamuka*	75.6	45.5	39.8	18.2	2.50	6.10	30.9	14.3	11.7	Tutuş *et al.,* 2010
Wheat stalks *slama žitarica*	77.6	52.5	40.2	17.3	7.10	5.50	42.1	13.0	9.00	Tutuş and Çiçekler, 2016
Softwood *meko drvo*	63-74	55-61	-	25-32	0.2-0.5	1-5.8	8-10	1-5	0.5-4	Kirci, 2006
Hardwood *tvrdo drvo*	72-82	38-55	-	18-26	0.2-0.7	1-6.2	12-25	1-8	0.2-4	Kirci, 2006

ue to hardwood and annual plants. Alpha cellulose content of okra stalks was slightly lower than that of other species. Lignin, which is one of the important components in terms of ease and economy of pulp production, was found to be lower than that of other lignocellulosic species. The ash content (4.9 %) was determined at similar rates with annual plants listed in Table 2. Although the high NaOH solubility rate is due to low molecular weight carbohydrates and other soluble alkaline substances, the high rate of fungal rot in okra stalks also affects it (Tutuş *et al.,* 2016b). When the chemical components of okra stalks are examined, it is understood that they are generally in harmony with the literature as seen in Table 2. The components that are important in pulp and paper production are holocellulose and cellulose (Smook, 1992; Tutuş and Çiçekler, 2016; Tutuş *et al.,* 2016b) in terms of yield, and these values are at the desired level. In addition, lower lignin contents make the fiber stronger and harder to break (Tran, 2006; Daud *et al*., 2014; Tutuş *et al*., 2015)

The fiber length, fiber width, lumen diameter, and cell wall thickness of okra stalks were measured as 0.64 mm, 16.97 µm, 9.64 µm, and 3.66 µm, respectively. The fiber parameters of okra stalks are presented in Table 3.

Felting ratio is calculated as the ratio of fiber length to fiber width. If this ratio is higher than 70, the tear, tensile, and double-folding strengths of the paper

Table 3 Fiber parameters of okra stalks in comparison with wood and other non-wood lignocellulosic materials
Tablica 3. Svojstva vlakana od stabljika bamije u usporedbi s vlakancima drva i ostalih nedrvnih lignoceluloznih materijala

Fiber dimensions *Dimenzije vlakana*	**Okra stalks** *Stabljike bamije*	**Wheat stalks** *Slama žitarica*	**Cotton stalks** *Stabljike pamuka*	**Rice stalks** *Rižina slama*	**Hardwoods** *Tvrdo drvo*	**Softwoods** Meko drvo
Felting ratio /*vitkost*	37.7	62	33	58	45-65	60-80
Elasticity /*elastičnost*	56.8	27	63	71	35-65	70-80
Rigidity /*krutost*	21.6	36	18	15	15-35	15-20
Runkel ratio / *Runkelov omjer*	0.76	2.7	0.6	0.4	0.5-2	0.3-0.5
Literature / *Literatura*	Current study *ovo istraživanje*	Tutuş and Çiçekler, 2016	Tutuş *et al.*, 2010	Tutuş *et al.*, 2004	Kirci, 2006	Kirci, 2006

will be high (Bektas *et al*., 1999; Kirci, 2006; Istek *et al*., 2009). The felting ratio of okra stalks fibers was calculated as 37.7 on average. This ratio shows that okra stalk fibers are more similar to hardwood fibers than softwood. Fibers with a coefficient of elasticity ratio between 50 and 75 are partly thick-walled. It is stated that these values are obtained from lignocellulosic materials with a density of 0.5–0.7 g/cm^3 (Bektas *et al.*, 1999). Since these fibers are partially crushed during papermaking, quality papers can be produced (Bektas *et al*., 1999; Ozdemir *et al*., 2015). The rigidity coefficient is between 15-35 for softwoods and hardwoods. When this value is high, the strength properties of the paper such as tensile, burst, and tear strengths are adversely affected (Bektas *et al*., 1999). In this study, the rigidity coefficient of okra stalk fibers was calculated as 21.6, which is within the acceptable limit for paper production. The Runkel ratio of okra stalk fibers is 0.76, and fibers with a Runkel index less than 1 are thin-walled and very suitable for paper production (Bektas *et al*., 1999; Ozdemir *et al.,* 2015). Based on the results of the analyzed chemical components and fiber morphological properties of okra stalks, it was concluded that okra stalk is suitable for pulp and paper production.

3.2 Chemical, physical and optical properties of okra stalk pulps
3.2. Kemijska, fizička i optička svojstva pulpe od stabljika bamije

The pulp yields and some chemical properties of the okra stalk pulps obtained by different cooking experiments are given in Table 4. The total yield of pulp produced from okra stalks was around 32-35 %. It was found to be lower than that of wheat stalk (50-55 %), cotton stalk (37-44 %) and rice stalk (41-56 %) pulps, which we have published in our previous works (Tutuş and Çiçekler, 2016; Tutuş *et al.,* 2004; Tutuş *et al.,* 2010). It is thought that the reason why the yields of pulp produced from okra stalks are lower than those of other stalks is due to the excess of substances dissolved in cold water and hot water, the lower cellulose content than other stalks, and the higher amount of material soluble in 1 % NaOH.

Kappa numbers of the okra stalk pulps show similar characteristics as stated in previous studies (Jahan *et al*., 2012), and similar to other species such as wheat, cotton, and rice stalks (Tutuş and Çiçekler, 2016; Tutuş *et al.,* 2004; Tutuş *et al.,* 2010). A low kappa number indicates that the lignin content of the pulp is low and is easier to bleach (Correia *et al.,* 2018; Przybysz Buzała *et al*., 2019; Birinci *et al*., 2020). Since okra stalk pulps have low kappa numbers (ranging from 25 to 33), they are easy to bleach, but considering the viscosity and DP values presented in Table 4, the strength properties of the papers produced after pulp bleaching will be low, since the chemicals used during bleaching will also damage carbohydrates.

It was observed that there was a certain increase in both pulp yield and viscosity values as a result of the addition of $NaBH_4$ to the cooking liquor. $NaBH_4$ has an effect on the yield as it has the capability of stopping the peeling reactions occurring in the cellulose chains. Although $NaBH_4$ decreased the kappa numbers at certain rates, it also increased the viscosity values. In recent studies, it has been observed that the kappa numbers of the pulps obtained by adding the $NaBH_4$ chemical to the cooking liquor in certain proportions decrease (Copur and Tozluoglu, 2008; Gülsoy and Eroglu, 2011; Istek and Gonteki, 2009).

Some physical and optical properties of the papers produced from okra pulps are given in Table 5.

The tensile index, burst index and brightness of the okra pulps were found as 60.7 (N·m/g), 3.00 kPa·m^2/g and 27.5 ISO%. The physical and optical properties were determined similarly as in previous studies dealing with okra stalk pulp, as indicated in Ta-

Table 4 Pulp yields and some chemical properties of okra stalk pulps
Tablica 4. Prinos vlakana i neka kemijska svojstva pulpe od stabljika bamije

Cooking No *Kuhanje*	**$NaBH_4$ charge** *Udio $NaBH_4$,* %	**Screened yield** *Prinos, nakon prosijavanja,* %	**Screen reject** *Odbačeno na situ,* %	**Total yield** *Ukupan prinos,* %	**Kappa No** *Kappa broj*	**Viscosity** *Viskozitet,* cm^3/g	**DP**
1	0	31.81^c	0.56^b	32.37^c	33^c	611^c	871^c
2	0.3	32.22^b	0.59^c	32.81^c	32^c	690^a	997^a
3	0.5	34.24^{ab}	0.54^{ab}	34.78^b	29^b	681^{ab}	983^b
4	0.7	34.43^a	0.46^a	34.89^a	25^a	673^b	970^b
Sig.	-	.002	.003	.015	.010	.012	.002
Jahan *et al.,* 2012	Soda-AQ	30.4-32.2	5.5-7.1	37.5-37.7	28-30	-	-
Jahan *et al.,* 2012	Kraft	37.6-39.0	0.8-6.4	38.4-45.4	22-41	-	-
Atik, 2002	Soda-AQ	-	-	45.0-47.3	82-86	-	-

*DP refers to degree of polymerization. Mean values with the same lower-case letters are not significantly different at 95 % confidence level according to Duncan's mean separation test.
**DP se odnosi na stupanj polimerizacije. Srednje vrijednosti s jednakim slovom prema Duncanovu se testu značajno ne razlikuju za razinu pouzdanosti od 95 %.*

Table 5 Physical and optical properties of papers produced from okra pulp
Tablica 5. Fizička i optička svojstva papira izrađenih od pulpe proizvedene od bamije

Cooking No *Kuhanje*	**$NaBH_4$ charge** *Udio $NaBH_4$*, %	**Tensile index** *Vlačni indeks,* N·m/g	**Burst index** *Indeks probijanja,* kPa·m²/g	**Brightness** *Svjetlina* ISO, %
1	0	60.7[c]	3.00[c]	27.5[d]
2	0.3	61.7[b]	3.11[b]	28.3[c]
3	0.5	62.6[ab]	3.26[ab]	28.9[b]
4	0.7	62.8[a]	3.33[a]	29.4[a]
Sig.	-	.003	.000	.000
Jahan *et al.*, 2012	Soda-AQ	39.6-71.4	2.3-4.3	-
Jahan *et al.*, 2012	Kraft	51.3-80.7	3.5-5.3	-
Atik, 2002	Soda-AQ	70-72	1.76-2.85	11.22-12.84

*Mean values with the same lower-case letters are not significantly different at 95 % confidence level according to Duncan's mean separation test. / *Srednje vrijednosti s jednakim slovom prema Duncanovu se testu značajno ne razlikuju za razinu pouzdanosti od 95 %.*

ble 5. It was reported that respective values for wheat stalks were determined as 60.6 (N·m/g), 3.10 kPa·m²/g and 34.8 ISO% (Tutuş and Çiçekler, 2016). In another study, where pulp was produced from cotton stalks, the breaking length and burst index were found to be 4.30 km and 2.60 kPa.m²/g, respectively (Jahan *et al*., 2004). The physical properties of pulp produced from okra stalks are generally similar to other annual plant stalks. Although the brightness value of okra stalks is low, the pulps can be increased to higher brightness values because they are easy to bleach.

It was observed that the physical and optical properties of the papers produced from okra pulp improved with the increase in the $NaBH_4$ charge. Namely, with the addition of 0.7 % $NaBH_4$ to the cooking solution, the brightness increased by about 7 %. In their study Gülsoy *et al*. (2016) conducted kraft pulping from maritime pine wood with the addition of KBH_4, which is a powerful reducing agent similarly as $NaBH_4$. They stated that the optical properties of the obtained papers increased in parallel with the increase in the KBH_4 charge.

In many studies, it has been reported that boron compounds prevent the peeling reaction during cooking and cause less damage to carbohydrates, and therefore, the physical and optical properties of the produced papers improve (Akgül and Temiz, 2006; Istek and Özkan, 2008; Istek and Gonteki, 2009; Gumuskaya *et al*., 2011; Erisir *et al*., 2015; Gülsoy *et al*., 2016).

4 CONCLUSIONS
4. ZAKLJUČAK

In this study, pulp and paper production from okra stalks and the characterization of pulp properties were made. According to the obtained results, the kappa numbers of the pulp produced from okra stalks were low and the viscosity values were high. It was noticed that the pulp yield was slightly lower than found in literature for other non-wood stalks. However, chemical properties show that okra stalk can be used as an alternative raw material for the pulp and paper industry. The physical properties of paper produced from okra stalk pulp are similar to those produced from wheat stalk and cotton stalk. The brightness values of the pulp of okra stalk were lower than literature values of the other non-wood stalks. However, due to the low kappa numbers, it is easily possible to obtain high-brightness pulps by bleaching. By adding 0.7 % $NaBH_4$ to the cooking liquor, the chemical, physical and optical properties of okra stalk pulps were improved. Based on the results of the analyzed properties, okra stalk pulps may be used to make a variety of papers by blending them with long fibers in certain proportions. Furthermore, by adding okra stalk fibers, the losses in the strength characteristics of the paper manufactured in waste paper recycling may be minimized. Similarly, the potential for using okra stalk fibers in the production of other papers is quite high.

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Identification of Defective Joints in Beams via Torsional Vibration

Identifikacija neispravnih spojeva u gredama uz pomoć torzijskih vibracija

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ABSTRACT • *The determination of the shear dynamic modulus in wood product panels is of greater importance than its determination in wood beams. Yet with regards to the effect of defects on the obtained values of shear modulus, this factor is used to detect the defects and the degree of the defects. In this study, the effect of gluing defects in two joint shapes (scarf (45°) and finger joints (10 mm length and 3 mm pitch) were evaluated non-destructively using the torsional vibration method. Rectangular wood specimens with dimensions of 20 mm × 40 mm × 360 mm (R × T × L) were prepared from clear eastern beech. The joints were located at similar positions in the middle of the beams. The results showed that creating a healthy joint did not significantly change the values obtained for the shear modulus in either of the joint shapes. However, reducing the adhesion level in both types of joints caused a significant reduction in the shear modulus values. The results show that the changes in the dynamic shear modulus values in both joint types, as a result of changes in the glue coverage rating (fully, medium and low-glued joints) allows the detection of the defects as well as the degree of the defects.*

KEYWORDS: *joint; prediction; defect; torsional vibration; beech; adhesive*

SAŽETAK • *Određivanje dinamičkog modula smicanja u drvnim pločama važnije je od određivanja tog modula u drvenim gredama. Ipak, s obzirom na utjecaj grešaka na vrijednosti modula smicanja, određivanje tog faktora važno je za otkrivanje grešaka i njihova stupnja na drvnim proizvodima. U ovom je istraživanju metodom torzijskih vibracija nedestruktivno procijenjen utjecaj grešaka lijepljenja u dva oblika spoja: kosim sljubom (45°) i klinastim zupcima (duljine 10 mm i koraka 3 mm). Uzorci drva dimenzija 20 mm × 40 mm × 360 mm (R × T × L) izrađeni su od čiste bukovine, a spojevi su izvedeni na približno istim mjestima na sredini greda. Rezultati su pokazali da izrada zdravog spoja nije bitno promijenila vrijednosti modula smicanja ni za jedan od primijenjenih tipova spoja. Međutim, smanjenje razine adhezije u obje vrste spojeva uzrokovalo je znatno smanjenje vrijednosti modula smicanja. Rezultati pokazuju da promjene vrijednosti dinamičkog modula smicanja u oba tipa spoja kao rezultat promjene stupnja pokrivenosti ljepilom (potpuna, srednja i slaba pokrivenost) omogućuju otkrivanje grešaka lijepljenja, kao i stupnja grešaka drvnih spojeva.*

KLJUČNE RIJEČI: *spoj; predviđanje; greška; torzijske vibracije; bukovina; ljepilo*

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1 INTRODUCTION
1. UVOD

The application of adhesive joints has become widespread in many wood utilization fields since their first commercial use in the late 1950s. It has become one of the most frequently used jointing methods to produce larger products while using short pieces of lumber. Adhesive joints have numerous applications in structural members, e.g., glulam and end to end joints, which were developed to reduce the waste of high-quality lumber, and are some of the most economically valuable ways to utilize wood. Low-grade wood can be used to produce high quality final products with improved strength and appearance, and without undesirable characteristics (Roohnia *et al.,* 2012). However, some of the bonding joints may lose their efficiency due to environmental factors (temperature changes or moisture absorption/desorption) or mechanical stresses that occur during service time. Thus, as producing reliable joints is important, improving them throughout service time would be equally important (Biechele *et al.,* 2010). Various techniques are used for the quality control of bonding joints. According to the type of joint, some standards have been edited (ASTM D905, ASTM D906 2004; ASTM D1073 2012), but these methods are time-consuming and destructive, so controlling and inspecting of all the joints might not be possible (Ayarkwa et al. 2001). In addition, destructive techniques are not applicable throughout service time. Non-destructive testing (NDT) is an effective method for quickly testing and evaluating the properties of materials, and does not alter the physical, chemical, and mechanical properties of the materials. Furthermore, it has no influence on future performance. The exploitation and application of this technology has been quickly developed in the field of wood and wood-products, evidencing its advantages (Ross and Pellerin 1994). In recent years, nondestructive techniques have been used by many researchers to identify the defects in wood and wood products (Lee et al. 2000; Axmon et al. 2004; Baskaran and Janawadkar 2005). Most of the research on joints and non-destructive testing is related to the changes in the modulus of elasticity; some of these studies are outlined below. Rohnia et al. (2012) stated that defects in the joint adhesion (scarf and finger joints) reduce the modulus of elasticity due to longitudinal and flexural vibration. In 2014, Roohnia et al. reported that, by reducing the joint angle in the scarf joints species of oak wood (Quercus Castaneifolia), the modulus of elasticity of this species was reduced (the study of joint angles of 60, 65, 70 and 75 degrees). They also stated that joining with isocyanate adhesive causes a greater drop in dynamic modulus values than polyvinyl acetate adhesive. In 2017, Lara-Bocanegra et al. examined the effect of adhesive type on the flexural strength of Eucalyptus globulus-type finger joints. They stated that the use of 1C-PUR glue significantly increases the finger joint strength in this species. Although torsional properties are considered in the plates, they are less considered in vibrating beams (Bodig and Jayne 1993). However, during troubleshooting, the dynamic response of the beams can be discussed in terms of the healthiness or unhealthiness of the beams. Roohnia and Kohantorabi (2015) discussed the efficiency of the torsional vibration method, which was based on the ASTM standard C1548-02 (2002), and is used to determine the value of the shear modulus of wooden beams and plates turned into beams. The main purpose of this study was to investigate the impact of joint disadvantages on the dynamic shear modulus of the structure with different glue covering values for beech wood (*Fagus orientalis* L.). If the applied techniques in this study were effective in detecting defected joints, they would be valuable for grading the joints in structures during service time.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

Fifty five samples without any visual defects were prepared from eastern beech (*Fagus orientalis* L.) commercial lumber with dimensions of 20 mm × 40 × mm × 360 mm (R × T × L), according to ISO standard 3129 (2019). In the selection procedure, all visually sound beams with clear radial and tangential surfa˝wces were accepted. The samples were placed in a conditioning room at 21 °C and a relative humidity of 65 % until reaching equilibrium moisture content (EMC). The specimens were subjected to free flexural vibration via a both-end free beam test. Considering the three initial vibration modes, and based on Timoshenko's bending theory, the specimens with the highest Timoshenko's correlation coefficient in the initial vibration modes were selected for further experiments. This selection criterion was based on the procedure described in the published literature (Brancheriau and Baillères, 2002; Roohnia *et al*., 2012). Accordingly, the total number of specimens decreased to 42. The selection procedure seemed to be necessary to ensure the clarity of the specimens. Therefore, any extra footprints were calibrated as a result of the positive or negative perfectness of the joints. Each sample was then individually subjected to the torsional vibration test (Figure 1). The torsional vibration was performed on the samples according to ASTM standard C1548 (2002). The selected specimens were divided into two groups, based on the differences in joint shape, i.e., scarf and finger joint. Three different amounts of gluing coverage were applied to the surface of each type of joint. The length of the finger joint was selected at 10 mm, the pitch was selected at 3 mm, and the total number of fingers was 7, with a joint member every 6 in. across the joint width. The joints were created with maximum accuracy using sharp blades manufactured by Makita® Co. (Aichi, Japan) for scarf joints and by Laguna Tools (Grand Prairie, TX) for finger-joints. The separated members were re-assembled in three different manners: fully glued (100 % of the surface glued), medium glued, and low glued. The medium glued and low glued joints for both joint types were considered defective in this study (Figure 2 and Table 1). The glue used in this study was conventional white glue (polyvinyl acetate, with the concentration of 65 %, pH of 5, and hardening time of 20 min) (Chasb Chob Shomal, Tehran, Iran). The glue solution was spread on the joint surfaces at a rate of 150 g/m^2 to 200 g/m^2, based on joint surface area. The joint surfaces were pressed in a constant mechanism during the hardening time. For the scarf joints, two lateral supports were also applied to ensure the exact axial orientation of the joint members without any unwanted shear movement by the joint surfaces.

2.2 Methods
2.2. Metode

Finally, each specimen set was individually subjected to the torsional vibration test. The shear modulus values were evaluated according to the equations in ASTM standard C1548-02 (2002) and the individual sets were compared. These evaluations were made using NDTlab® Software (Islamic Azad University-Karaj Branch, v.2.03, Karaj, Iran) (Roohnia *et al*., 2006). The shear modulus was calculated according to Eq. 1, Eq. 2, and Eq. 3,

$$G=\left\{\frac{4\cdot L\cdot m\cdot f_t^2}{b\cdot t}\right\}\left\{\frac{B}{1+A}\right\} \tag{1}$$

$$A=\left\{\frac{0.5062-0.8786\cdot\left(\frac{b}{t}\right)+.03504\cdot\left(\frac{b}{t}\right)^2-0.0078\cdot\left(\frac{b}{t}\right)^3}{12.03\cdot\left(\frac{b}{t}\right)+9.892\cdot\left(\frac{b}{t}\right)^3}\right\} \tag{2}$$

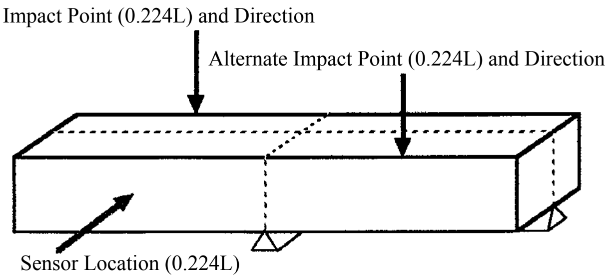

Figure 1 Experimental setup of torsional vibration test
Slika 1. Eksperimentalne postavke ispitivanja torzijskih vibracija

$$B=\left\{\frac{\left(\frac{b}{t}\right)+\left(\frac{t}{b}\right)}{4\cdot\left(\frac{t}{b}\right)-2.52\cdot\left(\frac{t}{b}\right)^2+0.21\cdot\left(\frac{t}{b}\right)^6}\right\} \tag{3}$$

Where G is the shear modulus (Pa), L is the total length of the beam (mm), m is mass of the bar (g), f_t is the fundamental resonant frequency of the bar in torsion (Hz), b is the width of the beam (mm), t is the thickness of the bar (mm), and A and B are the correction coefficients.

The correlation among the obtained values of the dynamic shear modulus of the clear beech beams, jointed in two shapes and three amounts of glue, i.e., fully glued, medium glued, and low glued, was assessed via the Pearson correlation test and the regression fit model. A comparison of each shear modulus obtained from each test stage was also made via a statistical *T*-test at a 95 % confidence level. The SPSS software (Version 11.5, IBM Co., Armonk, NY) was used for the statistical tests and Microsoft Excel 2013 was used to draw the regression lines and diagrams. The comparison was based on the clarity of the wood.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

Prior to presenting the research findings, a discussion on the definition of elastic properties is necessary. Elastic properties are material properties that do

Table 1 Description of joints and glued areas (as shown in Figure 2a and 2b)
Tablica 1. Opis spojeva i područja lijepljenja (prikazano na slikama 2.a i 2.b)

Joint *Spoj*	**Total nominal joint area** *Ukupna nominalna površina spoja* mm × mm	**Fully glued joint area** *Potpuna pokrivenost površine spoja ljepilom* mm × mm	**Medium glued joint area** *Srednja pokrivenost površine spoja ljepilom* mm × mm	**Low glued joint area** *Slaba pokrivenost površine spoja ljepilom* mm × mm
Scarf 45° *kosi sljub od 45°*	55 × 20 (width × height) *(širina × visina)*	55 × 20 (100 %)	5 × 20 near both edges + middle *5 × 20 blizu oba ruba + sredina* (27.3 %)	5 × 20 near both edges *5 × 20 blizu oba ruba* (18.2 %)
Finger *zupci*	120 × 20 (width x height) *(širina × visina)*	120 × 20 (100 %)	10 × 20 edge fingers + two middle fingers / *10 × 20 krajnji zupci + 2 srednja zupca* (33.3 %)	10 × 20 edge fingers only *10 × 20 samo krajnji zupci* (16.7 %)

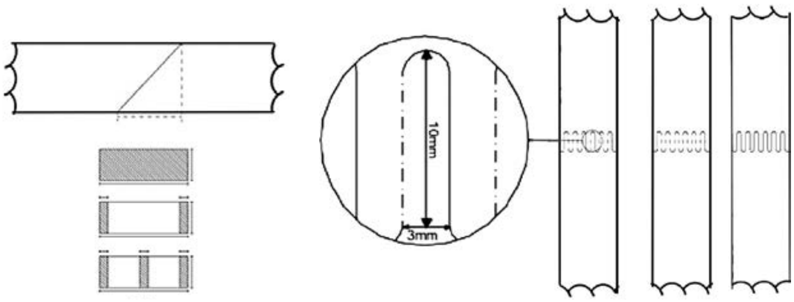

Figure 2 Joint shapes and glue coverage (view from the wider surface of the beam): a) Group one, 45° scarf joint; and b) Group two, finger joint (Note: -.-.- indicates the area without glue)
Slika 2. Tipovi spojeva i pokrivenost ljepilom (pogled na širu površinu grede): a) prva skupina, kosi sljub od 45°; b) druga skupina, spoj zupcima (-.-.- označava područje bez ljepila)

not change after geometrical and artificial manipulations of the beam. However, in this study, these properties have been specifically defined as the dynamic response of a beam, even though the dynamic response of the bar would be affected (Roohnia *et al*., 2012; Kohantorabi *et al*., 2015).

The shear moduli values of the fully glued specimens with the scarf joint and finger joint are plotted against a solid beam in Figure 3 and 4.

There was no significant difference between the solid wood and the fully glued specimens in terms of the dynamic shear modulus (Figure 3).

Statistically equivalent results of dynamic shear modulus values of solid beams and jointed beams (healthy joint without defect in glue coverage), obtained from ASTM C 1548 standard, has not been previously studied. However, according to the results of the first part of this study, which was published in 2012, the dynamic modulus of elasticity of the specimens remained unchanged due to the creation of both types of joints (Roohnia *et al*., 2012). In a similar study, Yavari *et al.* (2015) stated that finger joint and scarf joint in oak beams did not alter the values of dynamic modulus of elasticity compared to solid beams (Yavari *et al*., 2015). Therefore, according to the results of this study, it can be said that the creation of these two types of joints, if they are without defects in glue coverage, does not change significantly the values of the dynamic shear modulus.

The dynamic response of the shear modulus values for the medium glued specimens are plotted against a solid beam with respect to finger and scarf joints in Figure 5 and 6. When the glued members were assembled with a medium amount of glue, the values of the dynamic shear moduli decreased by 5 % in the finger

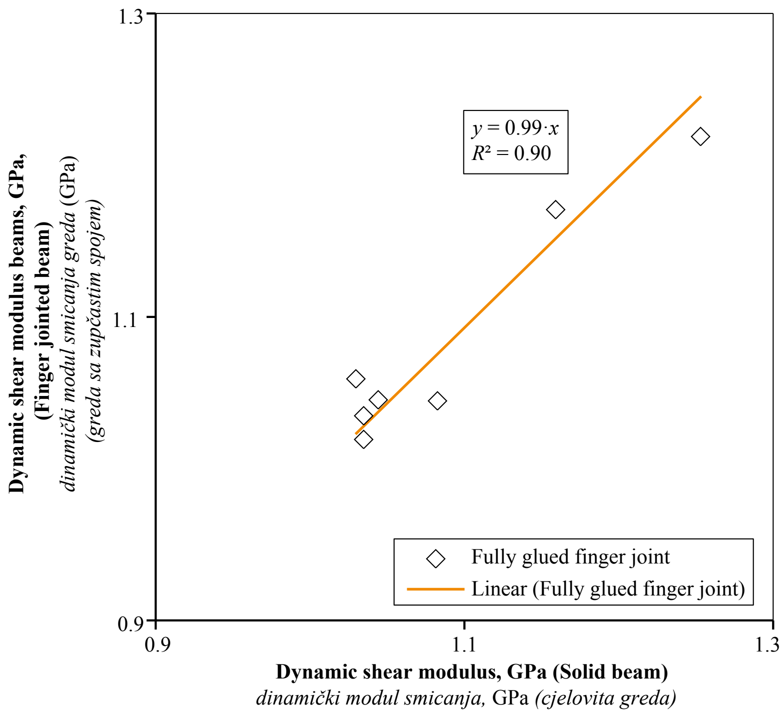

Figure 3 Shear modulus values of fully glued finger jointed specimens against a solid beam after torsional vibration test
Slika 3. Vrijednosti modula smicanja uzoraka spojenih zupcima, uz potpunu pokrivenost površine spoja ljepilom, u odnosu prema cjelovitoj gredi nakon ispitivanja torzijskih vibracija

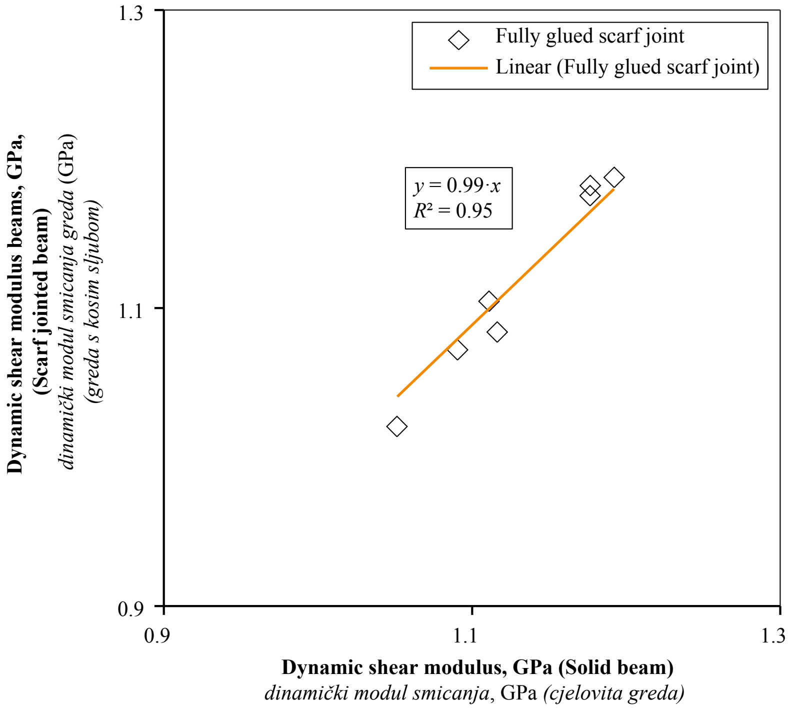

Figure 4 Shear modulus values of fully glued scarf jointed specimens against a solid beam after torsional vibration test
Slika 4. Vrijednosti modula smicanja uzoraka spojenih kosim sljubom, uz potpunu pokrivenost površine spoja ljepilom, u odnosu prema cjelovitoj gredi nakon ispitivanja torzijskih vibracija

joint samples and by 12 % in the scarf joint samples. The reason for this, in addition to the amount of glue used in the finger joint, could also be related to the physical shape of the finger joint.

The dynamic changes of the shear modulus values of the low glued specimens in finger and scarf joints are plotted against a solid beam with respects to finger and scarf joints in Figure 7 and 8. When the glued members were assembled with a low amount of glue, the values of the dynamic shear modulus decreased by 10 % in the finger joint samples and by 27 % in the scarf joint samples. As shown in Table 1, in

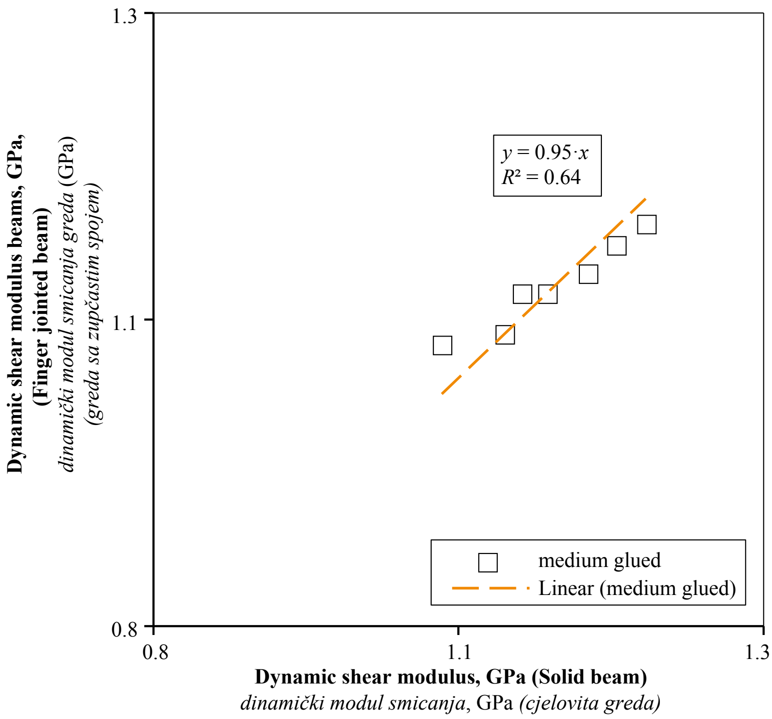

Figure 5 Shear modulus values of medium glue finger jointed specimens against a solid beam after torsional vibration test
Slika 5. Vrijednosti modula smicanja uzoraka spojenih zupcima, uz srednju pokrivenost površine spoja ljepilom, u odnosu prema cjelovitoj gredi nakon ispitivanja torzijskih vibracija

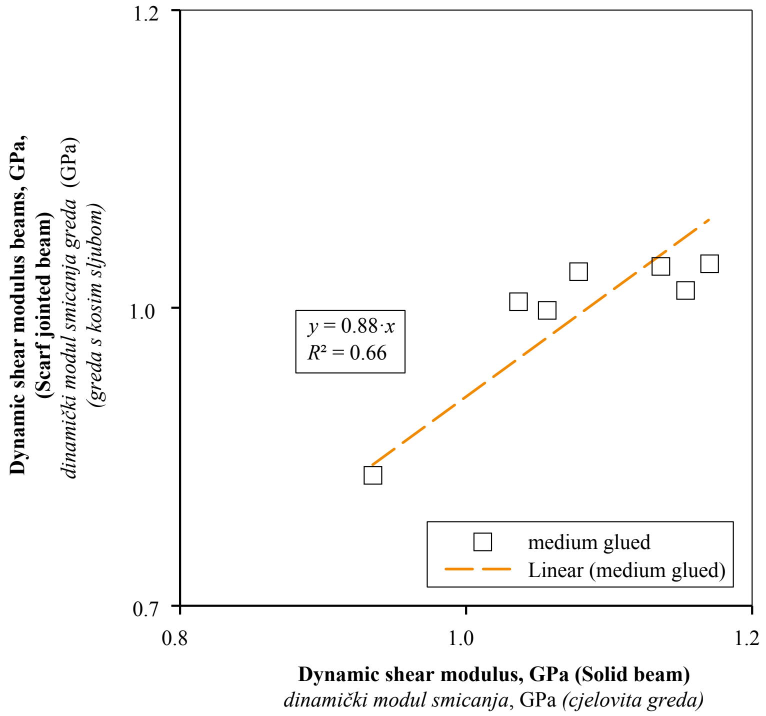

Figure 6 Shear modulus values of medium glue scarf jointed specimens against a solid beam after torsional vibration test
Slika 6. Vrijednosti modula smicanja uzoraka spojenih kosim sljubom, uz srednju pokrivenost površine spoja ljepilom, u odnosu prema cjelovitoj gredi nakon ispitivanja torzijskih vibracija

the low glued joint samples, the amount of adhesive used in the finger joint samples was greater than that in the scarf joint samples. However, it seems that the physical shape of the finger joint and fingers interlock had a greater effect on the dynamic shear modulus.

In previous researches on the elastic properties of the jointed beams, cases such as the effect of finger length, the effect of the type of adhesive, the effect of the angle of joint, etc. have been investigated. Each of the above has a different effect on the dynamic properties of the jointed beams (Ayarkawa *et al.*, 2000; Bustos *et al.*, 2003; Ozcifci and Yapici, 2008; Hemmasi *et al.*, 2014; Roohnia *et al.*, 2012, 2014; Yavari *et al.*, 2015). The effect of the dimensions of adhesion defects

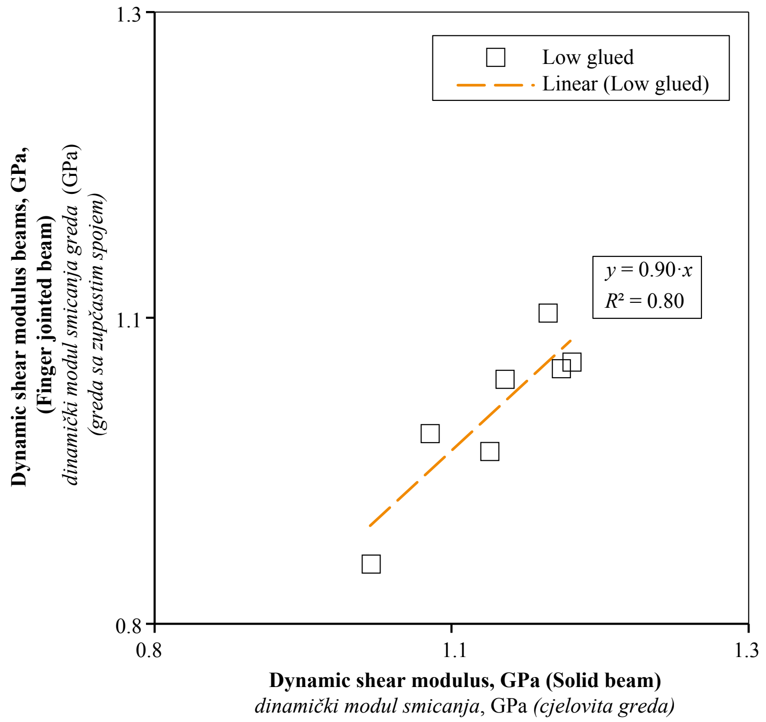

Figure 7 Shear modulus values of low glue finger jointed specimens against a solid beam after torsional vibration test
Slika 7. Vrijednosti modula smicanja uzoraka spojenih zupcima, uz slabu pokrivenost površine spoja ljepilom, u odnosu prema cjelovitoj gredi nakon ispitivanja torzijskih vibracija

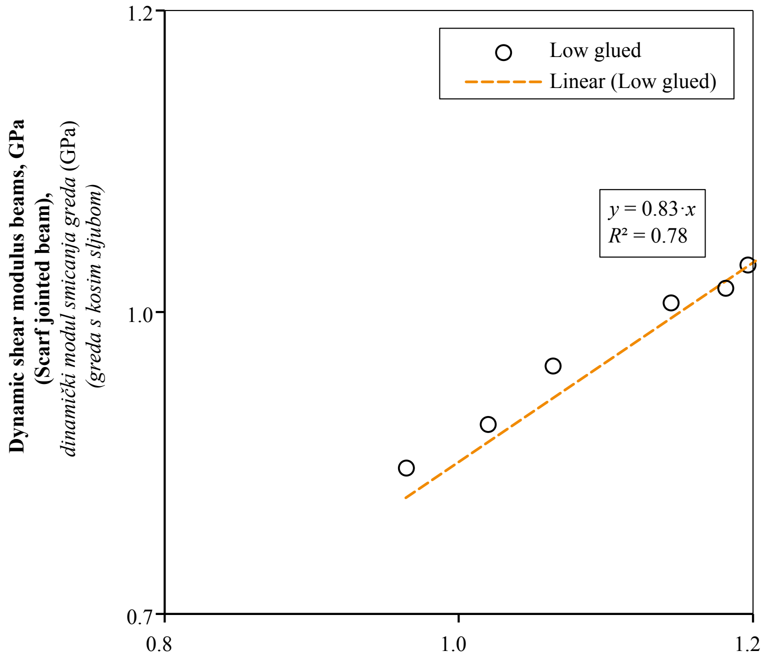

Figure 8 Shear modulus values of low glue scarf jointed specimens against a solid beam after torsional vibration test
Slika 8. Vrijednosti modula smicanja uzoraka spojenih kosim sljubom, uz slabu pokrivenost površine spoja ljepilom, u odnosu prema cjelovitoj gredi nakon ispitivanja torzijskih vibracija

on the dynamic properties has been frequently obtained by ultrasound test. Research has shown that the size of the defects in the adhesive has a significant effect on the values of elastic parameters of ultrasound tests (Tucker *et al*., 2003; Grimberg *et al.,* 2005; Baskaran and Janawadkar, 2007). In the first part of this study, the size of the defects in adhesive had a significant effect on the dynamic modulus of elasticity. As results indicate, the dimensions of defects in adhesive cause a significant reduction of the dynamic shear modulus in the jointed beams with defects. These changes in values are considered efficient in detecting the defect and its degree in scarf and finger joints.

4 CONCLUSIONS
4. ZAKLJUČAK

No significant differences were found between the calculated dynamic shear modulus values in terms of the fully glued specimens and the solid wood sample for both types of joint (scarf and finger joints). It seems that this lack of significant difference is a suitable criterion for identifying healthy glued joints.

Weaker joints, in both types of joints, showed unequal response to the evaluated dynamic shear modulus of the medium and low glued specimens obtained from the results of torsional vibration test.

The amount of reduction in the values of the dynamic torsional modulus in the scarf joint samples was greater than in the finger joint samples, which could be attributed to the physical shape of this joint.

Due to the inhomogeneous dynamic response received from both adhesion levels in both types of joints, when utilizing the torsional vibration method, in addition to the defective scarf and finger joints, the amount of defects in these two types of joints can be determined.

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Effects of Silane Treatment on Physical and Mechanical Properties of Particleboards Prepared with Urea Formaldehyde

Učinci obrade silanom na fizička i mehanička svojstva ploče iverice izrađene s urea-formaldehidnim ljepilom

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ABSTRACT • *In this study, the effect of silane on particleboard physical and mechanical properties was investigated. Silane was mixed in particleboards with two different methods. In the first method, silane was mixed with wood chips namely as a pretreatment of wood chips. In the second method, silane was mixed with urea-formaldehid adhesive. The amounts of silane used in both methods were 1 %, 2 % and 3 % of the particles based on the dry weight. Urea formaldehyde with 65 % solid content was used as adhesive. Ammonium sulfate (1 %) was added as a hardener to the used adhesive. The target density of three-layer particleboards manufactured was 550 kg/m³. The properties of particleboards evaluated include thickness swelling, bending properties and internal bond strength. Results indicate that particleboards thickness swelling values increase with the use of silane. Overall, pretreatment of the wood particles provided better mechanical properties than the addition of silane to the adhesive solution. Flexure strength and flexure modulus of particleboards were significantly improved by use of silane. Pretreatment of particles with 2 % silane yielded the best improvement of modulus of rupture and modulus of elasticity. For the particleboards prepared with pretreated wood chips with silane, the highest increase of the flexure modulus and flexure strength was 20 % and 40 %, respectively. Pretreatment of wood particles with 3 % of silane nearly doubled internal bond strength of the particleboards. Silane pretreatment can be an alternative method for the improvement of particleboard mechanical properties.*

KEYWORDS: *silane; particleboard; physical and mechanical properties*

SAŽETAK • *U ovom je istraživanju ispitivan učinak silana na fizička i mehanička svojstva ploče iverice. Obrada ploče iverice silanom provedena je primjenom dviju metoda. U prvoj metodi silan je pomiješan s drvnim iverjem, što je ujedno bila predobrada drvnog iverja. U drugoj metodi silan je pomiješan s urea-formaldehidnim ljepilom. Količine silana upotrijebljenoga u obje metode bile su 1 %, 2 % i 3 % s obzirom na sadržaj suhe tvari. Kao ljepilo je rabljeno urea-formaldehidno ljepilo sa 65 % suhe tvari. Ljepilu je dodan amonijev sulfat (1 %) kao otvrdnjivač.*

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Ciljana gustoća proizvedene troslojne ploče iverice bila je 550 kg/m³. Istražena svojstva iverice obuhvatila su debljinsko bubrenje, savijanje i čvrstoću raslojavanja. Rezultati pokazuju da se pri upotrebi veće količine silana vrijednost debljinskog bubrenja ploče iverice povećava. Prethodna obrada drvnog iverja rezultirala je boljim mehaničkim svojstvima iverice nego dodavanje silana ljepilu. Čvrstoća na savijanje i modul savitljivosti ploče iverice znatno su poboljšani primjenom silana. Predobradom iverja s 2 % silana postignuto je najbolje poboljšanje modula loma i modula elastičnosti. Za ploču ivericu pripremljenu s drvnim iverjem prethodno obrađenim silanom najveće povećanje modula savitljivosti i čvrstoće na savijanje iznosilo je 20 %, odnosno 40 %. Predobrada drvnog iverja s 3 % silana gotovo je udvostručila čvrstoću raslojavanja iverice. Predobrada silanom može biti alternativna metoda za poboljšanje mehaničkih svojstava ploče iverice.

KLJUČNE RIJEČI: *silan; ploča iverica; fizička i mehanička svojstva*

1 INTRODUCTION
1. UVOD

Particleboards which are commonly used in furniture construction are one of the oldest engineered wood base materials. Physical and mechanical properties of particleboards are affected by a variety of factors such as particle geometry, wood species, density, adhesive type and ratio, etc. (Sanabria *et al.*, 2013; Istek and Ozlusoylu, 2017; 2019). Urea formaldehyde (UF) adhesive is the most commonly used adhesive in the particleboard industry due to some advantages such as cheapness, availability, easy curing, etc. Formaldehyde emission is the most important issue that the manufacturers and users encounter. Properties of urea formaldehyde (UF)-bonded particleboards are usually inferior (Han *et al.*, 1998) comparing to the particleboards manufactured using phenol (PF) or melamine formaldehyde (MUF) isocyanate (Istek *et al.*, 2020). The dimensional stability of particleboards can be improved by using water resistance adhesives such as isocyanate (Han *et al.*, 1998), water repellents, higher adhesive ratio (Ayrilmis *et al.*, 2012) or chemical modification (Clemons *et al.*, 1992; Rowell *et al.*, 1995; Mahlberg *et al.*, 2001). Some pretreatments are applied to the particles such as cold or hot water soaking (Zheng *et al.*, 2006; Pan *et al.*, 2007; Hartono *et al.*, 2018). NaOH (Fenghu *et al.*, 1993; Ishak *et al.*, 2013), enzyme (Zhang *et al.*, 2003) is also found to be effective in order to improve some physical and mechanical properties of particleboards manufactured using urea-formaldehyde. Physical and mechanical properties of the particleboards can also be altered by coating materials applied (Norvydas and Minelga, 2006). Liquified wood (Cuk *et al.*, 2011) and nano cellulose (Veigel *et al.*, 2012; Leng *et al.*, 2017) were also used as modifiers for adhesive and found promising for improvement of particleboard properties.

Silane is known to be an efficient coupling agent mostly used in adhesive formulations (Han, 1998; Ishak *et al.*, 2013; Istek *et al.*, 2016). Although silane treatment was found to be effective on some physical and mechanical properties of urea bonded reed and wheat straw particleboards (Han *et al.*, 1998), and phenol bonded rice husk particleboards (Sawawi *et al.*, 2018), negative effects were reported on urea bonded oriented strand lumber (Taghiyari *et al.*, 2017). Lower wettability, superior hardness and improved roughness of poplar–wheat straw particleboards were reported by using silane coupling agent (Hafezi and Doosthoseini, 2014). Another study revealed that using silane in fiberboard production caused color darkening and surface roughness (Istek *et al.*, 2016). According to Kloeser *et al.* (2007), silanes have lowering effect on formaldehyde emission from wood based panels.

In this study, silane was used in both pretreatment of particles and mixture of urea formaldehyde adhesive, and its effects on some physical and mechanical properties of the particleboards manufactured were investigated.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

Particles consisting of pine (85 %) and poplar (15 %) wood and urea formaldehyde adhesive were provided from a local particleboard factory (Masstaş Aş, Mudurnu, Bolu, Turkey). Urea formaldehyde (E2 class) with a solid content of 65 %, gel time of 35 s, viscosity of 450 cp, density of 1.281 g/cm³, pH of 8.2 was used. The amount of adhesive was 11 % and 12 % of the particles based on the dry weight. As a hardener, 1 % ammonium sulfate (solid content of 33 %) was mixed with the adhesive used. Silane was purchased from Wacker Company in the form of Geniosil® GF 9 that has a bridge building property.

Particleboards measuring 18 mm × 600 mm × 600 mm were prepared in the laboratory conditions. Two approaches were considered in the production of experimental particleboards. In the first, wood particles were pretreated with silane. Then, adhesive was sprayed to the particles until a homogeneous distribution was obtained. In the second, silane was mixed with the adhesive solution and then sprayed on the particles. After mixing, mats were placed in a steel frame by hand and pressed for 4 minutes at the temperature of 190 °C. The target density of the experimental panels was 550 kg/m³. After pressing, experimental boards were conditioned in the laboratory climate at approximately +20 °C, relative humidity of 65 %.

Table 1 Experimental design used in the study
Tablica 1. Dizajn eksperimenta u ovom istraživanju

Treatment *Obrada*	T1	T2	T3	T4	T5	T6	T7	T8	T9	T10	T11	T12	T13	T14
Treatment type *Vrsta obrade*	-	**Particles treated** *Obrada drvnog iverja*			**Mixed with adhesive** *Miješanje s ljepilom*			-	**Particles treated** *Obrada drvnog iverja*			**Mixed with adhesive** *Miješanje s ljepilom*		
Amount of silane used, % *količina silana, %*	-	1	2	3	1	2	3	-	1	2	3	1	2	3
Amount of adhesive used, % *količina ljepila, %*	11	11	11	11	11	11	11	12	12	12	12	12	12	12

After conditioning, samples were cut to the required size in order to determine some physical and mechanical properties. Experimental design used in the study is presented in Table 1.

The following physical and mechanical properties were determined using the applicable standards: apparent density (TSE EN 323, 1999); moisture content (*MC*) (TSE EN 322, 1999); thickness swelling (*TS*) (Figure 1) after 24 hour of water immersion (TSE EN 317, 1999); internal bond strength (*IBS*) (TSE EN 319, 1999), (Figure 2); modulus of elasticity (*MOE*) (TSE EN 310, 1999) and modulus of rupture (*MOR*) (TSE EN 310, 1999), (Figure 2) in bending.

Ten replicates were used for each test and the obtained data were subjected to an analysis of variance. Experimental results were analyzed using ANOVA tests to identify their statistical significance. Duncan's multiple range tests were performed in order to find the least significant difference between all the variables.

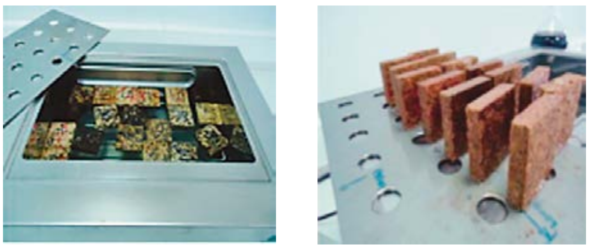


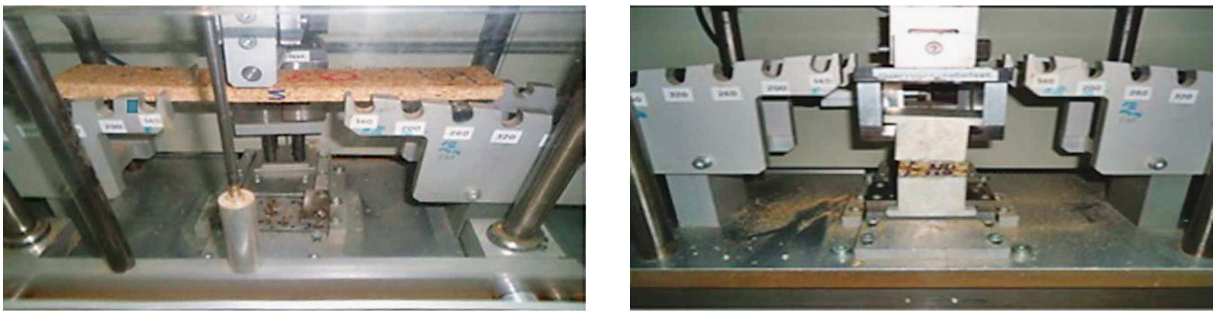
Figure 1 Thickness swelling
Slika 1. Debljinsko bubrenje

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

Table 2 shows some physical and mechanical properties of the laboratory manufactured particleboard samples. Density of the experimental particleboards ranges from 521 to 584 kg/m³ and was not significantly different among the tested group. The use of silane did not significantly alter the density of the particleboards manufactured. Thickness swelling of the particleboards varies between 11.5 % and 30.9 % (Figure 3).

Test results indicate that both the amount of adhesive and silane used influence the thickness swelling of the particleboards. Particleboards (T8) without silane had the lowest thickness swelling values (11.7 %). After 24 hours of water immersion, thickness swelling values were the highest for the treatment T5 with 1 % pretreated particles and 11 % silane. It was considered that T5 has the lowest density (512 kg/m³) among the other samples. The amount of adhesive has greater effect on thickness swelling values than silane used. The addition of a hydrophobic substance such as waxes usually mixed with the adhesive solution in the production of particleboards would change water affiliation, and thus lower thickness swelling values.

Increasing amount of adhesive lowers thickness swelling values of particleboards as expected (Ayrilmis and Nemli, 2017). The use of silane seems to have more apparent effect on particleboards manufactured



a) b)

Figure 2 a) Bending strength, b) Internal bond strength
Slika 2. a) Čvrstoća na savijanje, b) čvrstoća raslojavanja

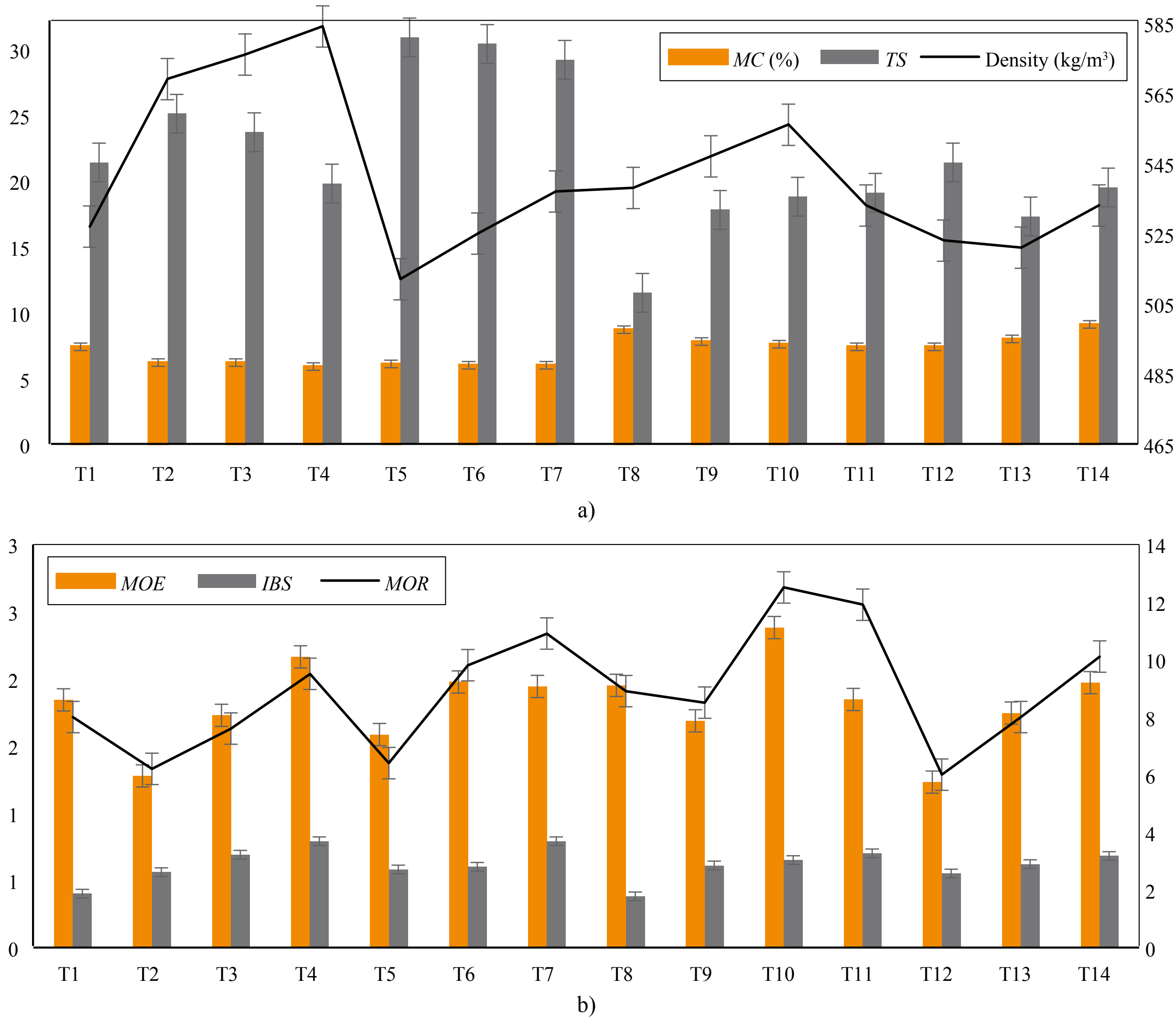


Figure 3 a) Physical and b) mechanical properties of particleboards
Slika 3. a) Fizička svojstva ploče iverice, b) mehanička svojstva ploče iverice

using 11 % adhesive than those manufactured using 12 % adhesive. Higher thickness swelling values may be attributed to the buffering effect of silane. The effect of silane on thickness swelling values of wood based panels in the literature is controversial. After the use of silane, increase of TS was reported for OSL manufactured using poplar and urea formaldehyde (Taghiyari *et al.*, 2017) and particleboards manufactured using 0.5 mesh Kelampayan particles (Ishak *et al.*, 2013). An investigation on reed and wheat straw particleboards (Han *et al.*, 1998) presented that the use of silane coupling agents improves TS values. A study conducted by Kharazipour *et al.* (2009) shows that the use of silane in the manufacture of MDF reduces the 2-hour TS values by half. Improvement in TS for rice husk particleboards was also reported when silane was used with phenol formaldehyde (Sawawi *et al.*, 2018).

Modulus of elasticity in bending values of tested particleboards varies between 1230 N/mm^2 and 2380 N/mm^2. Bending strength of the panels ranges from 6 N/mm^2 to 12.5 N/mm^2. ANOVA results indicate that bending properties among the treatments differ, but all results are in the same group (A and AB). The highest values of bending properties were obtained by treatment T10 in which particles were pretreated with 2 % silane and 12 % adhesive used (Figure 3). It seems that pretreatment of particles yields better bending *MOE* and *MOR* values than when silane is mixed with the adhesive solution. 1 % increase in the amount of adhesive causes approximately 5 % increase in *MOE,* while the increase in *MOR* is more than 10 %. Comparing with the control groups (T1 and T8), the increase in bending properties with the addition of silane is more efficient (more than 20 % for stiffness, 40 % for bending strength) than adhesive quantity increases (from11 % to 12 %). Higher bending properties are expected with the use of a higher amount of adhesive (Rangavar *et al.*, 2016; Ayrilmis and Nemli, 2017) but interaction of silane with the adhesive used induces excelsior bending properties.

The static bending strength requirement for general-purpose particleboards (TS EN 312, 2005) is 11.5 N N/mm^2. Treatment T10, manufactured with pretreated silane, exceedingly meets the minimum values of bending properties required by the standards.

Table 2 Some physical and mechanical properties of particleboard samples
Tablica 2. Fizička i mehanička svojstva uzoraka ploče iverice

Treatment *Obrada*	**Density, kg/m³** *Gustoća,* kg/m³	***MC*, %**	***TS*, %**	***MOE*, N/mm²**	***MOR*, N/mm²**	***IBS*, N/mm²**
T1	527	7.4	21.4 AB*	1842 AB	8 AB	0.4 A
T2	569	6.2	25.1 AB	1276 A	6.2 A	0.56 A
T3	576	6.2	23.7 AB	1727 A	7.6 A	0.69 AB
T4	584	5.9	19.8 AB	2162 AB	9.5 AB	0.74 AB
T5	512	6.1	30.9 B	1584 A	6.4 A	0.58 A
T6	525	6	30.4 AB	1975 AB	9.8 AB	0.6 AB
T7	537	6	29.2 AB	1942 AB	10.9 AB	0.79 B
T8	538	8.7	11.5 A	1950 AB	8.9 AB	0.38 A
T9	547	7.8	17.8 A	1586 A	8.5 AB	0.61AB
T10	556	7.6	18.8 A	2380 B	12.5 B	0.65 AB
T11	533	7.4	19.1 A	1845 AB	11.9 AB	0.7 AB
T12	523	7.4	21.4 AB	1230 A	6 A	0.55 A
T13	521	8	17.3 A	1744 AB	8 A	0.62 AB
T14	533	9.1	19.5 AB	1970 AB	10.1 AB	0.68 AB

*Indicates Duncan grouping / *označava Duncanovo grupiranje*

Internal bond strength of control groups is not significantly different. Internal bond strength is increased by the amount of adhesive used and addition of silane. The minimum requirement of internal bond strength for general-purpose particleboards (TS EN 312, 2005) is 0.24 N/mm². All of the boards manufactured in the study exceed the minimum requirements.

Internal bond strength is nearly doubled by the treatment groups of T7 and T11, manufactured with pretreated particles with 3 % silane. Similar results were also reported for internal bond strength of MDF (Kloeser *et al.*, 2007; Kloeser, 2010), particleboards manufactured using Kelampayan particles (Ishak *et al.*, 2013).

4 CONCLUSIONS
4. ZAKLJUČAK

This study investigated the influence of silane pretreatment of particles and silane addition to the adhesive solution on some physical and mechanical properties of particleboards. Contrary to previous studies, silane negatively affected the thickness swelling and moisture content of particleboards in this study. It is considered that particleboard density played an important role in this difference. In this study, lower particleboard density was observed than in controversial studies. Mechanical properties of the particleboards improved with silane because silane increased the internal bond across the particle and adhesive in the board. *MOE*, *MOR* and *IBS* test results show that silane increases the internal bonding of particleboard because it joins the particles to each other very effectively. Pretreatment of particles was found to be a more efficient approach than silane addition to the adhesive solution. Water repellents and manufacturing higher density board could yield better board properties. Silane could be used for improvement of particleboard properties, and it could be an alternative to other pretreatments or chemical additives.

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Fracture Behavior of Wood Under Mode I Loading in Tangential Direction

Ponašanje loma drva pri vlačnom opterećenju (model I.) u tangentnom smjeru

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ABSTRACT • *The aim of this study was to determine the fracture behavior of southern yellow pine (Pinus taeda L.) and red oak (Quercus falcata) wood under mode I loading in the tangential-radial and tangential-longitudinal crack propagation systems by a compact tension test method. The results of the study indicated that, in general, red oak had a significantly different fracture behavior than southern yellow pine for each of the two crack propagation systems. The fracture toughness was higher in the tangential-radial crack propagation system than that in the tangential-longitudinal crack system, but there was no significant difference between the two crack propagation systems for southern yellow pine. The specific fracture energy of the tangential-longitudinal crack propagation system for both wood species was significantly lower than that of the tangential-radial crack propagation system. It means that more energy per unit area for the tangential-radial crack propagation system was needed to separate a wood sample into two halves. The difference in the fracture behavior of wood by the crack propagation system can be explained by the structural features of the tested samples since the crack propagation of the tangential-radial system crosses the annual ring and wood fibers can bridge the crack surface.*

KEYWORDS: *fracture; fracture toughness; southern yellow pine; red oak; specific fracture energy*

SAŽETAK • *Cilj ovog istraživanja bio je utvrditi ponašanje loma drva južnoga žutog bora (Pinus taeda L.) i crvenog hrasta (Quercus falcata) u tangentno-radijalnome i tangentno-longitudinalnom smjeru širenja pukotine primjenom kompaktne vlačne metode ispitivanja (model I.). Rezultati istraživanja pokazali su bitno drugačije ponašanje loma drva crvenog hrasta od drva južnoga žutog bora za svaki od dva načina širenja pukotine. Lomna žilavost drva crvenog hrasta bila je veća u tangentno-radijalnom smjeru širenja pukotine nego u tangentno-longitudinalnom smjeru, dok na drvu južnoga žutog bora nije uočena značajna razlika između dva načina širenja pukotine. Specifična energija loma u tangentno-longitudinalnom smjeru širenja pukotine za obje vrste drva bila je mnogo niža od one u tangentno-radijalnom smjeru. To znači da je bilo potrebno više energije po jedinici površine da se uzorak drva odvoji na dvije polovice u tangentno-radijalnom smjeru širenja pukotine. Razlika u ponašanju loma drva, odnosno u širenju pukotine može se objasniti strukturnim obilježjima ispitivanih uzoraka jer širenje pukotine u tangentno-radijalnom smjeru prelazi granicu goda i drvna vlakanca mogu premostiti površinu pukotine.*

KLJUČNE RIJEČI: *lom; lomna žilavost; južni žuti bor; crveni hrast; specifična energija loma*

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1 INTRODUCTION
1. UVOD

It is well-known that wood is an orthotropic material having independent mechanical properties in three different grain orientations of longitudinal (L), radial (R), and tangential (T) direction (Figure 1). Orthotropic materials have six principal systems of crack propagation as shown in Figure 2 (Schniewind and Centeno, 1973), and each of the six systems is indicated by two letters, i.e., the first letter specifies the grain orientation perpendicular to the crack plane, whereas the second letter specifies the direction of crack propagation. For instance, TR indicates that the system has its crack growing in the radial direction on the tangential direction perpendicular to the crack plane.

Fracture is usually defined as a process that changes the structure of the material results in broken bonds and new surfaces are formed when a sufficient load is applied (Vasic, 2000; Smith *et al*., 2003). Atack *et al.* (1961) first applied the concepts of fracture mechanics to wood. Walsh (1972) mentioned that linear elastic fracture mechanics (LEFM) are ideally applicable to wood because a wood member under tensile or shear loads behaves like brittle materials (Smith *et al*., 2003; Stanzl-Tschegg, 2009). Therefore, LEFM can be an efficient tool to investigate wood fracture-related problems (Qiu *et al*., 2012). Zink *et al*. (1995) also mentioned that fracture mechanics is useful in predicting the strength of wood subjected to tensile loads at high angles in grain orientation.

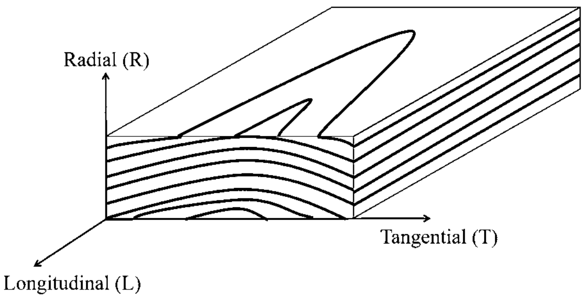

Figure 1 Three principal axes of wood in reference to wood grain orientations
Slika 1. Tri glavne osi drva u odnosu prema orijentaciji drvnih vlakanaca

Fracture toughness is a geometry-independent material property of wood (Mall *et al*., 1983), which is defined as the material's resistance to crack growth (Smith *et al*., 2003). Fracture toughness test can be performed based on three different loading conditions (Conrad *et al*., 2003) as shown in Figure 3: Mode I (tensile mode), Mode II (in-plane shear mode), and Mode III (out-of-plane shear mode). Mode I is typically the dominant failure mode for most engineering materials (Smith *et al*., 2003). Mode I and Mode II are the most common failure modes observed in wooden structures, whereas Mode III fracture occurs in wooden beams with side checks (Patton-Mallory and Cramer, 1987). There is no standardized test method for measuring fracture toughness of wooden materials. Previous studies have been referencing ASTM (E399-09) standard (2009) for metallic materials. The fracture toughness of a wood specimen can be evaluated using a variety of specimen configurations subjected to tensile, shear, or bending loads, i.e., single-edge-notched bending (SENB), single-edge-notched tension (SENT), and compact tension (CT). The CT test method is suitable for testing wood materials when different wood grain orientations are considered (Fonselius and Riipola, 1992). A combination of Mode I and Mode II often occurs together in wooden components in the form of cracking along the grain direction (Qiu *et al*., 2012).

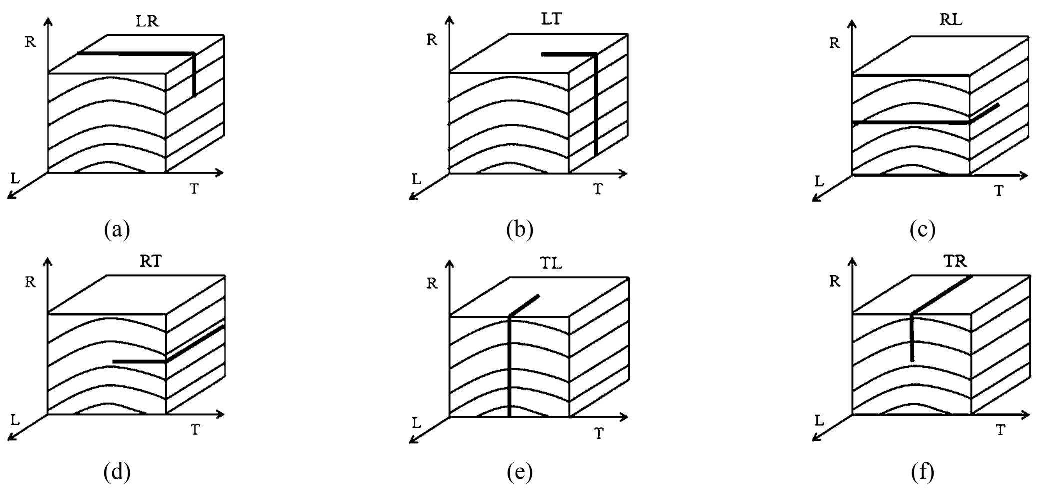

Figure 2 Six crack growth orientations, LR (a), LT (b), RL (c), RT (d), TL (e), and TR (f) in reference to wood grain orientation axis of radial (R), tangential (T), and longitudinal (L) directions, respectively
Slika 2. Šest orijentacija širenja pukotine: LR (a), LT (b), RL (c), RT (d), TL (e) i TR (f) u odnosu prema radijalnoj (R), tangentnoj (T) i longitudinalnoj (L) osi orijentacije žice drva

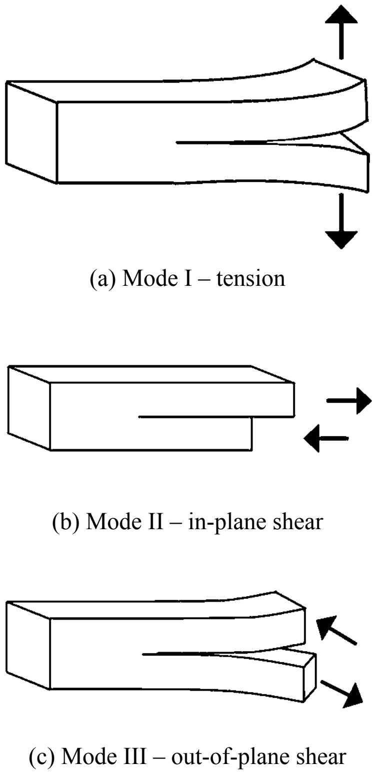

Figure 3 Basic fracture modes of specimens subjected to a tensile load (a), an in-plane shear load (b), and an out-of-plane shear load (c)
Slika 3. Osnovni modeli loma uzoraka izloženih vlačnom opterećenju (a), smičnom opterećenju u ravnini (b) i smičnom opterećenju izvan ravnine (c)

Tan *et al*. (1995) studied fracture toughness of wood failing in Mode I and Mode II and concluded that fracture toughness values of Model II were 4-5 times those of Mode I.

Fracture toughness is a function of applied load, specimen geometry, and crack length. However, fracture toughness of wood is strongly influenced by its natural characteristics such as specific gravity (SG) and environmental conditions such as relative humidity (RH) and temperature (Porter, 1964; Johnson, 1973; Mall *et al*., 1983; Patton-Mallory and Cramer, 1987; Smith *et al*., 2003; Dourado and de Moura, 2019).

All six principal systems (Figure 2) can be divided into two groups according to the location of the crack plane and the direction of crack propagation. The LT and LR systems have been defined as one group, and the RL, RT, TL, and TR as another. Schniewind and Centeno (1973) studied the fracture toughness of air-dry Douglas fir in all six principal systems and concluded that the fracture toughness of two principal systems (LT and LR) were significantly higher than the ones in the rest of the systems. The fracture toughness of air-dry Douglas fir measured in LT and LR systems ranged from 2.42 to 2.69 MPa√m, whereas the fracture toughness in RL, RT, TL, and TR systems ranged from 0.31 to 0.41 MPa√m. The RL and TL systems have been mostly studied because of the low strength and stiffness values of wood perpendicular to the grain (Kretschmann *et al*., 1991; Smith *et al*., 2003) even though these four of six systems (RL, RT, TL, and TR) are of practical importance (Barrett, 1976). Moreover, Qiu *et al*. (2012) mentioned that the RL and RT crack propagation systems were the common cracks observed in wood composites because of their low strength in tension perpendicular to grain.

The fracture toughness of wood increases with increasing its SG (Ashby *et al*., 1985; Conrad *et al*., 2003; Petterson and Bodig, 1983; Schniewind *et al*., 1982). Kretschmann *et al*. (1991) found that the fracture toughness values of wood failed in both Mode I and Mode II and were positively correlated to its SG. Schniewind *et al*. (1982) studied the effects of SG on the fracture toughness of wood using five softwood species (douglas-fir, incense-cedar, pondersa pine I, pondersa pine II, redwood, white fir) and nine hardwood species (apitong, balsa, beech, birch, black oak, lauan, madrone, hard maple, tanoak I, tanoak II) in TL and LT systems. The results indicated that the fracture toughness values can be predicted by highly correlated linear equations with knowing their average SG values (r^2= 0.73 for the TL system and r^2= 0.74 for the LT system).

The fracture toughness of wooden materials can be affected and reduced by increasing their moisture content (MC) (Majano-Majano *et al*., 2012; Reiterer and Tschegg, 2002; Tukiainen and Hughes, 2016a; Tukiainen and Hughes, 2016b; Vasic and Stanzl-Tschegg, 2007). Atack *et al*. (1961) also indicated that the fracture toughness of wooden materials with a higher MC could become questionable because their plasticity could increase during crack propagation. Kretschmann *et al*. (1991) studied the effects of MC on fracture toughness values of Mode I and Mode II for southern pine in the TL system, respectively. The results showed that the fracture toughness values of wood increased with decreasing MC. However, the fracture toughness reached the maximum values with MC ranging from 7.5 and 10 %. Ewing and Williams (1979) studied the effects of specimen thickness and MC on the fracture toughness of scots pine and concluded that the fracture toughness reached the maximum values at the MC of 10 % for all thickness levels investigated.

In this study, we investigated the fracture behavior of wood under pure Mode I loading in the TR and TL crack propagation systems using the CT test method. Results of two wood species, one softwood, southern yellow pine (*Pinus taeda* L.), and one hardwood, red oak (*Quercus falcata*), are presented. Differences between the softwood and hardwood are shown and discussed. Moreover, differences between the two crack propagation systems are discussed.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

Southern yellow pine (*Pinus taeda* L.) (SYP) and red oak (*Quercus falcata*) (RO) lumber were purchased from a local lumber company in Starkville, Mississippi. The selected lumber was straight-grain and free from defects. The CT test specimens were prepared according to the standard established by ASTM (E399-09) standard (2009). Figure 4 shows the general configuration of a fracture toughness testing specimen for a CT test method. Twenty samples were investigated for each crack propagation system and wood species. An initial crack in the test specimens, 1 mm thick, was first cut using a band saw and then extended about 1 mm ahead of the crack tip with a razor blade to make a sharp crack. Prior to the fracture test, all specimens were kept in the conditioning room with the temperature and relative humidity controlled at 20 °C and 42 %, respectively. The average measured density of SYP and RO was 487±24 kg/m³ and 609±7 kg/m³, respectively.

The test was performed on an INSTRON 5566 universal test machine with a loading speed of 2 mm/min. Load-deformation curves of all tested specimens loaded until the complete separation of surfaces occurred were recorded. Three fracture parameters were obtained from the curves, i.e., fracture toughness (K_{IC}), initial slope (k_{init}), and specific fracture energy (G_f). The fracture toughness, K_{IC} (MPa√m), was calculated using the following equation (ASTM 2009):

$$K_{IC} = \frac{P_Q}{B\sqrt{W}} f\left(\frac{a}{W}\right) \tag{1}$$

Where:

$$f\left(\frac{a}{W}\right) = 29.6\cdot\left(\frac{a}{W}\right)^{1/2} - 185.5\cdot\left(\frac{a}{W}\right)^{3/2} + 655.7\cdot\left(\frac{a}{W}\right)^{5/2} - 1017.0\cdot\left(\frac{a}{W}\right)^{7/2} + 638.9\cdot\left(\frac{a}{W}\right)^{9/2} \tag{2}$$

Where: P_Q is the failure load initiating crack propagation (N), W is the distance between the loading point and the end of a CT test block (m), B is the thickness of a CT test specimen (m), a is the initial crack length (m) (Figure 4), and $f(a/W)$ is the polynomial function for wood as an orthotropic material (Fonselius and Riipola, 1992; Smith *et al*., 2003; Ohuchi *et al*., 2011; Wu *et al*., 2012).

The load-deformation curve of a wooden material characterizes its fracture process. In order to characterize the elastic behavior of the material, the initial slope, k_{init}, of the load-deformation curve in the elastic region can be determined by dividing ΔP (the difference between the upper and lower limit of load within the linear elastic region) by $\Delta\delta$ (the deflection difference corresponding to ΔP) (Reiterer et al., 2002; Reiterer and Tschegg, 2002) (Figure 5). The specific fracture energy representing the work required to separate the fracture surfaces was calculated from the integrated area under the whole load-deformation curve (Figure 5) divided by the area of the fracture surface using Eq. 3 (Majano-Majano *et al*., 2010; Reiterer and Tschegg, 2002):

$$G_f = \frac{1}{(W-a)\cdot B}\int_0^{\delta_{max}} P(\delta)\,d\delta \tag{3}$$

Where P is the applied load (N), δ is the deflection at the loading point, W is the width of the test specimen (m), a is the initial crack length (m), B is the thickness of the test specimen (m).

All statistical analyses were carried out using the SAS 9.4 statistical software. A two-factor analysis of variance (ANOVA) general linear model (GLM) procedure was first performed for each property evaluated to analyze the main effects and their interactions, followed by performing mean comparisons if the significant interaction was identified using the protected least significant difference (LSD) multiple comparisons procedure. Otherwise, the main effects were concluded. All statistical analyses in this study were performed at the 5 % significance level.

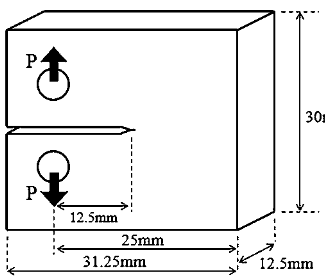

Figure 4 General configuration of a fracture toughness testing specimen
Slika 4. Opća konfiguracija uzorka za ispitivanje lomne žilavosti

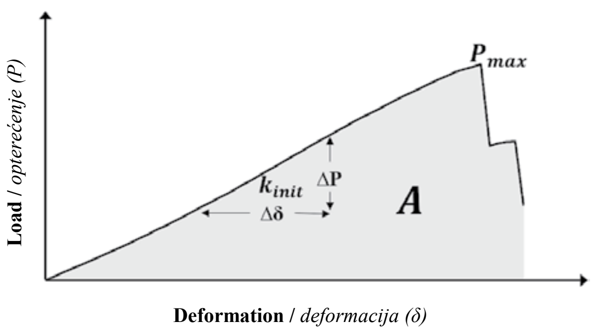

Figure 5 Graphical illustration of how to define initial slope and integrated area based on load-deformation curve
Slika 5. Grafički prikaz definiranja početnog nagiba i integrirano područje na temelju krivulje opterećenje – deformacija

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

Typical load-deformation curves obtained by the CT tests in the TR and TL crack propagation systems of both SYP and RO are shown in Figure 6. The curves can clearly illustrate the effect of wood species and crack propagation systems on fracture behavior. The maximum loads in the load-deformation curve for all specimens in both crack systems were defined as the failure loads because the load was found earlier than the intersection drawn with a 5 % reduction in initial slope (Konukcu *et al.*, 2021). In general, the RO had a greater failure load than the SYP. It was also observed that the failure load in the TR crack system for both wood species was higher than in the TL crack system.

Table 1 summarizes mean values of failure load, fracture toughness, initial slope, specific fracture energy, and brittleness of SYP and RO in the TR and TL crack propagation systems. ANOVA results (Table 2) indicated that the two-factor interaction was significant for fracture toughness. This suggested that further analyses should be focused on the significant interaction. Table 1 also summarizes mean comparisons of fracture toughness for crack propagation system and wood species. The results were based on a one-way classification with four treatment combinations with respect to the two-factor interaction and mean comparisons among these combinations using a single LSD value of 0.05 MPa√m.

In general, RO had a significantly higher fracture toughness than SYP for each of the two crack propagation systems evaluated. Mean comparison results (Table 1) indicated that, in general, the RO had a significantly higher fracture toughness value of 0.78 MPa√m in the TR crack system than in the TL crack system (0.61 MPa√m). The results show that the fracture toughness of RO in the TR crack system was about 28 % higher than that in the TL crack system, while in the case of SYP the difference was about 3 %. The SYP had a greater fracture toughness value of 0.39 MPa√m in the TR crack system than in the TL crack system (0.38 MPa√m), but there was no significant difference between the two crack propagation systems. Previous studies have reported similar results that the TR crack system has a higher fracture toughness value than the TL crack system (Schniewind and Centeno, 1973; Schniewind and Pozniak, 1971; Thuvander and Berglund, 2000).

The specific fracture energy is more suitable for characterizing the fracture behavior of wood (Stanzl-Tschegg *et al.*, 1995) because the fracture is determined not only by the crack initiation process but also by the propagation energy of an existing crack (Majano-Majano *et al.*, 2012). The specific fracture energy, including crack initiation and propagation energies, characterizes

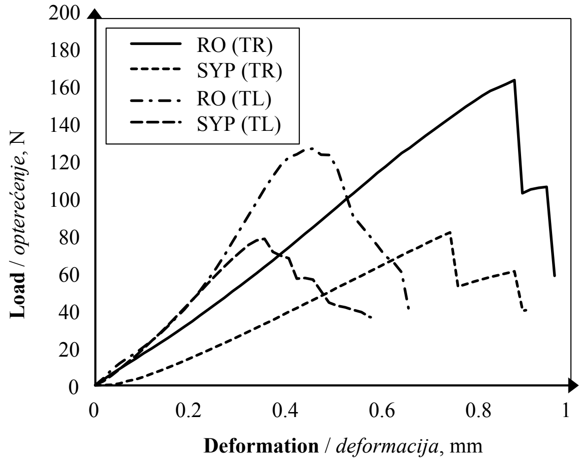

Figure 6 Typical load – deformation curves obtained by CT test in TR and TL crack propagation systems
Slika 6. Tipične krivulje opterećenje – deformacija dobivene CT testom pri TR i TL načinu širenja pukotine

Table 1 Mean values of failure load, fracture toughness, initial slope, specific fracture energy, and brittleness within each combination of crack propagation system and wood species and mean comparisons of fracture toughness and specific fracture energy for crack propagation system and wood species*
Tablica 1. Srednje vrijednosti sile loma, lomne žilavosti, početnog nagiba, specifične energije loma i krtosti unutar svake kombinacije načina širenja pukotine i vrste drva te usporedbe srednjih vrijednosti lomne žilavosti i specifične energije loma za načine širenja pukotine i vrste drva*

Species *Vrsta drva*	**Failure load** *Sila loma,* N		**Fracture toughness** *Lomna žilavost,* MPa/m		**Initial slope** *Početni nagib,* N/mm		**Specific fracture energy** *Specifična energija loma,* J/m²		**Brittleness** *Krtost,* mm	
	TR	TL	TR	TL	TR	TL	TR	TL	TR	TL
SYP	81.16 (9)	77.85 (13)	0.39 (18) (A) (b)	0.38 (18) (A) (b)	128.05 (14)	266.75 (24)	298.26 (21) (A) (b)	181.03 (13) (B) (b)	14.80 (30)	10.29 (11)
RO	159.98 (10)	126.17 (17)	0.78 (12) (A) (a)	0.61 (11) (B) (a)	223.92 (8)	334.10 (20)	444.97 (8) (A) (a)	296.90 (16) (B) (a)	20.83 (18)	12.70 (19)

*Values in parentheses are coefficients of variation in percentage; means not followed by the same uppercase letter in the same row are significantly different one from another at the 5 % significance level considering crack propagation system effect; means not followed by the same lowercase letter in the same column are significantly different one from another at the 5 % significance level considering wood species effect.
**Vrijednosti u zagradama koeficijenti su varijacije u postotcima; srednje vrijednosti iza kojih ne slijedi isto veliko slovo u istom retku međusobno se značajno razlikuju na razini značajnosti od 5 % uzimajući u obzir učinak načina širenja pukotine; srednje vrijednosti iza kojih ne slijedi isto malo slovo u istom stupcu međusobno se značajno razlikuju na razini značajnosti od 5 % uzimajući u obzir učinak vrste drva.*

Table 2 Summary of analysis of variance (ANOVA) results obtained from a general linear model (GLM) procedure performed on two factors for each property evaluated
Tablica 2. Sažetak rezultata analize varijance (ANOVA) dobivenih iz postupka općega linearnog modela (GLM) provedenoga na dva faktora za svako od procijenjenih svojstava

Source / *Izvor*	**Property** / *Svojstvo*			
	Fracture toughness *Lomna žilavost*		**Specific fracture energy** *Specifična energija loma*	
	***F* value** *F-vrijednost*	***p* value** *p-vrijednost*	***F* value** *F-vrijednost*	***p* value** *p-vrijednost*
Wood species / *vrsta drva*	364.18	< 0.0001	170.28	< 0.0001
Crack system / *način loma*	31.03	< 0.0001	173.83	< 0.0001
Wood species × crack system / *vrsta drva × način loma*	20.95	< 0.0001	2.35	0.1296

the whole fracture process until the CT specimen is separated into two halves. Crack initiation energy is the energy that causes the formation of micro-cracks and irreversible deformations around the crack tip, whereas crack propagation energy is the energy that is dissipated through the formation of microcracks that ultimately turn into the main crack (Majano-Majano *et al*., 2010; Reiterer and Tschegg, 2002; Smith *et al*., 2003).

For the specific fracture energy values, ANOVA results (Table 2) indicated that the two-factor interaction was not statistically significant, while the two main effects were both considered statistically significant at the 5 % level. The crack system and wood species effect on the specific fracture energy was determined based on mean comparisons of the main effect directly. Mean comparison results of the specific fracture energy for wood species are summarized in Table 2. The wood species on the specific fracture energy was analyzed by considering the non-significant two-way interaction because the nature of conclusion from interpretation of the main effects also depends on the relative magnitudes of the interaction and individual main effects (Freund and Wilson, 1997). The results were based on a one-way classification with four treatment combinations with respect to the two-factor interaction and mean comparisons among these combinations using a single LSD value of 28.34 J/m^2 for specific fracture energy.

Mean comparison results (Table 1) indicated that in general, the RO had a significantly higher specific fracture energy value of 444.97 J/m^2 in the TR crack system than in the TL crack system (296.90 J/m^2), whereas the SYP had a greater specific fracture energy value of 298.26 J/m^2 in the TR crack system than in the TL crack system (181.03 J/m^2). The results show that the specific fracture energy of SYP in the TR crack system was about 65 % higher than that in the TL crack system, while in the case of RO the difference was about 50 %. A similar result was also reported by Watanabe *et al*. (2011). They researched fracture behavior of sugi (Cryptomeria japonica) in the TR, TL, and intermediate crack systems. They stated that the fracture energy, which means the area under the load-deformation curve in the TR crack system, was more than twice that in the TL and intermediate systems. It means that the crack growing in the radial direction needed more energy per unit area to separate a wood sample into two halves than in the longitudinal direction.

The initial slope is characteristic for the elastic properties and proportional to an effective modulus of elasticity (Harmuth *et al*., 1996; Reiterer *et al*., 2002). The initial slope of the TL specimen was higher than that of the TR specimen. The RO shows higher initial slopes than SYP in both systems. The initial slope results for both wood species indicate that the modulus of elasticity would be expected to be higher under Mode I loading in the TL crack system than in the TR. Ductility increased with increasing both the dissipated energy during the crack initiation and the crack propagation (Reiterer and Tschegg, 2002). As all CT test specimens in this study had the same dimensions, a brittleness parameter was used to characterize whether the material behavior was more ductile or brittle. The parameter was determined from the load-deformation curves using the failure load, initial slope, and specific fracture energy (Reiterer *et al*. 2002; Tschegg *et al*. 2001). It was calculated using Eq. 4:

$$B = \frac{P_{max}^2}{L \cdot k_{init} \cdot G_f} \quad (4)$$

Where P_{max} is the maximum force (N), L is the ligament length, k_{init} is the initial slope of a tested specimen (N/mm), G_f is the specific fracture energy (J/m^2). According to the parameter, a lower value indicates that the material behavior is more ductile, whereas a higher value refers to the material behavior as more brittle. The obtained values clearly show that RO in both crack systems was more brittle than SYP. In general, the TR crack system showed a more brittle behavior than the TL crack system.

Differences in the fracture behavior depending on grain orientation and wood species could be explained by structural features of the tested wood species. Konukcu *et al*. (2021) mentioned that Mode I fracture behavior of a tested specimen can be affected by not only its density but also by its microstructure. The fracture toughness (or failure load) and the specific fracture energy of the TR were larger than those of the TL. This is because the TR specimens were loaded in a tangential direction, while the crack propagated in a radial direction. Therefore, the crack progress in the TR specimen crossed the annual ring and wood fibers could bridge the crack surface. On the other hand, the crack process of TL was small because all the cracks were growing along the longitudinal direction of the CT test specimen. Schniewind and Pozniak (1971) explained that the crack in the TL system can run along the cell axis where the only cell ends and ray cells crossings provide for temporary arrest of crack growth, whereas the crack in the TR system can grow perpendicular to the cells. Kretschmann (2010) also mentioned that the TL is one of the predominant crack systems because of the low strength and stiffness of wood perpendicular to the grain. The difference between SYP and RO could be explained by the fact that RO is denser than SYP because previous studies show that the fracture toughness values of wood were increased with the increase of its density (Conrad *et al*., 2003; Kretschmann *et al*., 1991; Petterson and Bodig, 1983; Schniewind *et al*., 1982).

4 CONCLUSIONS
4. ZAKLJUČAK

In this study, the fracture behavior of SYP and RO was investigated in the TR and TL crack propagation systems using the CT test method in Mode I. The results show that, in general, the RO had a significantly different fracture behavior than SYP for each of the two crack propagation systems. The fracture toughness indicating the resistance against crack initiation was higher in the TR crack propagation system than that in the TL crack system, but there was no significant difference between the two crack propagation systems for SYP. The initial slope indicating the stiffness was higher in the TL than in the TR. However, the specific fracture energy of the TL was significantly lower than that of the TR. It means that more energy per unit area was needed for the TR to separate a wood sample into two halves. It was also found that the behavior of wood in the TR crack system became more brittle than in the TL. Differences in the fracture behavior of wood depending on the crack propagation systems could be explained by structural features of the tested samples because the crack propagation in the TR system crosses the annual ring and wood fibers can bridge the crack surface; however, the crack in the TL system would run along the longitudinal direction of the wood.

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Optimization for Green Path in Wood Extractives by Taguchi Analysis

Primjena Taguchijeve analize za optimizaciju procesa ekstrakcije drva na načelima zelene kemije

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ABSTRACT • *Extractive composition of Pinus pinaster (Maritime pine) wood was studied with choline chloride based deep eutectic solvents (DES). Two different eutectic mixtures (ChCl: Et-Gly; ChCl:Urea 1:2 molar ratio), two different extraction methods (hot water bath, ultrasound assisted extraction), and temperature (40 – 60 °C), time (30 – 60 min.) and solid:liquid ratio (1:10 – 1:20 g/mL) parameters were applied. For the optimization of operation conditions, Taguchi analysis was performed. Resin acids formed the chemical composition of Pinus pinaster (Maritime pine) wood. Dehydroabietic acid, abietic acid and isopimaric acid were found to be the major compounds. The impact of parameters on the performance of the system was determined as follows: DES type > solid:liquid ratio > extraction method > temperature > time. So, for the extraction of lipophilic compounds in Maritime pine wood, the optimum conditions were determined as hot water bath extraction at 60 °C with ethylene glycol and 1:10 solid liquid ratio for 60 min.*

KEYWORDS: *deep eutectic solvent; Pinus pinaster; resin acids; choline chloride; ethylene glycol; urea; Taguchi analysis*

SAŽETAK • *U radu su istražene ekstraktivne tvari drva primorskog bora (Pinus pinaster) izolirane primjenom eutektičkog otapala (DES) na bazi kolin klorida. Pritom su primijenjene dvije eutektičke mješavine (ChCl: Et-Gly i ChCl: Urea, molarni omjer 1:2) i dvije metode ekstrakcije (vrućom vodom i uz primjenu ultrazvuka), pri temperaturi 40 i 60 °C i s vremenom ekstrakcije od 30 i 60 minuta, uz varijabilni odnos kruto – tekuće 1 : 10 i 1 : 20 g/mL. Za optimizaciju procesa ekstrakcije primijenjena je Taguchijeva analiza. Rezultati istraživanja pokazuju da se ekstraktivne tvari drva primorskog bora (Pinus pinaster) uglavnom sastoje od smolnih kiselina s dehidroabietinskom, abietinskom i izopimarnom kiselinom kao glavnim spojevima. Usto je utvrđen i utjecaj pojedinog parametra na efikasnost procesa ekstrakcije kako slijedi (rangirano od onoga s najvećim prema onome s najmanjim utjecajem): vrsta eutektičkog otapala > odnos kruto – tekuće > metoda ekstrakcije > temperatura ekstrakcije > vrijeme ekstrakcije. Na temelju analize dobivenih podataka određeni su optimalni parametri za ekstrakciju drva primorskog bora. To su: ekstrakcija vrućom vodom pri 60 °C, uz upotrebu etilen glikola i omjer kruto – tekuće 1:10 tijekom 60 minuta.*

KLJUČNE RIJEČI: *eutektička otapala; Pinus pinaster; smolne kiseline; kolin klorid; etilen glikol; urea; Taguchijeva analiza*

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1 INTRODUCTION
1. UVOD

In the concept of "green chemistry", reduction of unsafety and petroleum based solvent use in industry becomes a priority for EU between 2010-2050 (Bubalo *et al.,* 2015). Different solvents, e.g. supercritical-subcritical solvents, ionic liquids (ILs), deep eutectic solvents (DES), low-melting mixtures (LMMs), are classified as safe and non-hazardous solvents (Fischer, 2015).

DES, consisting of two or more compounds, are formed from hydrogen bond donors (HBD) and hydrogen bond acceptors (HBA). DES are negligibly volatile, cheap, non-toxic, low-flammable and thermally stable, often biodegradable, and not requiring purification (Ozturk *et al.,* 2018a). They are classified into four groups: Type I (organic salts + metal salts), Type II (organic salts + metal hydrates), Type III (organic salts + HBD) and Type IV (metal chlorides + HBD). Type III deep eutectic solvents, used in this study, are applied in fractionation of lignocellulosic biomass, biodiesel production, metal processing and extraction of polar molecules and bioactive compounds. They are also used in pharmaceutical and biomedical applications (Ozturk *et al*., 2018a; Cao *et al*., 2018; Meng *et al*., 2018; Zdanowicz *et al*., 2018; Barbieria *et al*., 2020). Choline choride (ChCl), a non-toxic and biodegrable compound, is mostly used in the DES mixtures as a HBD. With these features, ChCl based DES solvents are convenient for the pharmaceutical and cosmetic use (Häkkinen, 2020).

Extractives, defined as low molecular compounds in the woody plants, are composed of different chemical substances. These compounds can be classified into two groups - lipophilics and hydrophilics (Sjöström, 1981). For the extraction of lipophilic compounds, non-polar solvents are used, while polar solvents are used for hydrophilics (Vek *et al*., 2020). Although the amount of extractives is less than 10 % of dry wood, they are used in different areas, e.g lipophilics in pharmacy, food and cosmetic industries. Hydrophilic compounds have antioxidant, antimicrobial, antiviral, cancerogenic and cardio protective effects (Fengel and Wegener, 2003; Benouadah *et al*., 2018). Because of these features, extractives are becoming increasingly important. The use of non-toxic, biodegradable chemicals has also become popular in the last years.

Pinus pinaster Aiton occurs naturally in Southwest Europe (e.g Spain, Portugal), Western Mediterranean and Northwest Africa as a fast growing species. This species was first planted in 1881 in different regions of Turkey and today it covers a total of 57,378.4 ha (Velioglu *et al*., 2020; Koch, 1972).

It is mainly used in pulp and paper industry both in Turkey and in Europe. It is also used in particleboard and packaging industries. Due to its high resin content, special resin production sites have been created for *Pinus pinaster* in different countries (e.g Portugal, Spain, France, Italy). The yield of resin is 1457-2500 g/tree with acid-paste method in Turkey (Aydin, 2017). It is reported that the turpentine part of this resin is used as antiseptic, diuretic and anthelmintic. Also, local people use the cone extracts of this species to prevent bronchitis and cough (Kurtca and Tumen, 2020). There are several studies regarding the extractives of *Pinus pinaster.* In the wood part, simple phenolics, stilbenes, lignans, flavonoids, organic acids, steryl esters and triglycerides are found to be the major extracts in the hot water aliquot (Conde *et al*., 2014). However, in the bark part, diterpenic compounds that have nutraceutical effects are found to be the main compounds with fatty acids, long-chain alcohols and sterols (Sousa *et al*., 2018).

In this study, a new generation of choline chloride based on two different deep eutectic solvents was used to determine the extractive composition of *Pinus pinaster* wood. Different temperatures (40-60 °C), time (30-60 min.), solid:liquid ratio (1:10-1:20 g/mL) and extraction methods (hot water bath- ultrasound assisted extraction) were used. For the optimization of operation conditions, Taguchi method was performed. Although there are studies about the phenolic compositions of wood with deep eutectic solvents, lipophlics are studied for the first time.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijal

In this study, *Pinus pinaster,* obtained from Zonguldak –Turkey, was used as wood material. Bark was removed before chipping the woody parts into small matchstick size. Samples were grounded in a Wiley mill and freeze-dried before the extraction. Choline chloride, urea and ethylene glycol were purchased from Merck in analytical grades and used without further purification.

2.2 Deep eutectic solvents (DES) preparation
2.2. Priprema eutektičkih otapala (DES)

Two different DES mixtures were prepared. Molar ratios are listed in Table 1. Mixing procedure was performed at 80 °C until a homogenous clear liquid was obtained.

2.3 Extraction methods
2.3. Metode ekstrakcije

Two different extraction procedures were applied. As a control, sequential extraction with n-hexane and

Table 1 Molar ratios of deep eutectic solvents
Tablica 1. Molarni odnosi eutektičkih otapala

HBA	HBD	Abbreviation *Skraćenica*	Molar ratio (wt/wt) *Molarni omjer* (m/m)
Choline chloride / *kolin klorid*	Urea / *urea*	ChCl : Ur	1 : 2
Choline chloride / *kolin klorid*	Ethylene glycol / *etilen glikol*	ChCl : Et-Gly	1 : 2

acetone:water (95:5 v/v) was carried out for 6 hours in a soxhlet apparatus. With DES mixtures, hot water extraction (HW) and ultrasound assisted extraction (UAE) were performed. Wood samples and DES were put in a test tube and extracted according to the below conditions. After each extraction method, samples were centrifuged for 15 min. at 4000 rpm. 1 mL of aliquot was taken to a new test tube and extracted (liquid-liquid) with acetone before silylation. For the optimization of extraction, some parameters were tested: type of DES (Table 1), solid:liquid ratio (1:10 -1:20 g/mL), temperature (40 – 60 °C) and extraction time (30 – 60 min.). Three repetitions were made for each parameter.

2.4 FID-GC and GC-MS analysis
2.4. Analiza FID-GC i GC-MS

Shimadzu GCMS-QP2010 GC-MS equipped with TRB-5MS column (30 m × 0.25 mm (0.25 μm thickness)) was used for the identification. Temperature program was 120 °C for 1 min. then raised to 310 °C with a 6 °C/min. waiting for 20 minutes. The injection temperature was 260 °C, split ratio was 1:25, ion source was 200 °C and ionization energy 70eV. Wiley and NIST libraries were used. For quantitative analysis, Shimadzu GC 2010 FID-GC was used.

2.5 Taguchi Design
2.5. Dizajn Taguchijeve metode

Taguchi is an effective statistical method to optimize the operation condition settings. With this method, the number of experiments was reduced and so were the costs and time (Kumar *et al*., 2015; Uslu and Aydin, 2020). The method consists of an orthogonal array, signal-to-noise ratio (S/N or SNR), response table and graph (Main Effect Analysis) (Ozakin and Kaya, 2020). To start with Taguchi, operation parameters, quality characteristics and orthogonal array are selected for designing and doing the experiments. Then, the results are analyzed by using signal-to-noise ratio (S/N). Finally, optimum parameters are obtained with the analysis results (Sun *et al*., 2013; Liu *et al*., 2019).

2.5.1 Selection of parameter levels and orthogonal array of Taguchi
2.5.1. Odabir razina parametara i ortogonalni niz Taguchijeve metode

The parameters, affecting the amount of selected compounds (abietic acid, dehydroabietic acid and isopimaric acid, the most abundant compounds), were determined as extraction method, extraction temperature, extraction time and solid/liquid ratio. Table 2 illustrates the factors considered and their levels.

Table 2 Parameters and their levels
Tablica 2. Parametri i njihove razine

Codes *Oznake*	Parameters *Parametri*	Levels / *Razine*	
		1	2
A	Method / *metoda*	HW	UAE
B	Time, min / *vrijeme,* min	30	60
C	Temperature, °C *temperatura, °C*	40	60
D	DES	Et-Gly	Ur
E	Solid/Liquid ratio, g/mL *odnos kruto – tekuće,* g/mL	10	20

According to the number of selected parameters and their levels, the L_{32} (2^5) orthogonal array of Taguchi was selected, as shown in Table 3. The main feature of the orthogonal indices is that all the factors are included in the experiment with an equal number of trials.

2.5.2 Signal to noise ratio
2.5.2. Omjer signala i šuma

In the Taguchi method, generally, the S/N is adopted as the indicator of quality (Jiang *et al*., 2020). It is defined as undesired random noise value, desirable signal ratio and shows the quality characteristics of experimental data (Kurt *et. al*., 2009; Gunay *et al*., 2011; Gunay and Yucel, 2013). Depending on the particular characteristics of the design problem, different S/N ratios may be applicable, including “lower is better”, “nominal is best”, or “higher is better” (Chen *et al*., 2007; Kurt and Can, 2021). In this study, for the calculation of S/N ratio “higher-is-better” performance character was preferred as shown in Eq. 1.

$$S/N = -10\log\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right) \quad (1)$$

n is the number of observations of the experiment and y_i is the observed data at the i^{th} experiment (Taguchi *et al*., 2005).

2.5.3 Grey relations analysis
2.5.3. Siva relacijska analiza

In order to verify the Taguchi results and specify the best experiment conditions, Grey relation analysis (GRA) was done. GRA is a part of a system theory, improved for solving complex relationships between alternatives and variables (Yang and Chen, 2006). In case of many criteria and alternatives, this method is

Table 3 Orthogonal array of Taguchi L_{32} (2^5)
Tablica 3. Ortogonalni niz Taguchijeve metode L_{32} (2^5)

Experiment No *Broj eksperimenta*	**A**	**B**	**C**	**D**	**E**	**Experiment No** *Broj eksperimenta*	**A**	**B**	**C**	**D**	**E**
1	1	1	1	1	1	17	2	1	1	1	1
2	1	1	1	1	2	18	2	1	1	1	2
3	1	1	1	2	1	19	2	1	1	2	1
4	1	1	1	2	2	20	2	1	1	2	2
5	1	1	2	1	1	21	2	1	2	1	1
6	1	1	2	1	2	22	2	1	2	1	2
7	1	1	2	2	1	23	2	1	2	2	1
8	1	1	2	2	2	24	2	1	2	2	2
9	1	2	1	1	1	25	2	2	1	1	1
10	1	2	1	1	2	26	2	2	1	1	2
11	1	2	1	2	1	27	2	2	1	2	1
12	1	2	1	2	2	28	2	2	1	2	2
13	1	2	2	1	1	29	2	2	2	1	1
14	1	2	2	1	2	30	2	2	2	1	2
15	1	2	2	2	1	31	2	2	2	2	1
16	1	2	2	2	2	32	2	2	2	2	2

commonly used for alignment or streaming of options and choice between alternatives. GRA consist of following steps (Tosun, 2005; Haq *et. al*., 2008; Shi *et. al*., 2015; Panda *et. al*., 2016).

Step 1. Form the decision matrix and uniform the data in order to prohibit unit variations. It is actually necessary because variations between data can be different. Reproduce a value to form the array between 0 to 1 from original value. Three different equation "higher is better", "lower is better" and "nominal is best" are used according to the problem in the normalization process. In this study, "higher-is-better" performance character was preferred as shown in Eq. 2.

$$x_i^*(k) = \frac{x_i^0(k) - min\, x_i^0(k)}{max\, x_i^0(k) - min\, x_i^0(k)} \tag{2}$$

Where $x_i^*(k)$ specifies the sequence after data preprocessing, $x_i^0(k)$ is the measured results, $min\, x_i^0(k)$ is the minimum value $x_i^*(k)$, and $max\, x_i^0(k)$ is the maximum value of $x_i^0(k)$, i is the number of experiments, and k represents the measurement values.

Step 2. Calculation of grey relational coefficient:

$$\xi_i(k) = \frac{\Delta_{min} - \xi\Delta_{max}}{\Delta_{0i}(k) + \xi\Delta_{max}} \tag{3}$$

Where, Δ_{0i} is the deviation sequence of the reference sequence and comparability sequence, Δ_{min} is the minimum value in the sequence, Δ_{max} is the maximum value in the sequence. ξ is defined as identification coefficient and the range is between 0 to 1. Generally, the value of ξ is taken as 0.5.

Step 3. Calculation of grey relational grade is defined as final step. It is calculated according to Eq. 4 averaging the sum of the grey relational coefficients

$$\gamma_i = \frac{1}{n}\sum_{k=1}^{n}\xi_i(k) \tag{4}$$

Where γ_i changes in the range of 0 to 1, and n is the number of experiments. The higher grey relational grade signifies more ideal results.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Chemical composition
3.1. Kemijski sastav

The amount of lipophilic compounds after the sequential extraction with n-hexane and acetone are given in Table 4. As seen, it is mainly formed from resin acids and the main compounds are abietic, dehydroabietic and levopimaric acids. In addition, oxidized resin acids (6 %) were found in the n-hexane extract.

Two types of resin acids (Abietane-type and pimarane-type) were found in the DES extracts of *P. pinaster* wood. Compared to organic solvent (eg. acetone), no fatty acids were found in the DES extracts. Also, hydroxy resin acids were not seen. In between all DES types and all experimental conditions, dehydroabietic acid (18-37 %) and abietic acid (14-28 %) were found to be the major compounds (Table 5 and Table 6). Isopimaric acid, a pimarane type, was determined as 7.3-15 % in both DES. However, the highest amount was obtained with ChCl:Et-Gly in HW at 60 °C from 1 g of *P. pinaster* wood. With the organic solvent (n-hexane), the amount of isopimaric acid was 7.7 %.

PSMME (pinosylvin monomethyl ether) a typical stilbene for Pinus species was not found in ultrason bath extraction with ChCl:Ur (Table 6), whereas in hot water extraction (HW) the amount of PSMME was 0.4-2.4 %. ChCl:Ur is more viscous and it is difficult to handle during silylation.

Table 4 Amount of lipophilics extracted with organic solvents from *P.pinaster* wood (%)
Tablica 4. Udio lipofilnih spojeva dobivenih ekstrakcijom iz drva primorskog bora (*Pinus pinaster*) organskim otapalima (%)

No *Broj*	**RT** *Retencijsko vrijeme*	**Compounds** *Spojevi*	**n-Hexane** *n-heksan*	**Acetone** *Aceton*
1	18.274	16:00	0.4	-
2	20.925	linoleic (18 : 2) / *linolna kiselina (18 : 2)*	0.7	11.4
3	20.994	oleic (18:3) / *oleinska kiselina (18 : 3)*	1.1	38.8
4	21.36	18 : 0	-	6.8
5	22.343	PSMME	0.6	25
6	22.658	pimaric acid / *pimarna kiselina*	8.7	-
7	22.889	sandracopimaric acid / *sandrakopimarna kiselina*	1.4	-
8	23.087	isopimaric acid / *izopimarna kiselina*	7.7	-
9	23.370	palustric acid / *palustrinska kiselina*	10.2	-
10	23.702	levopimaric acid / *levopimarna kiselina*	15.9	-
11	23.810	dehydroabietic acid / *dehidroabietinska kiselina*	17.3	11.5
12	24.245	abietic acid / *abietinska kiselina*	18.2	6.7
13	25.622	neoabietic acid / *neoabietinska kiselina*	11.8	-
14	26.545	hydroxy-resin1 / *hidroksi-smola 1*	1.8	-
15	26.866	hydroxy-resin2 / *hidroksi-smola 2*	2.1	-
16	27.265	hydroxy-resin3 / *hidroksi-smola 3*	2.1	-

Table 5 Amount of lipophilics extracted from *P. pinaster* wood with ChCl:Et-Gly (%)
Tablica 5. Udio lipofilnih spojeva dobivenih ekstrakcijom iz drva primorskog bora (*Pinus pinaster*) smjesom ChCl : Et-Gly (%)

Solid / Liquid ratio *Odnos kruto – tekuće*	**Compounds** *Spojevi*	**UAE**				**HW**			
		40 °C		**60 °C**		**40 °C**		**60 °C**	
		30 min	60 min	30 min	60 min	30 min	60 min	30 min	60 min
1 : 10, g/mL	18 : 00	4.0±2.7	3.1±0.3	2.0±0.2	2.4±0.3	3.3±1.0	6.5±0.2	2.5±0.8	5.0±1.2
	PSMME	1.2±0.5	1.2±1.0	3.3±1.0	2.3±2.4	3.8±2.8	1.3±0.3	4.0±1.8	2.1±1.0
	Pimaric acid / *pimarna kiselina*	10±0.8	9.6±0.6	9.2±0.6	9.6±1.0	9.2±1.7	8.5±0.0	9.3±2.1	8.0±0.1
	Pimaric acid / *pimarna kiselina*	1.3±0.1	1.4±0.2	1.3±0.5	1.5±0.1	1.3±0.2	1.0±0.1	1.3±0.1	1.7±0.3
	Isopimaric acid *izopimarna kiselina*	11±0.1	10±0.1	10±0.6	10±0.2	10±1.3	11±0.6	10±1.1	15±1.9
	Palustric acid *palustrinska kiselina*	12±0.7	12±0.4	8.9±1.6	11±2.0	10±2.2	12±1.7	9.7±2.8	13±1.3
	Levopimaric acid *levopimarna kiselina*	9.8±0.5	10±1.7	3.8±2.1	9.7±1.4	5.0±4.1	7.6±0.8	4.2±4.4	3.2±0.4
	Dehydroabietic acid *dehidroabietinska kiselina*	29±0.5	27±2.7	38±0.6	28±3.2	33±2.5	29±0.1	35 ±3.6	31±1.5
	Abietic acid *abietinska kiselina*	17±1.6	18±0.3	18±2.3	19±0.3	18±3.1	19±0.6	18 ±3.0	14±1.1
	Neoabietic acid *neoabietinska kiselina*	5.3±2.3	6.6±1.5	5.1±0.2	6.2±1.0	5.7±0.2	5.3±1.4	4.8 ±1.2	9.6±5.0
1 : 20, g/mL	18 : 00	3.2±0.8	2.6±0.1	3.1±0.4	3.1±0.5	2.8±0.0	5.1±2.2	2.4±0.4	8.0±0.7
	PSMME	4.6±0.3	2.1±0.3	3.8±0.9	1.7±0.4	5.5±1.5	2.9±0.6	3.3±0.3	1.9±0.4
	Pimaric acid / *pimarna kiselina*	11±0.6	12±0.2	10±0.5	12±0.0	8.2±2.0	9.0±2.1	11±1.1	7.9±0.4
	Pimaric acid / *pimarna kiselina*	1.2±0.1	1.4±0.2	1.2±0.1	1.4±0.2	1.2±0.3	1.3±0.0	1.4±0.2	1.4±0.1
	Isopimaric acid *izopimarna kiselina*	9.4±0.6	11±0.0	9.8±1.2	11±0.0	9.6±2.0	9.7±1.2	10±0.2	9.5±0.3
	Palustric acid *palustrinska kiselina*	10±0.5	12±0.3	9.7±0.4	12±0.4	11±2.2	12±0.1	11±0.5	11±0.1
	Levopimaric acid *levopimarna kiselina*	6.4±1.5	9.1±0.6	5.7±0.9	8.6±0.7	5.7±0.8	5.6±2.4	6.4±0.6	6.2±1.3
	Dehydroabietic acid *dehidroabietinska kiselina*	36±3.1	29±1.4	37±2.3	29±0.8	33 ±4.0	30±3.5	34±2.0	28±0.4
	Abietic acid *abietinska kiselina*	14±0.5	16±0.1	16±0.4	17±0.5	17 ±0.4	18±3.2	16±0.4	19±1.0
	Neoabietic acid *neoabietinska kiselina*	4.1±0.1	4.7±0.2	3.9±0.2	4.0±0.3	6.1±1.5	6.0±3.0	3.7±0.3	6.9±0.6

Table 6 Amount of lipophilics extracted from *P.pinaster* wood with ChCl:Ur (%)
Tablica 6. Udio lipofilnih spojeva dobivenih ekstrakcijom iz drva primorskog bora (*Pinus pinaster*) smjesom ChCl : Ur (%)

Solid / Liquid ratio *Odnos kruto – tekuće*	**Compounds** *Spojevi*	**UAE**				**HW**			
		40 °C		**60 °C**		**40 °C**		**60 °C**	
		30 min	60 min	30 min	60 min	30 min	60 min	30 min	60 min
1 : 10, g/mL	18 : 00	9.0±2.3	4.1±0.7	9.4±1.5	2.9±0.1	5.5±2.7	4.7±0.4	6±3.4	5.2±0.9
	PSMME	-	-	-	-	0.9±0.1	1.2±0.0	1.6±0.5	2.4±1.0
	Pimaric acid / *pimarna kiselina*	7.0±0.0	7.2±0.0	7.8±0.4	7.7±0.0	7.9±0.5	8.1±0.2	7.7±0.7	6.0±0.6
	Pimaric acid / *pimarna kiselina*	0.9±1.3	0.7±1.0	1.4±0.1	1.4±0.0	1.5±0.1	1.5±0.0	1.4±0.1	1.2±0.0
	Isopimaric acid *izopimarna kiselina*	8.4±0.3	8.3±0.6	8.8±0.1	8.9±0.3	9.5±0.2	9.3±0.5	9.3±0.7	8.8±1.0
	Palustric acid *palustrinska kiselina*	9.9±0.1	9.7±1.7	9.0±0.5	10±0.1	7.2±0.1	8.1±1.2	8.7±1.2	11±0.5
	Levopimaric acid *levopimarna kiselina*	9.5±1.7	12±1.0	10±1.0	11±0.0	±7.9±0.3	7.8±0.6	8.6±1.3	7.2±1.6
	Dehydroabietic acid *dehidroabietinska kiselina*	25±1.5	27±0.8	24±1.0	26±0.9	29±1.9	30±0.2	27±3.0	28±2.6
	Abietic acid *abietinska kiselina*	20±1.1	19±0.5	18±0.3	20±0.1	21±0.3	21±0.5	20±1.4	19±0.5
	Neoabietic acid *neoabietinska kiselina*	9.9±0.5	12±1.6	10±0.3	11±0.1	9.1±0.3	8.7±1.0	9.7±0.0	11±2.6
1 : 20, g/mL	18 :00	9.2±3.0	9.8±1.4	9.0±5.2	7.7±2.3	29±2.9	25±1.3	24±0.2	28±4.1
	PSMME	-	-	-	-	0.4±0.0	0.6±0.2	0.9±0.2	1.0±0.1
	Pimaric acid / *pimarna kiselina*	6.2±0.8	7.9±0.1	7.0±1.3	8.1±0.2	3.6±3.5	6.3±0.3	5.9±0.6	5.1±1.1
	Pimaric acid / *pimarna kiselina*	0.9±0.5	1.4±0.0	1.4±0.0	1.6±0.3	4.9±5.4	1.5±0.2	1.0±0.1	1.0±0.0
	Isopimaric acid izopimarna kiselina	8.6±0.3	9.8±0.5	9.3±0.2	8.8±0.7	7.3±0.1	8.8±0.7	11±1.6	12±0.5
	Palustric acid *palustrinska kiselina*	8.4±1.2	9.5±0.5	9.6±0.8	9.1±0.9	5.9±0.3	7.2±1.2	6.6±0.4	7.1±0.2
	Levopimaric acid *levopimarna kiselina*	8.7±0.1	9.0±3.0	12±0.9	12±0.7	12±6.0	6.3±0.9	6.9±0.9	6.6±0.1
	Dehydroabietic acid *dehidroabietinska kiselina*	23±0.8	24±2.3	21±0.8	22±1.5	19±3.	19±0.6	20±0.6	18±2.1
	Abietic acid *abietinska kiselina*	28±1.7	19±0.0	20±0.4	20±0.6	11±6.0	18±1.5	18±0.9	16±0.5
	Neoabietic acid *neoabietinska kiselina*	7.4±1.3	9.1±0.7	11±0.9	11±0.3	6.9±0.3	7.9±0.5	6.2±0.8	4.8± 2

The effects of experimental conditions, e.g. time and temperature, on the main compounds like dehydroabietic, abietic and isopimaric acids are shown in Figure 1 and 2. As seen, the amount of the main compounds are higher with ChCl:Et-Gly than with ChCl:Ur. At the optimum conditions discussed in Taguchi analysis (solid:liquid ratio of 1:10, extraction temperature: 60 °C, extraction time: 60 min, extraction method HW), dehydroabietic acid was found to be (31.1 ± 1.5) % with ChCl:Et-Gly and (28±3) % with ChCl:Ur. The second important compound, abietic acid, was (16.2±1.1) % and (20±0.5) % in ChCl:Et-Gly and ChCl:Ur, respectively.

Temperature is another factor affecting the efficiency of extraction. Increasing the temperature from 40 to 60 °C, changed the amount of dehydroabietic acid from (28.7±0.1) % to (31.1±1.5) %. For isopimaric acid, this amount is (10.8±0.6) % to (13.1±1.9) % respectively. Increasing the temperature, decreased the solvent viscosity and mass transfer limitations, but increased the diffusivity. Similar results were obtained by Ozturk *et al*. (2018b) for polyphenolics from orange peel.

Solid:liquid ratio is an important parameter for the operation. The increase of the amount of liquid increases positive interaction between the solid and liquid states. Two different ratios were applied in this study (1:10 and 1:20 g/mL). Changing the liquid ratio from 10 mL to 20 mL, decreased the amount of dehydroabietic acid (31.1±1.5 – 28±0.4 %). Similar decline was observed for isopimaric acid (13.1±1.9 – 9.5±0.3 %). However, for abietic acid, increasing the liquid ratio positively affects the amount (16.2±1.1 – 18.7±1.0 %).

As seen in Figure 1, at the optimum conditions, the amount of dehydroabietic was found to be (31.1±1.5) % with hot water bath and (28±3.2) % with UAE. To the contrary, the amount of abietic acid was found low with HW. UAE is a simple method that requires less time and solvent. In the recent studies, UAE

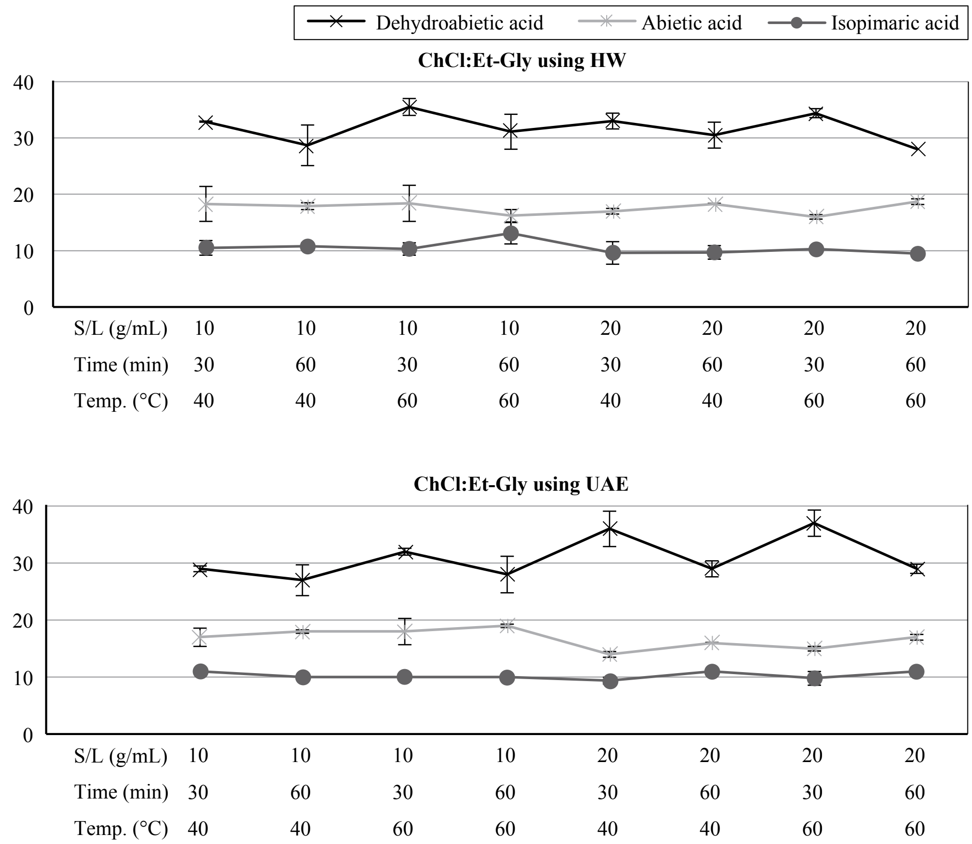

Figure 1 The amount of main compounds extracted with ChCl:Et-Gly using HW and UAE methods
Slika 1. Udjeli osnovnih spojeva ekstrahiranih smjesom ChCl: Et-Gly uz primjenu vruće vode i ultrazvuka

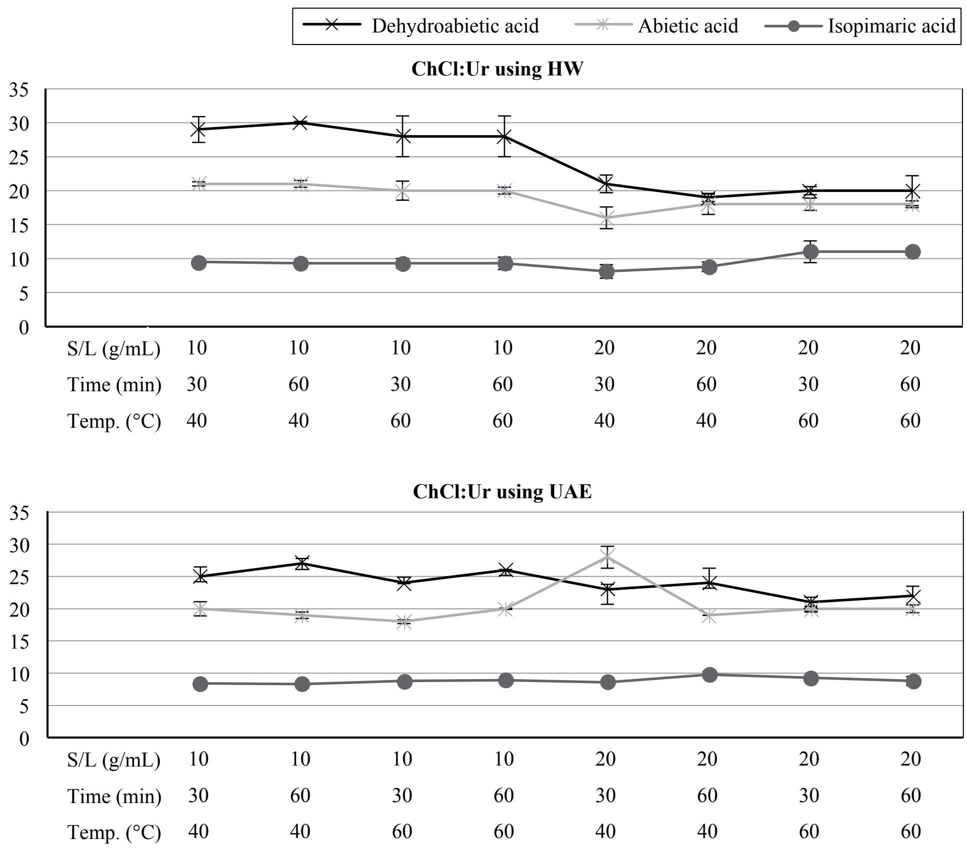

Figure 2 The amount of main compounds extracted with ChCl:Ur using HW and UAE methods
Slika 2. Udjeli osnovnih spojeva ekstrahiranih smjesom ChCl : Ur uz primjenu vruće vode i ultrazvuka

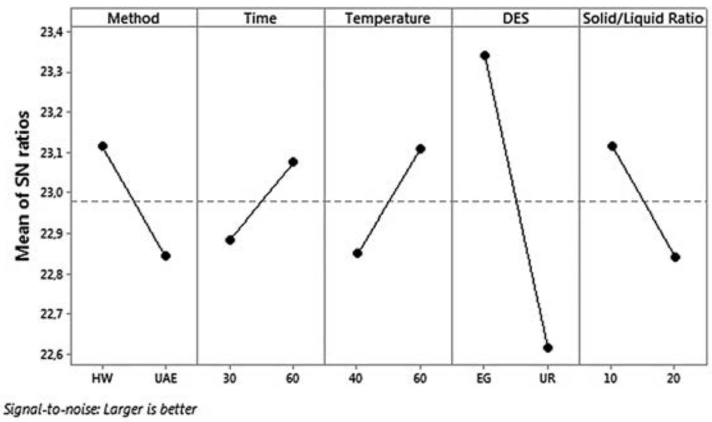

Figure 3 Main effect plots of S/N ratio
Slika 3. Dijagrami glavnog učinka omjera S/N

Table 7 S/N ratios of factor levels for experimental parameter
Tablica 7. Omjeri signala i šuma (S/N) razina faktora za eksperimentalne parametre

Level *Razina*	**A**	**B**	**C**	**D**	**E**
1	23.11	22.88	22.85	23.34	23.12
2	22.84	23.07	23.11	22.62	22.84
Delta	0.27	0.19	0.26	0.73	0.27
Rank *Rang*	3	5	4	1	2

and microwave extraction (MW) have started to be applied to biomass, however, mainly using water or oil baths (Skulcova *et.al*., 2018; Li *et.al*., 2017; Chen and Wan, 2018; Gülsoy and Kilic-Pekgözlü, 2021).

3.2 Taguchi analysis
3.2. Taguchijeva analiza

S/N ratios related to control parameters obtained from Taguchi analysis are given in Table 7. The highest S/N of each parameter indicates the optimal level of that parameter. For example, as seen in Table 7, the 23.11 S/N ratio value in the method factor is defined as level 1 and shows that HW is the method with the best results. Likewise, with the 23.24 S/N ratio value, Et-Gly is the best DES solvent. Overall, among all parameters, the most effective factor was DES solvent type.

As seen in Figure 3. the greatest S/N ratio ensures the best levels of experimental parameters. The optimal factors for this study were determined as A: Hot water bath extraction; B: 60 min.; C: 60 °C; D: Ethylene glycol; E: 1:10 solid:liquid ratio.

Table 8 Values of Grey relational grade
Tablica 8. Vrijednosti ocjena sive relacijske analize

No *Broj*	**A**	**B**	**C**	**D**	**E**	**Grey Grade** *Ocjena prema sivoj relacijskoj analizi*	**Rank** *Rang*	**No** *Broj*	**A**	**B**	**C**	**D**	**E**	**Grey Grade** *Ocjena prema sivoj relacijskoj analizi*	**Rank** *Rang*
1	1	1	1	1	1	0.5321	7	17	2	1	1	1	1	0.4873	11
2	1	1	1	1	2	0.4993	10	18	2	1	1	1	2	0.5455	5
3	1	1	1	2	1	0.4797	16	19	2	1	1	2	1	0.5003	9
4	1	1	1	2	2	0.3539	32	20	2	1	1	2	2	0.5828	4
5	1	1	2	1	1	0.5835	3	21	2	1	2	1	1	0.5004	8
6	1	1	2	1	2	0.5387	6	22	2	1	2	1	2	0.5937	2
7	1	1	2	2	1	0.4545	19	23	2	1	2	2	1	0.3962	30
8	1	1	2	2	2	0.4338	23	24	2	1	2	2	2	0.4078	28
9	1	2	1	1	1	0.4835	14	25	2	2	1	1	1	0.4440	22
10	1	2	1	1	2	0.4745	17	26	2	2	1	1	2	0.4804	15
11	1	2	1	2	1	0.4864	13	27	2	2	1	2	1	0.4179	27
12	1	2	1	2	2	0.3709	31	28	2	2	1	2	2	0.4259	26
13	1	2	2	1	1	0.6588	1	29	2	2	2	1	1	0.4613	18
14	1	2	2	1	2	0.4464	21	30	2	2	2	1	2	0.4873	11
15	1	2	2	2	1	0.4545	19	31	2	2	2	2	1	0.4299	25
16	1	2	2	2	2	0.4338	23	32	2	2	2	2	2	0.4031	29

3.3 Grey relations analysis
3.3. Siva relacijska analiza

Table 8 shows grey relational grade values obtained by using Eqs. 2, 3 and 4. As seen from the table, the optimum parameters were observed in the experiment number 13, which has the highest grey grade value (0.6588). These parameters belong to the combination of experiments, as obtained in the Taguchi analysis. A: Hot water bath extraction; B: 60 min.; C: 60 °C; D: Ethylene glycol; E:1:10 solid:liquid ratio (g/mL).

4 CONCLUSIONS
4. ZAKLJUČAK

Extraction of lipophilic compounds, e.g fatty and resin acids which have antimicrobial and antifungal activities, was investigated with deep eutectic solvents. Choline chloride based on two different eutectic mixture urea (1:2) and ethylene glycol (1:2) was used. Ten different compounds, mainly resin acids, were identified in the DES mixtures with GC-MS. Dehydroabietic acid, abietic acid, isopimaric acid and palustric acid were found to be the major compounds. L_{32} orthogonal array from Taguchi was applied for optimization. Extraction method, extraction time, extraction temperature, solid:liquid ratio and DES type were the main parameters.

The sequence of individual parameters in this study is ranked as follows: DES type > solid:liquid ratio > extraction method > temperature > time. Ethylene glycol was found to be more effective compared to urea to extract the lipophilic compounds from *Pinus pinaster* wood. 1 g of wood meal and 10 mL of DES mixture (solid:liquid ratio) were found to be sufficient. Hot water extraction at 60 °C for 60 min. are the optimum factors for this study. Compared to traditional wood extraction with soxhlet apparatuses, DES application needs only 1 g of wood meal and 60 min.

Further, with different DES mixtures and wood species, more effective extraction methods can be developed in the concept of green chemistry and wood extractives.

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Consumers' Perception of Eco-Services Innovations Related to Furniture

Percepcija potrošača o ekološkim inovativnim uslugama vezanima za namještaj

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ABSTRACT • *Innovations and eco-innovations are effective in building relationships with current and potential customers. One of the possibilities is to innovate in a way that can "materialize" services and at the same time turn them into ecological ones. Such eco-innovations promote sustainable development. The paper focuses on the eco-innovation in services related to furniture. These innovations include ideas such as long-lasting design, maintenance and repair services, remanufacturing of used furniture, and servitization of furniture that includes furniture leasing and renting. The research applied through the Kano model points out the most accepted service innovations in the area of furniture by Slovak respondents. The results present the differences in preferences of customers regarding the individual provided services. It follows that the popularity of individual services is connected with their perception either as traditional innovations or radical ones. Slovak customers prefer financial services supporting purchase of furniture, transport, second chance service, and services related to furniture maintenance and repair.*

KEYWORDS: *innovation; ecological innovation; services; consumers; furniture industry; Kano model*

SAŽETAK • *Inovacije i ekološke inovacije na području proizvodnje namještaja učinkovite su za izgradnju odnosa s postojećim i potencijalnim kupcima. Jedna od mogućnosti inoviranja usluga jest da se one „materijaliziraju" te da istodobno budu ekološke. Spomenute ekološke inovacije promiču održivi razvoj. Ovaj je rad usmjeren na ekološke inovacije usluga vezanih za namještaj. Takve inovacije podrazumijevaju ideje kao što su dugotrajni dizajn, usluge održavanja i popravaka, obnovu rabljenog namještaja te upotrebu namještaja uz plaćanje naknade (npr. leasing i najam). U radu su primjenom Kanoov modela prikazane preferencije inovativnih usluga vezanih za namještaj u Slovačkoj. Rezultati predočuju razlike u preferencijama korisnika glede pojedinih usluga. Pokazalo se da je popularnost određenih inovativnih usluga povezana s percepcijom ispitanika ovisno o tome smatraju li te inovacije tradicionalnima ili radikalnima. Kupci iz Slovačke financijski preferiraju usluge koje podržavaju kupnju namještaja, prijevoz, ponovnu upotrebu te usluge povezane s održavanjem i popravkom namještaja.*

KLJUČNE RIJEČI: *inovacije; ekološke inovacije; usluge; potrošači; industrija namještaja; Kanoov model*

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1 INTRODUCTION
1. UVOD

Many companies providing services significantly contribute to the macroeconomic and social development of society. This is also the reason why many countries currently support entities that focus primarily on service delivery in their activities. Data, information and knowledge are intangible goods produced and provided mainly by the service sector. Effective distribution and use of knowledge is not an automatic process, it requires support functions. This is one of main reasons why the focus has been given to put the services sector and its specificities in the field of innovation in the centre of economic policy research during the previous years. The structural change from a technology-oriented economy made up of industrial production to a company providing services is also reflected in the change in innovation processes. In essence, the innovation process is considered to be a learning process that creates or acquires new knowledge and enables its economic use (Miles, 2008).

The importance of innovation processes, widely recognized at empirical and theoretical levels, and the growing significance of services in productive systems caused a higher attention in the area of services innovation. However, the analysis of innovation in the service sectors is complex in two respects – on one hand, the theory of innovation has evolved with regard to the analysis of technological innovations in manufacturing; on the other hand, it is difficult to measure the specific characteristics of services (especially their output) by traditional economic methods (productivity) and to reveal options how to improve or change them (at a qualitative level) (Gallouj and Weinstein, 1997).

Innovations supported by new technologies are being introduced at an increasing rate (Ringe *et al.*, 2015). Shorter life cycle times point to the importance for the companies to timely make their decisions to innovate. The success of innovation is influenced by the time to embrace innovation, which seems to be an important marketing decision. It is more challenging to be successful with innovation in a market where service companies operate. Service innovation is a challenging concept that is difficult to define unambiguously. This problem is understandable taking into account the size and scope of the service sector.

Service innovation cannot be one-dimensional. Various innovations may serve different objectives and may take various forms: to differ from the competition, provide services more effectively, provide assistance, create a unique experience or achieve results. These objectives help to understand the type of development of service innovation (including service package innovation, process innovation, social innovation, experience innovation or business model innovation). Service innovation also causes changes in consumers' behaviour which may result in a change of brand perception, etc. (Martin *et al.*, 2016; Loučanová *et al.*, 2015).

Technological development is usually used as a means to create new or improve existing products and processes within the services innovations, but it does not offer technological progress itself. Adequate sales and marketing methods are essential, too. The organization of the innovation process focuses not only on the research and development departments that are known in manufacturing companies, but also includes other areas. Information and communication technologies play a central role in the innovation process for service providers due to their orientation to data processing and the resulting intensity of information.

However, services should consider their specific characteristics, which are also taken into account for the implementation of innovation processes (e.g. services are intangible and thus their properties are not easily explained to the customer). The specificity of some services limits efforts to standardize them. A very significant feature of services is their integration with customers because many services are characterized by very close relations with customers or by the integration of external factors into the production process. The process orientation of most services requires close contact with customers, and this can be considered a success factor for service providing companies. Customer integration is based on the mutual production and consumption of services which is the main characteristic of services. However, information technologies help to reduce the synchronization of time and place between the service provider and the customer, which makes this specific feature irrelevant in some areas (Hipp and Grupp, 2005).

The diversity of services is their typical feature so it follows that service innovation and innovation processes may take various forms. The services sector includes economic activities, which are relatively diverse, and therefore, it is difficult to generalize them.

We can conclude that what applies to the service sector as a whole, does not apply to individual types of services. Den Hertog (2000) presents that services innovations can be better understood from several perspectives:

– In terms of the service concept, which means providing a new service in a particular market or a new value for the customer that is included in this service. Many innovations present new ways to solve potential problems, or for some types of services innovations represent a new arrangement/structure (e. g. possibilities of organizing business in different ways - whether they are more or less specialized, or more or less focused on quality or cost savings).
– In terms of the client interface, changes are obvious in the way clients are involved in service design,

production and consumption of services that are taken into account (e.g. some services offer self-service options for their clients).
– In terms of service delivery system – changes are reflected in the way how service providers distribute their services. Many innovations concern the electronic providing of services, but there are also innovations in the area of transport or packaging.
– In terms of technology that allows better efficiency of information processing, as well as increasing the overall quality and efficiency of provided services.

Many innovations in services represent a combination of four above mentioned perspectives (aspects). Service innovation referring primarily to one dimension may require changes in other dimensions. The first aspect mainly concerns the service itself in its main feature - immaterial, while dimensions 2 and 3 mainly concern the intensity of the client's involvement in the services production. Dimension 4 has more in common with traditional product innovations, with a particular emphasis on new innovations in information technology. Innovations in any of these respects can be more or less complementary or completely radical, requiring more or less new knowledge and reorganization of production processes (Miles, 2008).

There are several ways in which the service innovation process itself can take place. Toivonen and Tuominen (2009) identified five innovation processes with respect to their degree of formality. In the sequence from less to more formal processes, we recognize:
– internal processes without a specific project (i.e. unintentional and incremental innovations related to an existing service),
– internal innovation projects (i.e. intentional projects aimed at improving service production systems and their content),
– innovative projects with pilot customers (i.e. new ideas are tested in cooperation with the customer),
– innovative projects tailored to the customer (i.e. the service provider seeks to solve a specific problem with the client),
– innovative projects funded externally (i.e. research collaboration focused on the generation of new service concepts and / or platforms).

Some authors (Agarwal *et al.,* 2003; Toivonen and Tuominen, 2009) mention that the market is customer orientated when associated with innovation. In times of strong competition in the market, only those who innovate can be successful. The current trend, within the interconnection of innovations and resources, rests in the implementation of eco-innovations, which are based on the principles of sustainable development in terms of socially responsible business. Therefore, in this paper we focus on the consumers' perception of services innovation and ecological innovation in furniture.

Currently, there is a big progress in servicing. The most significant environmental impacts of furniture are the aspects related to the production of raw materials and the disposal of old furniture (Nickel *et al.*, 2003). Literature suggests two major strategies to tackle the environmental problems associated with furniture consumption: one strategy is to design furniture so that it is more suitable for material recycling (Witte, 2000). The second one is to decrease furniture consumption by e.g. prolonging furniture lifetime (Vollmer, 1999, cited in Besch, 2005). Witte (2000) intensively examined different strategies for the implementation of closed loop recycling in the furniture (including material recycling). A material recycling strategy involves the design of furniture that facilitates dismantling and separation of all materials that are then recycled. This strategy is the most suitable for cheap, mass furniture. He pointed out that the most important aspect of material recycling of furniture is the recycling cost. The sorting and disassembly of old furniture is expensive and it is questionable whether the revenue from the recycled materials can cover these costs. He also assessed the costs for the disassembly and transport of the desk for material recycling and concluded that it is not economically feasible. The profitability of material recycling from furniture is closely dependent on the volume of waste: the more furniture is transported and disassembled together, the more profitable the process becomes. In addition, it is extremely important that furniture is designed for easy dismantling and that all materials are labelled to facilitate the separation. The prices for the recycled materials on the market also heavily influence whether it makes economic sense to recycle. Witte's results (2000) indicated that material recycling for furniture does not seem to be a promising strategy under the current conditions, since the recycling costs will most probably exceed the revenue from the recycled materials.

Several authors (Vollmer, 1999, cited in Besch, 2005; Witte, 2000) have brought forward the idea of prolonging the lifetime of furniture in order to reduce environmental impacts. Lifetime extension strategies for furniture can include one or several of the following elements:
– Design for durability: In order to reduce the environmental impacts of furniture consumption, the furniture should last for long periods. Environmentally proactive manufacturers are committed to product durability (Wilkhahn, 2002). The disadvantage of this strategy is that it undermines the business case for producers: if their products can be used over long periods, they will sell less furniture in the end.
– Maintenance and repair services: Manufacturers can prolong the lifetime of their products by offering

maintenance and repair services parallel to the sale of furniture. Witte (2000) concludes that lifetime extension services for chairs can be economically implemented. Furthermore, manufacturers can gain additional revenue from offering supplementary services to product sales.

– Reuse of furniture parts: This strategy is concerned with reuse of certain furniture parts for the production of new furniture. The main idea of this strategy is that certain parts of used furniture (e.g. metal pillars or desk support frameworks) are still valuable and should be reused and not disposed of. Such utilization of old parts in new furniture could lead to cost reductions of up to 35 %. One problem for the implementation of this strategy is the development of a take back system that ensures producers a constant reflow of furniture or furniture parts in good condition. Witte (2000) suggested a model where producers offer a full-service contract with the product's sale. Within this contract, producers offer maintenance and repair of their products and take back old elements to reintegrate them into the production of new furniture. The risk of this business model is that it could lead to lower sales figures.
– Remanufacturing of used furniture: The take back and remanufacturing of used furniture can facilitate the second or the third usage period. Furniture is a simple product that in principle does not lose its function over many decades. Witte (2000) and Besch (2005) stated that the costs of raw material are a major fraction of production costs for furniture. Therefore, it seems promising to remanufacture furniture. According to a case study, chair remanufacturing could be realized economically but difficulties remained in finding a market for second hand chairs.
– Servitization - leasing or renting: The idea to lease or rent out products instead of selling them evolved from the consideration that this strategy creates more incentives for producers to prolong the lifetime of their products (Besch, 2005). If the ownership of a product stays with the producer, one can directly benefit from the longer usage period. Vollmer (1999, cited in Besch, 2005) examined existing leasing concepts for furniture and concluded that these offers are mainly financial instruments that do not support closed loop recycling. Goedkoop *et al.* (1999) presented an interesting case study where furniture was leased out to customers from a furniture pool that was included in the full-service-package of the building. Servitization is one of the business models of the circular economy, focused on the provision of services both beyond the product itself, as well as the provision of the product as a service, for example in the form of its rental. Surveys show that almost 70 % of manufacturing companies are already working with some form of servitization and many are just starting to work on service innovations. At the same time, the portfolio of services offered is really diverse and many have already reached Slovakia (Sumne, 2019). The trend of servitization is gradually beginning to penetrate into areas not traditionally associated with renting.

The Swedish furniture manufacturer IKEA decided to use this model and started offering its products for rent. It is possible to rent a new kitchen unit and return it after a while. Instead of the furniture being discarded over time, the company will refurbish it a bit when it is returned and can sell it to extend its life cycle. This leasing project is a part of the company's endeavour to develop a business model that will be based not only on sales, but also on the reuse of furniture parts in the production of new ones. The service is currently provided only in selected countries. In addition to the above, the company also implements other projects of eco-innovation in services while, at the same time, supporting the circular economy. As an example either the Recovery Project can be mentioned, which is in charge of repairing and repackaging products that have been damaged during transport, display, or handling or the ecological service project "Second Life of Furniture" that allows customers to bring their old furniture to the IKEA department store and then sell it to other customers at a reduced price. The company is also trying to reduce its climate footprint by 15 % in absolute terms, which would mean a 70 % reduction in emissions per product due to growth by 2030 (Janove, 2019).

From an economic point of view, lifetime extension strategies seem to be a better option to close material loops in the furniture compared to material recycling. Nevertheless, literature has also identified obstacles that limit the implementation of these strategies. The main barrier concerns the lifetime extension strategies that do not match up with traditional business models. The overall objective of product-oriented companies is to sell as many products as possible, which is in a conflict with the strategy of prolonging the usage period of products. Furniture leasing or renting is a way how to solve these contradicting incentives. Existing leasing concepts for furniture do not consider lifetime extension strategies. The furniture market is still lacking a comprehensive service package that combines the different elements of the lifetime extension strategy to facilitate closed material loops.

2 METHODOLOGY
2. METODOLOGIJA

The Kano model (Kano *et al.*, 1984) was used as a primary method to evaluate the consumers' perception of innovation and ecological innovation in services of furniture. It considers theories of contradiction to identify the differentiation variables of the product by creating its unique position on the market. The analysis is primarily focused on finding the values of the services in furniture that the consumer considers to be a must, attractive and one-dimensional.

The must-be requirements are significant from the consumer point of view because in the case of their non-compliance they cause strong dissatisfaction to consumers. On the other hand, if they are met, they have little effect on consumer satisfaction. It is a basic product criterion that the consumer requires automatically.

One-dimensional requirements are defined as claims, where we can see a linear dependence between their fulfilment and consumer satisfaction. The more requirements are met, the more satisfied the consumer is. Attractive values include requirements that lead exponentially to an increase in consumer satisfaction. Regarding the above-mentioned information these requirements have the most significant impact on consumer satisfaction. In addition to the above explained requirements, there are also identified reverse, questionable and indifferent requirements not influencing the consumers. Of course, it is not possible to strictly separate individual requirements. They overlap and influence each other at the same time (Loučanová, 2021; Loučanová and Olšiaková, 2020).

The analysis of parameters, focused on the examined problem, was followed by the methodical procedure to assess the services innovation in furniture by Slovak consumers, such as:

- Creating a 3D furniture design made-to-measure
- Furniture made-to measure
- Furniture financing
- Furniture transportation
- Furniture assembly
- Disassembly of furniture
- Removal of old furniture
- Second chance for furniture
- Maintenance and repair of furniture
- Servitization of furniture

After precisely determining the parameters, a questionnaire was developed respecting the KANO model needs. The questionnaire development involved the generation and formulation of two questions for each examined parameter. In the first case, the question was formulated to detect the consumers´ responses as to whether their requests were met. On the contrary, in the second case, the question was formulated assuming that the consumers' requests were not met. Consumers had the opportunity to express agreement or disagreement with the question or statement on the Likert scale (1 – like, 5 – dislike). Then measures for the questionnaire implementation were determined. The sample consisted of 1335 respondents, so the minimum number of respondents (667) was reached. The minimum number was calculated with respect to the sample size calculation, with the average permanent population in Slovakia, gained from the data presented by the Statistical Office of the Slovak Republic (5,457,873 inhabitants on January 01 2020). The sample was calculated at the 99 % confidence level and margin of error of 5 %.

After performing the questionnaire survey, a database of obtained data was created, where the examined parameters for innovation in services in furniture were defined, and subsequently a numerical expression of consumer agreement or disagreement with the given question concerning the defined parameter was assigned.

The individual answers to the positively and negatively asked question (statement) were evaluated separately for each parameter using the cross rule of the KANO model. Further, individual properties – (attractive (A), must-be (M), reverse (R), one-dimensional (O), questionable (Q) and indifferent (I)) were specified by this determination (Table 1).

The identified consumer requirements were divided into groups and redistributed with regard to the proportions of respondents' sample in percentage. The most represented group of requirements characterized the resulting perception of the examined parameter or value. The derived individual categorizations can be utilized further by aggregating them across all respond-

Table 1 KANO model for evaluation of consumer requirements (Grapentine, 2015; Loučanová, 2021; Loučanová and Olšiaková, 2020)
Tablica 1. Kanoov model za procjenu zahtjeva potrošača (Grapentine, 2015.; Loučanová, 2021.; Loučanová and Olšiaková, 2020.)

Like *Sviđa mi se*		**Answer to dysfunctional question** *Odgovori na disfunkcionalna pitanja*				
		Acceptable *Prihvatljivo*	No Feeling *Bez dojma*	Must-be *Obvezno*	Do not like *Ne sviđa mi se*	
Answer to functional question *Odgovori na funkcionalna pitanja*	Like / *sviđa mi se*	Q	A	A	A	O
	Acceptable / *prihvatljivo*	R	I	I	I	M
	No Feeling / *bez dojma*	R	I	I	I	M
	Must-be / *obvezno*	R	I	I	I	M
	Do not like / *ne sviđa mi se*	R	R	R	R	Q

ents using the customer satisfaction and customer dissatisfaction indices (Berger *et. al*, 1993; Shahin *et al*., 2013; Beier *et al*., 2020):

$$Consumer\ satisfaction = \frac{A+O}{A+O+M+I} \quad (1)$$

$$Consumer\ dissatisfaction = -\frac{O+M}{A+O+M+I} \quad (2)$$

The categorization frequencies A, I, M, and O mean the number of respondents who classified the offering as attractive, indifferent, must-be, or one-dimensional.

The indices reflect the proportion of respondents for whom the existence (absence) of an offering attribute influences customer satisfaction (customer dissatisfaction). Additionally, consumer dissatisfaction has a minus sign to emphasize the negative effects on customer satisfaction (for historical reasons). For each offering, the satisfaction index is within the range of [0, 1] and for customer dissatisfaction within [−1, 0]. A value close to 1 of consumer satisfaction indicates a high proportion of customers among whom satisfaction can be generated, and a value close to −1 indicates a high proportion of respondents among whom dissatisfaction can be generated. The scale mean of 0.5 for consumer satisfaction (or −0.5 for consumer dissatisfaction) indicates whether the majority of respondents can be positively (or negatively) stimulated, yielding a two-dimensional grid with four quadrants:

Attractive offerings

, if {0.5 ≤ Consumer satisfaction ≤ 1 and 0 ≥ Consumer dissatisfaction > −0.5

Indifferent offerings

, if {0 ≤ Consumer satisfaction < 0.5 and 0 ≥ Consumer dissatisfaction > −0.5

Mandatory offerings

, if {0 ≤ Consumer satisfaction < 0.5 and −0.5 ≥ Consumer dissatisfaction ≥ −1

One−dimensional offerings

, if {0.5 ≤ Consumer satisfaction ≤ 1 and −0.5 ≥ Consumer dissatisfaction ≥ −1

The respondents classify the offering as reverse (category R, frequency R) or questionable (category Q, frequency Q). Those are not reflected in the consumers´ satisfaction and dissatisfaction indices and in the table because only respondents with "strong" assessments are taken into consideration. Besides the satisfaction indices, the total strength of each offering can be determined, indicating the proportion of attractive, one-dimensional, and must-be assessment of this offering among all assessments:

$$Total\ Strength = \frac{A+M+O}{A+I+M+O+Q+R} \quad (3)$$

Recently, an alternative to the above-described aggregated analysis has been proposed and applied, the so-called segmented Kano perspective, where respondents were grouped according to their assessments using cluster analysis with respect to their answers. When highly innovative offerings are investigated, the segmented Kano perspective is preferable because the usual categorizations such as attractive or indifferent at the aggregated level are reduced. Consumer segments that are highly receptive can be identified (Baier *et al.*, 2005). Therefore, in this paper cluster method was also conducted to gain a better insight into attitude of Slovak customers to services innovation related to furniture using SPSS package.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

After the survey by the Kano questionnaire, a database of acquired data was processed. The database consisted of data from 1335 respondents. Tab. 2 outlines the sample descriptive statistics. The survey was mainly completed by women (56.40 % of responses). Among the considered population, the largest share of respondents (36.40 %) were from 18 to 30 years old, then from 31 to 50 years old (32.73 %), and finally respondents over 51 years old (30.86 %).

From the database of obtained data related to our research, we evaluated the individual answers for each question by cross rule of the Kano model using the Kano table, which is presented in the methodology section. The determined properties were subsequently specified as one-dimensional (O), attractive (A), mandatory (M), questionable (Q), reverse (R) and indifferent (I) requirements.

Table 2 Descriptive statistics of the sample (n = 1335)
Tablica 2. Deskriptivna statistika (n = 1335)

Demographics *Demografska struktura*	**Specification** *Kategorija*	**Multiplicity** / *Višestrukost*	
		Absolute, *n* / *apsolutno, n*	**Relative, %** / *relativno, %*
Age / starost	18 – 30 years / *18 – 30 godina*	486	36.40
	31 – 50 years / *31 – 50 godina*	437	32.73
	51 years and older / *51 godina i stariji*	412	30.86
Gender / spol	Female / *žene*	753	56.40
	Male / *muškarci*	582	43.60

Table 3 Specific requirements for services innovation in furniture
Tablica 3. Posebni zahtjevi vezani za inovativne usluge glede namještaja

Attributes *Obilježje*	**Requirements** / *Zahtjevi*												**Identified require-ments*** *Identifici-rani zahtjevi**
	Attractive *Privlačni*		**One-dimensional** *Jednodimenzionalni*		**Mandatory** *Obvezni*		**Irrelevant** *Nevažni*		**Reverse** *Obrnuti*		**Questionable** *Upitni*		
	Multiplicity / *Višestrukost*												
	Absolute / *apsolutno, n*	**Relative** / *relativno, %*	**Absolute** / *apsolutno, n*	**Relative** / *relativno, %*	**Absolute** / *apsolutno, n*	**Relative** / *relativno, %*	**Absolute** / *apsolutno, n*	**Relative** / *relativno, %*	**Absolute** / *apsolutno, n*	**Relative** / *relativno, %*	**Absolute** / *apsolutno, n*	**Relative** / *relativno, %*	
Creating a 3D furniture design made-to-measure *3D dizajniranje namještaja po mjeri*	205	15.36	496	37.15	63	4.72	229	17.15	187	14.01	155	11.61	O
Furniture made-to measure *izrada namještaja po mjeri*	214	16.03	217	16.25	65	4.87	197	14.76	495	37.08	147	11.01	R
Furniture financing *financiranje namještaja*	453	33.93	185	13.86	24	1.80	453	33.93	61	4.57	159	11.91	A, I
Furniture transportation *transport namještaja*	886	66.37	65	4.87	165	12.36	87	6.52	124	9.29	8	0.60	A
Furniture assembly *montaža namještaja*	81	6.07	404	30.26	289	21.65	515	38.58	27	2.02	19	1.42	I
Disassembly of furniture *rastavljanje namještaja*	32	2.40	3	0.22	65	4.87	674	50.49	529	39.63	32	2.40	I
Removal of old furniture *uklanjanje starog namještaja*	204	15.28	493	36.93	67	5.02	227	17.00	195	14.61	149	11.16	O
Second chance for furniture *ponovna upotreba namještaja*	810	60.67	79	5.92	81	6.07	197	14.76	159	11.91	9	0.67	A
Maintenance and repair of furniture *održavanje i popravak namještaja*	451	33.78	204	15.28	25	1.87	436	32.66	62	4.64	157	11.76	A
Servitization of furniture *iznajmljivanje namještaja*	213	15.96	217	16.25	69	5.17	193	14.46	495	37.08	148	11.09	R

*Identified requirements: attractive (A), mandatory (M), reverse (R), one-dimensional (O), questionable (Q) or indifferent (I). / *Identificirani zahtjevi: privlačni (A), obvezni (M), obrnuti (R), jednodimenzionalni (O), upitni (Q) ili indiferentni (I).*

Regarding the values presented in Table 3, it can be stated that Slovak respondents perceive the innovation of services provided in furniture as positive. Services provided when purchasing the furniture, such as furniture financing, second chance, furniture maintenance and repair are attractive to respondents. It means that these services are unexpected by customers and they have a significant impact on customers satisfaction. Their significance lies in the fact that if they are not provided, they will not cause customers dissatisfaction. On the other hand, if they are provided, they will significantly influence customers shopping behaviour. The respondents perceive services such as the 3D custom-made furniture design and the removal of old furniture as one-dimensional requirements in the area of service innovations in furniture. These are the innovations of furniture services that lead to customer satisfaction in the case of their fulfilment, but if they are not provided, they cause customers dissatisfaction. However, one-dimensional requirements are not automatically expected. There is a direct relationship between the degree of satisfaction and fulfilment of customers' requirements.

Slovak respondents consider innovations such as the assembly and disassembly of the furniture to be insignificant innovations concerning the furniture services. These services do not affect them. It means that their meeting or not meeting does not affect customers´ satisfaction or competitiveness of furniture on the Slovak market. On the contrary, Slovak respondents perceive the production of custom-made furniture and servitization of furniture or furniture rental contradictory. This means that Slovak respondents perceive these innovations exactly in reverse compared to the previous ones and their reaction to them is reverse.

Based on the above findings and requirements identification of Slovak customers regarding services innovations in furniture, the values were calculated of total strength, customer satisfaction and dissatisfaction (Table 4).

Based on the calculated values of total strength, it can be stated that the transport of furniture (0.8360) and the second chance for furniture (0.7266) had the greatest impact on the respondents, while disassembly of furniture had the lowest impact on respondents. This statement also applies in terms of their satisfaction.

The parameters represented by individual services innovations are positioned with respect to their customer satisfaction and dissatisfaction values. The four quadrants visualize the respondents' majorities divided into mandatory, one-dimensional, attractive and indifferent requirement categories (Figure 1).

To obtain further insight as described, a cluster analysis of the data was applied, which identified three clusters. Table 5 provides additional information on categorizations at customers levels for individual clusters.

The specified clusters can be characterized as follows:

– Cluster 1 is represented by attractive requirements of Slovak customers concerning services provided in furniture, such as transport, servitization, made-to-measure production, furniture financing, and maintenance or furniture repair. These innovations are the most attractive for customers at the age of 18-30 years and for men population.
– Cluster 2 is represented by one-dimensional requirements of Slovak customers regarding the services provided in furniture. They include 3D design and removal of old furniture. They are perceived this way by customer at the age category from 31 to 50 years and especially for women population.
– Cluster 3 is represented by indifferent requirements of Slovak customers related to services provided in

Table 4 Indices of customer satisfaction and dissatisfaction with services innovations in furniture
Tablica 4. Indeksi zadovoljstva i nezadovoljstva kupaca inovativnim uslugama vezanima za namještaj

Parameters / *Parametri*	**Total strength** *Ukupna jakost*	**Customer satisfaction** *Zadovoljstvo kupca*	**Customer dissatisfaction** *Nezadovoljstvo kupca*
Furniture transportation / *transport namještaja*	0.8360	0.7905	-0.1912
Second chance for furniture *ponovna upotreba namještaja*	0.7266	0.7618	-0.1371
Furniture assembly / *montaža namještaja*	0.5798	0.3763	-0.4976
Removal of old furniture *ukljanjanje starog namještaja*	0.5723	0.7033	-0.5651
Creating a 3D furniture design made-to-measure *3D dizajniranje namještaja po mjeri*	0.5723	0.7059	-0.5629
Maintenance and repair of furniture *održavanje i popravak namještaja*	0.5094	0.5869	-0.2052
Furniture financing / *financiranje namještaja*	0.4959	0.5722	-0.1874
Servitization of furniture / *iznajmljivanje namještaja*	0.3738	0.6214	-0.4133
Furniture made-to measure *izrada namještaja po mjeri*	0.3715	0.6219	-0.4069
Disassembly of furniture / *rastavljanje namještaja*	0.0749	0.0452	-0.0879

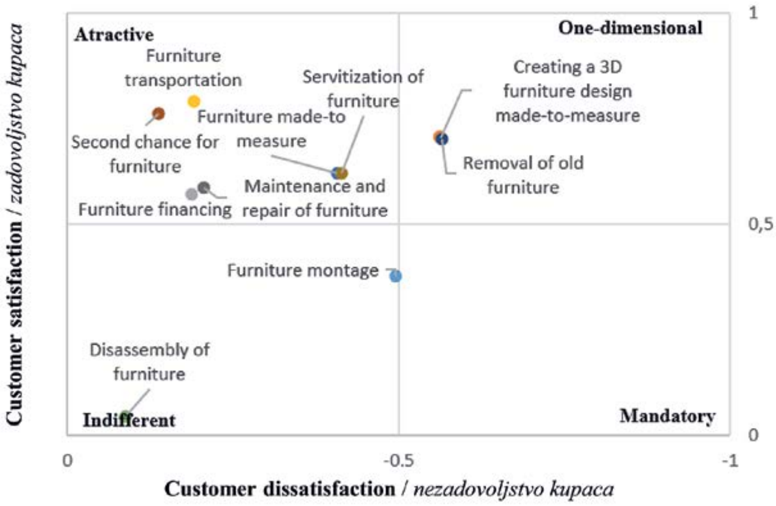

Figure 1 Overall assessment of services innovation in furniture based on indices of satisfaction and dissatisfaction
Slika 1. Ukupna procjena inovativnosti usluga vezanih za namještaj na temelju indeksa zadovoljstva i nezadovoljstva kupaca

Table 5 Specific descriptive statistics of customers clusters
Tablica 5. Specifična deskriptivna statistika klastera kupaca

Aspect / *Aspekt*	**Specifications** *Kategorija*	**Overall, %** *Cjelokupno, %*	**Cluster - *Segment* 1, %** *Klaster – dio 1., %*	**Cluster- *Segment* 2, %** *Klaster – dio 2., %*	**Cluster - *Segment* 3, %** *Klaster – dio 3., %*
Age / *starost*	18 – 30 years *18 – 30 godina*	36.4	43.27	34.31	31.12
	31 – 50 years *31 – 50 godina*	32.73	27.13	41.72	27.13
	51 years and older *51 godina i stariji*	30.86	29.60	23.98	41.76
Gender / *spol*	Female / *žene*	56.4	47.93	54.91	62.72
	Male / *muškarci*	43.6	52.07	45.09	37.28

furniture, such as furniture assembly and disassembly. This cluster includes mainly customers dominated by women older than 51 years.

The Kano model monitoring the perception of service innovations in furniture has shown that Slovak respondents perceived positively the services provided in furniture, but they saw obstacles in the area of radical services innovations in furniture such as the renting or removing old furniture, which could hinder their successful implementation in the country. The research pointed to a phenomenon that manifests in the implementation of all new ideas. People are resistant to change. They are afraid to abandon traditional business models. Generally, Slovaks are conservative and strongly loyal to the familiar structures and could not imagine a change in these structures. As Besch (2005) stated, radical innovations in furniture services must propose explicit advantages compared to traditional business model to get attention. Therefore, consumers will be willing to take risks and abandon traditional business models of buying furniture only when they expect significant benefits or profit. A general problem of environmentally innovative approaches to services (eco-innovation services) may be that their benefits are not explicit for most businesses and customers. As many environmental impacts are still not included in market prices in the economy, company leaders do not logically consider the environmental decisions of product and system designs (Mont, 2003, Loučanová, Nosáľová, 2020). Renting furniture can be difficult because people are not willing to rent furniture. This result may also illustrate the general problem of our society that hinders sustainability efforts. As Besch (2005) presents, this trend cannot be doubted in furniture either, but from the investment point of view, furniture does not represent such a high investment and therefore its rent is not as attractive as other innovations in offered furniture services.

Based on the above findings, we can list the services characteristics in furniture, which appear to be advantageous for successfully addressing Slovak customers. The results of the research support findings by Tischner (2002) that the products have one or more characteristics that appear to be particularly suitable for successfully reaching customers or implementing furniture sales strategy. Specifically, Slovak customers should be provided by:

- a way to finance their purchase of furniture,
- transport,
- second chance service,

– services related to furniture maintenance and repair.

The results show that furniture services in Slovakia are not strongly influenced by fashion or trends, as also stated by Tischner (2002).

4 CONCLUSIONS
4. ZAKLJUČAK

At present, service companies have to constantly renew their processes and service offerings in order to remain competitive. Advanced modern economies are increasingly specializing in services. As the level of services provided in the economy reflects its maturity, society should create the conditions for their further development, including the support of innovative activities of service companies.

The paper presents the results of the survey focused on evaluating the attitude of Slovak customers to services innovation related to furniture. Slovak furniture customers do not consider any of the furniture purchase services to be mandatory. They consider furniture transportation, second chance for furniture, furniture financing and maintenance and repair of furniture as attractive services. Satisfaction is growing for customers in offering services such as creating a 3D furniture design made-to-measure and removal of old furniture. Other innovative forms of providing services in the sale of furniture are perceived by customers as indifferent, or they have the opposite attitude to these services.

Many of these innovations are eco-innovations and they aim to reduce the negative impact on the environment. They are also provided to increase the comfort and satisfaction of customers not only while buying but also while using furniture and solve their problem how to arrange subsequent disposal. If customers use efficiently these services, they not only satisfy their various needs, but also actively and naturally participate in achieving sustainability.

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Impact of Circular Saw Blade Design on Forces During Cross-Cutting of Wood

Utjecaj izvedbe lista kružne pile na sile pri poprečnom piljenju drva

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ABSTRACT • *This paper evaluates the impact of the number of saw blade teeth on kinematic and dynamic parameters during cross-cutting of soft and hard woods with mitre saw BOSCH GCM 12 GLD Professional. The forces measured using a three-dimensional XYZ dynamometer Kistler 9257B and subsequent calculation based on a mathematical model were applied in the Ernst-Merchant diagram and the forces acting in the cutting of spruce and oak planks were analysed and compared. The wood cutting was performed by four geometrically and structurally similar blades with a 300 mm diameter and with different count of WZ-shaped teeth (z = 26, 36, 48, 60). The optimum saw blade for the given cutting conditions was subsequently assessed by statistical analysis. Unlike many studies researching the cutting resistances in transversal and longitudinal wood cutting, this experiment and force analysis was performed in dependence on the constant feed force.*

KEYWORDS: *saw blade; mitre saw; Kistler dynamometer; cross-cutting; force analysis; Ernst-Merchant model; statistical analysis*

SAŽETAK • *U ovom se radu ispituje utjecaj broja zubi lista pile na kinematičke i dinamičke parametre tijekom poprečnog rezanja mekih i tvrdih vrsta drva poprečnom klatnom pilom BOSCH GCM 12 GLD Professional. Vrijednosti sila izmjerene spomoću trodimenzionalnog XYZ dinamometra Kistler 9257B te vrijednosti naknadnih izračuna primjenom matematičkog modela uvrštene su u Ernst-Merchantov dijagram te je napravljena analiza i usporedba sila pri piljenju smrekovih i hrastovih piljenica. Uzorci drva piljeni su s četiri geometrijski i strukturno slična lista pile promjera 300 mm s različitim brojem zubi tipa WZ (z = 26, 36, 48, 60). Statističkom analizom određen je optimalan list pile za zadane uvjete piljenja. Za razliku od mnogih istraživanja u kojima su proučavani otpori pri poprečnome i uzdužnom piljenju drva, ovo istraživanje i analiza sila provedeni su pri konstantnoj posmičnoj sili.*

KLJUČNE RIJEČI: *list pile; poprečna klatna pila; Kistlerov dinamometar; poprečno piljenje; analiza sila; Ernst-Merchantov model; statistička analiza*

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1 INTRODUCTION
1. UVOD

Cutting of wood-based materials is one of the most widespread technologies in woodprocessing. A circular saw is used for cross-cutting or longitudinal cutting of materials, especially in the production of elements of timber structures or joinery products. Cross-cutting of wood in the production of final products is usually carried out as accurate cutting to the required dimensions using mitre or transverse saws (Sandak and Negri, 2005; Orlowski *et al*., 2020). In the past, most authors, Prokeš (1982), Pernica (1999), Wasilewski-Orlowski (2012) and others, examined the process of wood-cutting with a machine feed of a constant speed. The fixed speed during the machine feed of the workpiece determines the constant feed per tooth and the thickness of the layer cut.

It is also common knowledge that with an increasing tooth count, the feed per tooth decreases evenly as well as the thickness of the uncut chip removed with a tooth. On the other hand, the total cutting force and the force required to feed the workpiece grow proportionally with the number of teeth in contact. When cross-cutting wood using transverse or mitre saws, the saw blade feed is most often done manually. If such saws are used, it is necessary to select the geometry, the tooth count and achip thickness limiter so that the blade tooth is able to remove a chip and make a quality cut with minimal energy demands for the application of an average feed force. Similar issues have been addressed in, for example, Kminiak and Kubš (2016), Kminiak and Siklienka (2016), who sought to clarify this process from an energy point of view based on an assessment of the general cutting power.

This paper presents the measurement and analysis of the forces on the tool tooth so that the suitability of a saw blade in terms of the tooth count can be assessed.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Test samples
2.1. Ispitni uzorci

Samples of spruce (*Picea Abies*) and European oak (*Quercus robur*) were used for the experiment. Five samples of oak and spruce in the form of planks were taken from tangential timber without growth defects and with evenly distributed tree rings. The sample dimensions were as follows: 250 mm width, 1000 mm length, and 50 mm thickness.The density of oak was $\rho = 671$ kg/m^3 and spruce $\rho = 448$ kg/m^3 at the relative moisture content $w_r = 11$ % (±2 %).

2.2 Saw blades used
2.2. Upotrijebljeni listovi pila

Wood cross-cutting was carried out gradually by four blades FREUD (LU1C 0400, LU2A 1900, LU2A 2100 and LU2E 0200) with a diameter D = 300 mm; the blades were fitted with of tungsten carbide teeth, WZ-shaped, with the same width s = 3.2 mm and with the same tooth geometry - clearance angle $\alpha_f = 15°$, cutting edge angle $\beta_f = 60°$, rake angle$\gamma_f = 15°$ and set of cutting edges $\kappa_r = 10°$. The saw blades only differed in the tooth count $z = 26$, $z = 36$, $z = 48$, and $z = 60$ and were marked accordingly as PK26, PK36, PK48 and PK60. At least 25 cuts were made with each saw blade, in both hard and soft wood.

A measurement of the tooth blade rounding radius was carried out for each blade (Figure 1); it ranged at an interval $\rho = 7 \div 10$ µm, which corresponded to a correct sharpening. Measurement of blade rounding was performed using the Keyence VHX 5000 microscope.

2.3 Testing and measuring devices
2.3. Uređaji za ispitivanje i mjerenje

The experiment was carried out on a test device (Figure 2) consisting of a BOSCH GCM 12 GLD Professional mitre saw (P = 2 kW, n = 3800 rpm, v_c = 60

Figure 1 Saw blade PK48 (LU 2A 2100) and tooth blade edge
Slika 1. List pile PK48 (LU 2A 2100) i radijus oštrice

Figure 2 View of Bosch mitre saw test device with Kistler dynamometer
Slika 2. Pogled na poprečnu klatnu pilu Bosch s Kistlerovim dinamometrom

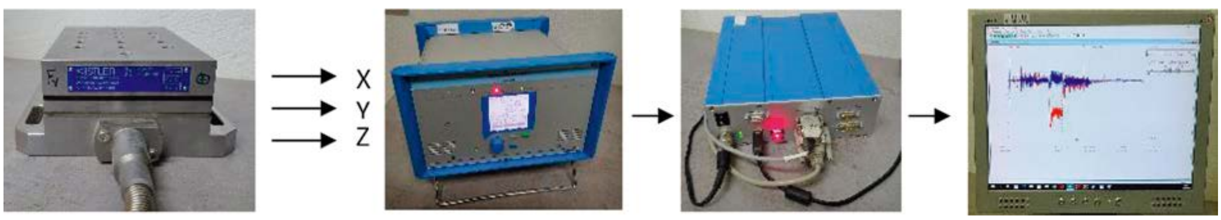
m/s), with adjustable angle 52° L / 60° R, with max. transverse cut 104 mm × 341 mm.

The saw was accompanied by a modified table, which allowed the installation of high-end dynamometer Kistler 9257 B with a measurement and evaluation system (Figure 3). The measuring chain consisted of a PC with DynoWare application, A-D converter with data bus 5679A, eight-channel amplifier 5070A, and four-sensor three-axis piezoelectric dynamometer (force sensor) Kistler 9257B. Further, the saw was equipped with a cable mechanism with a changeable weight (from 1 kg to 5 kg), which allowed the constant feed force to be set and the blade to be pushed towards the cut. An average constant feed force of 30 N was used throughout the experiment.

2.4 Methods
2.4. Metode

The cross-cutting of spruce and oak samples took place in a enclosed cutting mode (Lisičan *et al*., 1996; Varkoček *et al*., 2001). The main cutting edge of a WZ tooth with the set of cutting edges $\kappa_r = 10°$ cut in the tangential-transversal model with angles $\varphi_1 = 10°$ (angle between the tooth main cutting edge and the direction of wood fibres), $\varphi_2 = 10°$ (angle between the direction of wood fibres and the cut plane), $\varphi_3 = 90°$ (angle between the cutting force vector and the direction of wood fibres).

The measurement of forces by the Kistler dynamometer was carried out on two axes, *Y* and *Z*. Force



Piezoelectric dynamometer (force sensor, three-axis) Kistler 9257B → 8-channel amplifier 5070A → A-D data bus converter 5679A → PC with DynoWare A-D measuring application
Piezoelektrični dinamometar (senzor sile, tri osi) Kistler 9257B → 8-kanalno pojačalo 5070A → A-D pretvarač sabirnice podataka 5679A → PC s DynoWare A-D mjernom aplikacijom

Figure 3 Measuring chain of the Kistler dynamometer
Slika 3. Mjerni lanac Kistlerova dinamometra

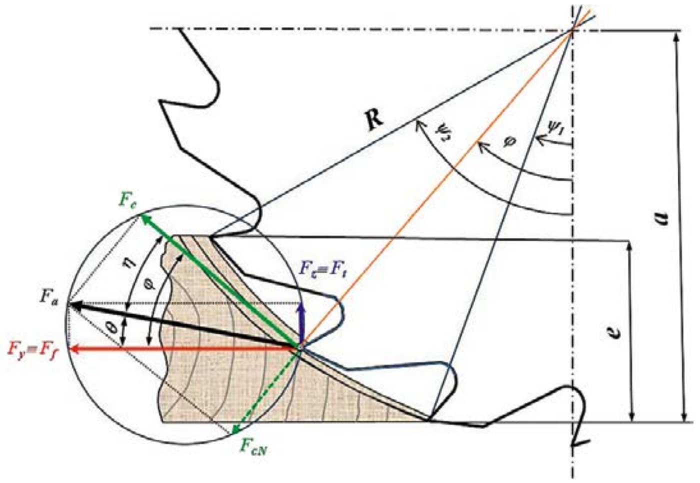


Figure 4 Circular sawing machine cutting force model
Slika 4. Model sile rezanja listom kružne pile

F_y, which is directly equal to the feed force F_f, was measured on Y axis. Force F_z, which equals the thrust force F_t, was measured in Z direction. This force can push the workpiece to the table or even away from it, in dependence on the current cutting conditions and resistances. The diagram of the saw blade and workpiece interaction captured by Ernst-Merchant circle is shown in Figure 4. Although this is not an orthogonal cutting for the main cutting edge (κ_r = 10°), it is possible to use an Ernst-Merchant circle for decomposition and force analysis.

The geometry of the saw blade and the Ernst-Merchant circle (Figure 4) yields a simple mathematical apparatus for calculating additional forces that classify the entire cutting process (Kopecký *et al*., 2019).

As the radius of the saw blade (R), the position of the table (workpiece) to the axis of the saw blade rotation (a) and the cutting height - thickness of the cut material (e) are known, it is possible to express the technological angles and tooth path in the workpiece:

Tooth angle when entering the workpiece

$$\psi_1 = \cos^{-1}\frac{a}{R} \quad (1)$$

Tooth angle when getting out of the workpiece

$$\psi_2 = \cos^{-1}\frac{a-e}{R} \quad (2)$$

Tooth position angle at the point of the mean uncut chip thickness

$$\varphi = \frac{\psi_1 + \psi_2}{2} \quad (3)$$

Angle of cut

$$\psi = \psi_2 - \psi_1 \quad (4)$$

Length of tooth path in workpiece

$$l = \frac{\pi \cdot D}{360}\psi \quad (5)$$

Active force

$$F_a = \sqrt{F_y^2 + F_z^2} \quad (6)$$

Cutting force

$$F_c = F_a \cdot \cos\eta \quad (7)$$

Angle between active force F_a vector and cutting force F_c vector

$$\eta = \varphi - \theta \quad (8)$$

Angle between active force F_a vector and feed force F_f

$$\theta = \tan^{-1}\frac{F_z}{F_y} \quad (9)$$

Number of teeth, z^I, which simultaneously remove a chip from the workpiece

$$z^1 = \frac{l}{t_p} \quad (10)$$

Where tooth pitch

$$t_p = \frac{\pi \cdot D}{z} \quad (11)$$

Cutting force per tooth of the saw blade

$$F_c^1 = \frac{F_c}{z^1} \quad (12)$$

Calculation of kinematic technological parameters:

Feed speed $v_f = f_z \cdot n \cdot z = L / t$ (13)

Feed per tooth

$$f_z = \frac{L}{\frac{n}{60} \cdot t \cdot z} \quad (14)$$

Mean thickness of the cutting layer

$$h_m = \sin\varphi \cdot f_z \quad (15)$$

Where L – cut length (mm), n – revolution (rpm), t – time of cutting (s) - (Table 1), z – number of teeth.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

At least 25 cuts were made with each saw blade, in both hard and soft wood. A selected measurement record of cutting spruce with the PK36 blade is presented in Figure 5.

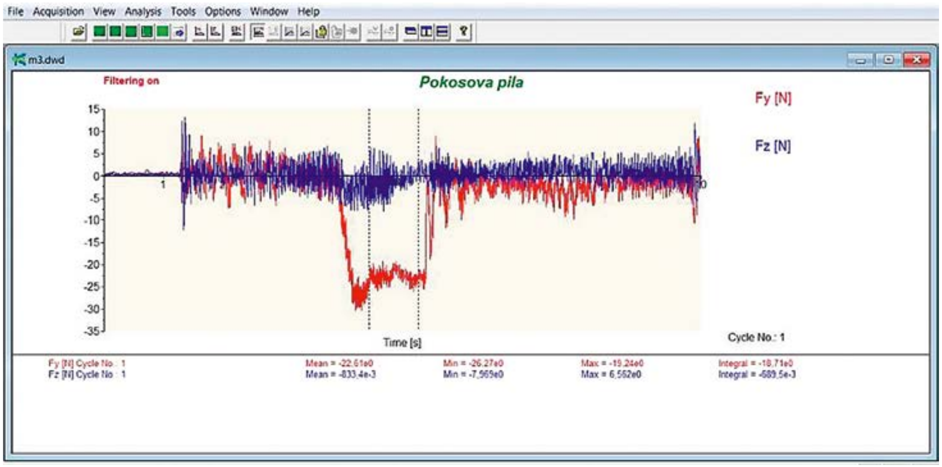


Figure 5 Record of measured forces F_y and F_z in DynoWare application - cutting spruce with saw blade PK36
Slika 5. Zapis izmjerenih sila F_y i F_z u aplikaciji DynoWare – piljenje smrekovine listom pile PK36

The DynoWare application of the Kistler measuring system allows direct analysis of the measured data in the displayed graph, i.e. it is possible to determine the mean values of the forces F_y (red progress) and F_z (blue progress) and the time of cutting t, from the measured selected progress of constant cutting.

The measured values can be stored in the memory and further processed in the form of tables or graphs, see Figure 6. For better clarity, the forces in the bar chart are expressed by the opposite sign than usually recorded by the Kistler dynamometer, in perspective of the direction of the force action on the saw handle.The measured forces F_y, F_z and the time of cutting t, can be further used to calculate other dynamic and kinematic parameters and construct Ernst-Merchant diagrams for individual blades and to assess the effect of the tooth count on the cutting process (in this paper, diagrams are only constructed for hardwood).

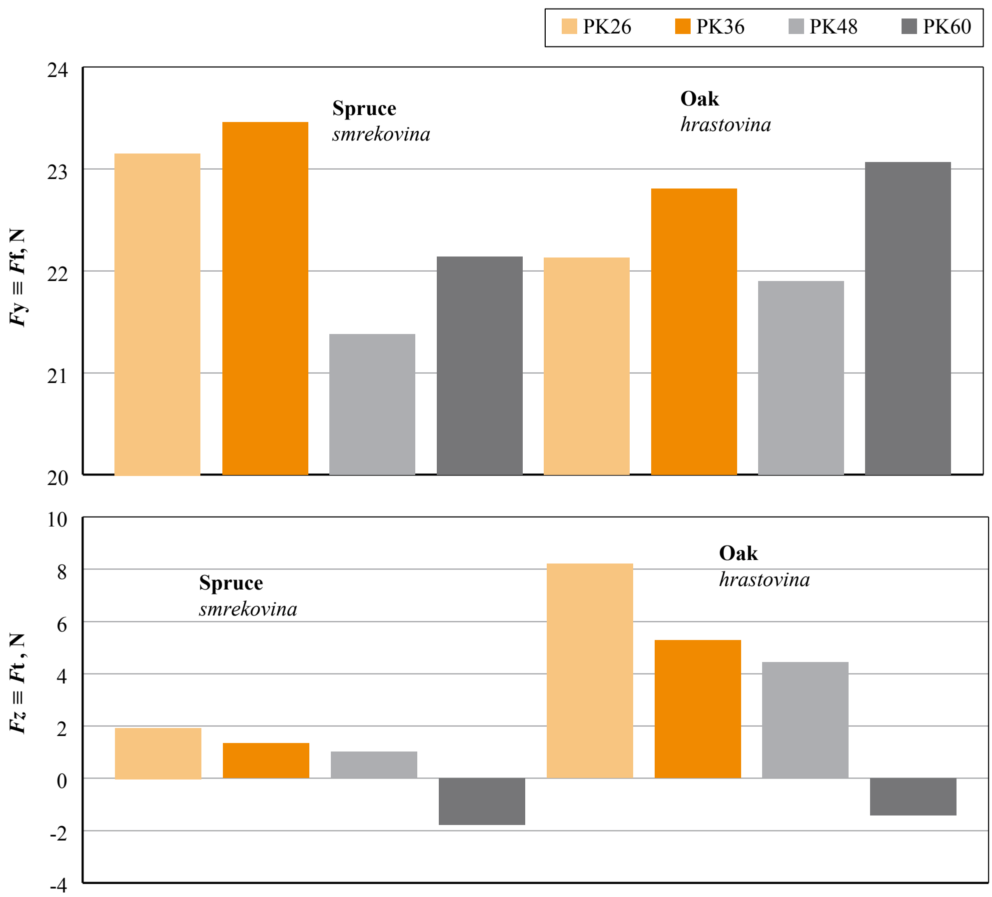


Figure 6 Mean values of forces for individual saw blades and woods
Slika 6. Srednje vrijednosti sila za pojedine listove pila i za ispitivane vrste drva

Table 1 Input and calculated values to generate Ernst-Merchant diagram for oak cutting
Tablica 1. Ulazne i izračunane vrijednosti za generiranje Ernst-Merchantovim dijagramom pri piljenju hrastovine

a	148 mm		PK26	PK36	PK48	PK60
e	50 mm	F_y, N	22.1	22.8	23.1	21.9
R	150 mm	F_z, N	8.2	5.9	4.4	-1.4
L	80 mm	*t*, s	1.3	2.1	2.5	6.7
Tooth angle - entry into the workpiece ψ_1, ° *Kut zuba – pri ulasku zubi u zahvat* ψ_1, °			9.367			
Tooth angle - exit to the workpiece ψ_2, ° *Kut zuba – pri izlasku zubi iz zahvata* ψ_1, °			49.207			
Tooth position in the middle chip thickness φ, ° *Položaj zuba pri srednjoj debljini strugotine* φ, °			29.287			
Resulting (active) force F_a, N / *Rezultantna (aktivna) sila* F_a, N			23.6	23.4	23.5	22.0
Angle between active force vector and feed force vector θ, ° *Kut između vektora aktivne sile i vektora posmične sile* θ, °			20.329	13.023	10.859	-3.733
Angle between cutting and active force η, ° *Kut između sile rezanja i aktivne sile* η, °			8.957	16.264	18.428	33.020
Cutting force F_c, N / *Sila rezanja* F_c, N			23.4	22.4	22.3	18.4
Length of tooth path in workpiece (length of layer taken) *l*, mm *Duljina putanje zuba u obratku (duljina luka zahvata) l,* mm			104.297			
Number of teeth z^l per length of layer taken *Broj zubi u zahvatu,* z^l *po duljini luka zahvata*			2.877	3.984	5.312	6.640
Cutting force per tooth , N / *Sila rezanja po zubu,* , N			8.1	5.6	4.2	2.8
Feed speed v_f, m/min^1 / *Posmična brzina* v_f, m/min			3.609	2.254	1.912	0.717
Feed per tooth f_z, mm / *Pomak po zubu* f_z, mm			0.037	0.023	0.019	0.007
Mean thickness of the cutting layer h_m, mm *Srednja debljina odvojene strugotine* h_m, mm			0.019	0.012	0.010	0.004

When manually feeding the workpiece at a mean constant feed force, the feed per tooth f_z and mean thickness of the cutting layer h_m depend not only on the current cutting resistance of the cut material, but also the number of cutting edges that simultaneously remove a chip from the workpiece. When cutting oak, as opposed to spruce, the cutting time gets significantly longer, the feed per tooth and chip thickness decrease, the tooth gradually loses the ability to form a chip. The calculated values presented in Table 1 lead to a partial conclusion that, with a slightly decreasing total cutting force F_c, the cutting force per tooth decreases proportionally with an increasing number of saw teeth (Figure 7). This phenomenon is also known from previous research by authors (Stewart, 1979; Porankiewicz *et al.*, 2007) who dealt with machine feeds with fixed feed per tooth. When using a constant feed rate (simulation of machine feed), the feed force applied constantly "pushes" the saw teeth into the cut at the same speed, so the blade tooth is forced to form a chip and, when

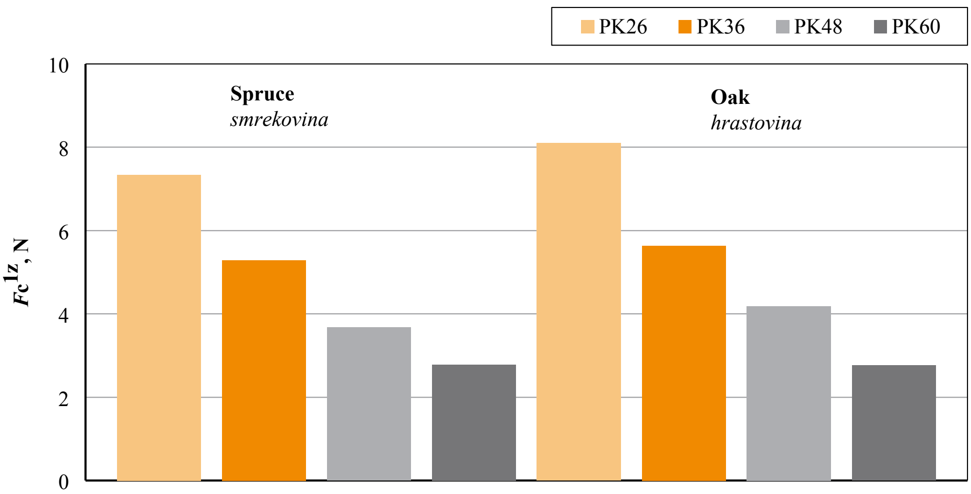


Figure 7 Cutting force per tooth
Slika 7. Sila rezanja po zubu

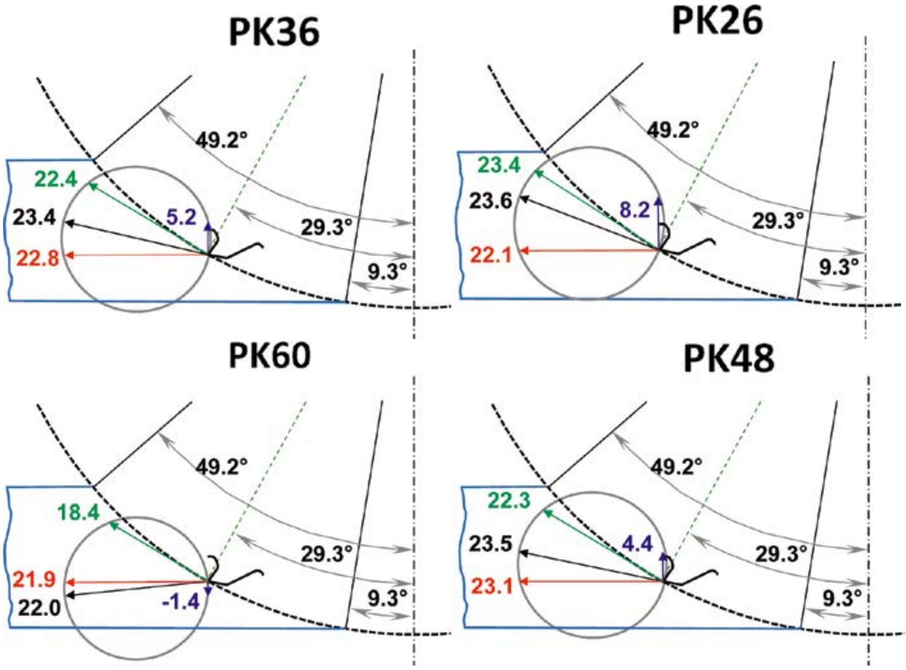


Figure 8 Graphical comparison of forces in Ernst-Merchant diagrams - oak cutting
Slika 8. Grafička usporedba sila u Ernst-Merchantovu dijagramu – piljenje hrastovine

the cutting resistance of the cut material increases, the cutting power increases too. This conclusion is in line with previous studies by Prokeš (1982) and Pernica (1999). Other studies by Kminiak and Kubš (2016) and Kminiak and Siklienka (2016), which dealt with issues similar to this research, were based on an evaluation of the cutting power depending not only on the saw tooth count, but also on the cutting direction. In the case of tangential-transversal cutting model, the authors concluded that the total cutting power slightly decreases with an increasing tooth count, which is in harmony with our results.

However, when evaluating cutting power only, without a detailed analysis of the forces and kinematic parameters of the saw blade tooth, it is difficult to determine the effect of the number of teeth on the total cutting process, since the total cutting force and power change only slightly when blades with different tooth counts are used.

Ernst-Merchant diagrams (Figure 8) show that all forces slightly decrease with an increasing tooth count in a saw blade. In our experiment, a constant feed force of 30 N was used, which is the common mean value of manual mitre saw arm shift in the case of a saw of the size used in the experiment. However, in the cutting process, part of this force is consumed by the chip formation and part to overcome friction in the cable mechanism and the saw feed mechanism; therefore, the measured values of the feed forces, F_f, are slightly lower, by 6 to 8 N.

Also, the overview of forces in Ernst-Merchant diagrams for individual blades (Figure 8) indicates a fundamental change in the direction of thrust force F_t in the case of PK60 blade. The thrust force changed the direction of action and began to push the workpiece against the table. The resultant active force F_a of the cutting process also acts towards the material. Based on these values, we can form a hypothesis that the teeth of PK60 blade form a chip with difficulty and they compress the layer of material taken under the clearance face. This is also related to the relatively small feed per tooth f_z = 0.007 mm and the mean thickness of the cutting layer h_m = 0.004 mm (Table 1), the size of which is below the current radius of tooth blade edge (Figure 1). It could be that there really is a small negative angle in the portion of the saw tooth at the tip where the rake angle becomes negative. Statistical analysis of variance ANOVA, Rak (2020), was used for an unbiased assessment of the effect of the different number of saw blade teeth on the cutting parameters in the cross-cutting of spruce and oak planks using constant feed force.

The probability value P is lower than 0.05. Therefore, there is a statistical significance between the data, and Scheffe's multiple comparison was used. Scheffe's multiple comparison (Tab. 3) shows the statistical difference in the PK60 saw blade. It differs significantly from the other blades. Cutting with blades PK24 and PK48 differs from each other and they both differ from the PK60. Based on the test results, but also the size and direction of the forces applied, the acceptable feed per tooth f_z = 0,023 mm and the mean uncut chip thickness h_m = 0,012 mm, it can be concluded that the PK36 saw blade with 36 teeth (Freud LU2A 1900) is the most suitable for the cross-cutting process of the timber studied.

Table 2 Results of statistical analysis of variance for cross-cutting of spruce
Tablica 2. Rezultati statističke analize varijance za izmjerene sile pri poprečnom piljenju smrekovine

FACTOR

Groups *Listovi pila*	**Count** *Broj rezova*	**Sum** *Zbroj*	**Average** *Srednja vrijednost*	**Variance** *Varijanca*
PK26	3836	7286.15	1.90	92.81
PK36	4253	5668.07	1.33	96.86
PK48	5122	5183.59	1.01	142.79
PK60	7467	-13313.68	-1.78	80.96

ANOVA

Source of variation *Izvor varijacija*	***SS***	***df***	***MS***	***F***	***P-value***	***F crit***
Between groups *između listova pile*	49252.1781	3	16417.39	161.36	2.14E-10[3]	2.61
Within groups *unutar lista pile*	2103391	20674	101.74			
Total *Ukupno*	2152643.18	20677				

Table 3 Scheffe's multiple comparison
Tablica 3. Scheffeova višestruka usporedba

			PK24	PK36	PK48	PK60
	Count *Broj rezova*		3836	4253	5122	7467
		Average *Srednja vrijednost*	1.90	1.33	1.01	-1.78
PK24	3836	1.90	0.00	2.52	**4.12**	**18.38**
PK36	4253	1.33	2.52	0.00	1.53	**16.08**
PK48	5122	1.01	**4.12**	1.53	0.00	**15.27**
PK60	7467	-1.78	**18.38**	**16.08**	**15.27**	0.00

4 CONCLUSIONS
4. ZAKLJUČAK

Choosing the tooth count of the saw blade used is a fairly important step when using a saw. It affects not only the energy aspect, but also the tooth wear and the quality of the cut, Mikleš *et al*. (2010).

The results of this paper confirm the fact that, in the case of manual feed by constant force, the energy performance of the cutting process is directly dependent on the interaction of the material properties and the tool construction – especially the number of actively cutting teeth, Schajer and Wang (2002). In this process, the said interaction adjusts the feed per tooth and the nominal uncut chip thickness, making the process analysis much more complicated.

With an increasing tooth count, the constant feed force is divided among a larger number of cutting edges that concurrently participate in the cutting. If the force per a cutting edge is too small, the material may not be cut at all. In general, the more teeth are cutting, the lower force is applied to a tooth blade in the cutting process and the tooth is less forced to form a chip. There is a risk that the cutting process will stop, the workpiece will get burnt and the tool will get heavily worn.

Another finding was that, with an increasing tooth count and a decreasing mean uncut chip thickness, the vector of the resultant (active) force of the cutting process, F_a, "turns" into the material cut. This causes greater elastic plastic deformation of the material under the tooth edge, there is more friction, especially under the tooth clearance face, and the chip formation is reduced or stopped.

If the quality of the cut is not critical, it is preferable to use blades with a medium tooth count for mitre saws when cutting wood transversely, from the perspective of energy demands.

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Eser Sözen[1]

Determination of Physical and Mechanical Properties of Waste Paper Boards Supported by Wood Chips and Chopped E-glass Fiber

Određivanje fizičkih i mehaničkih svojstava ploča izrađenih od otpadnog papira i ojačanih drvnim iverjem te usitnjenim staklenim vlaknima

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ABSTRACT • *Today, legal regulations, along with social awareness, have made waste management a necessity. Depending on the type of waste, technological developments have enabled an increase in the number of approaches, e.g., reusing, recycling, and manufacturing of different products. In this study, low-density boards were produced using different proportions of office waste paper, wood chips, and chopped E-glass fiber. Waste paper and glass fiber formed the middle layer of the boards and wood chips comprised the surface layers. The density, water absorption, and thickness swelling properties of the boards were investigated. Among the mechanical properties, the modulus of rupture, modulus of elasticity, and internal bond strength were determined. The use of wastepaper led to a reduction in the modulus of rupture and modulus of elasticity of the boards. An increase in the glass fiber ratio contributed positively to the water absorption and thickness swelling properties, whereas it directly led to decreases in the internal bond strength, and the modulus of rupture and modulus of elasticity values. Nonetheless, the low-density boards were able to meet the minimum modulus of rupture and modulus of elasticity specified in ANSI 208.1 standards, while only one variation met the IB requirements.*

KEYWORDS: *office waste paper; paper board; wood chips; chopped glass fiber*

SAŽETAK • *U današnje je vrijeme zakonska regulativa, uz društvenu svijest, gospodarenje otpadom učinila nužnošću. Ovisno o vrsti otpada, tehnološki je razvoj omogućio povećanje broja pristupa gospodarenju otpadom, npr. pri ponovnoj uporabi, recikliranju i proizvodnji različitih proizvoda. Za ovo su istraživanje izrađene ploče niske gustoće s različitim udjelima otpadnoga uredskog papira, drvnog iverja i usitnjenih staklenih vlakana. Pritom su otpadni papir i usitnjena staklena vlakna iskorišteni za izradu središnjeg sloja, a drvno je iverje upotrijebljeno za vanjske slojeve ploča. Izrađene su ploče ispitane i utvrđena je njihova gustoća, moć upijanja vode i debljinsko bubrenje.*

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Dodatno su određeni modul loma, modul elastičnosti i čvrstoća raslojavanja. Rezultati ispitivanja pokazali su da je uporaba otpadnog papira pridonijela smanjenju vrijednosti modula loma i modula elastičnosti. Istodobno je povećanje udjela usitnjenih staklenih vlakana imalo pozitivan učinak na vrijednosti upijanja vode i na debljinsko bubrenje, ali je izravno utjecalo i na smanjenje čvrstoće raslojavanja, modula loma i modula elastičnosti. Ipak, izrađene ploče niske gustoće zadovoljile su minimalne zahtjeve norme ANSI 208.1 glede modula loma i modula elastičnosti, a samo je jedna skupina eksperimentalno izrađenih ploča zadovoljila uvjete čvrstoće raslojavanja.

KLJUČNE RIJEČI: *otpadni uredski papir; ploče od papira; drvno iverje; usitnjena staklena vlakna*

1 INTRODUCTION
1. UVOD

Today's increasing population, developing economy, rapid urbanization, and rising living standards have accelerated the production of solid waste worldwide, especially in developing countries (Minghua *et al.,* 2009; Guerrero *et al.,* 2013). Many countries are enacting measures and new policies against environmental problems caused by the management of diminishing resources and waste (Kızıltaş *et al.,* 2020). Public awareness of recycling and waste management has recently emerged in many countries. Following this stage, the need arises to integrate materials obtained from recycling into value-added products. Different recycled materials can be used in a variety of ways, revealing the potential of these products to provide benefits to national economies (Sözen and Gündüz, 2021).

Researchers have defined "zero waste" as a new paradigm that promotes the recovery of resources from the overall waste stream, reducing consumption, and applying a life-cycle approach to product design (Dinshaw *et al.,* 2006). The term has been used frequently since its inception and has played a role in creating social awareness, especially in high-income countries. High-income countries, which comprise 16 % of the world's population, account for 34 % (683 million tons) of the total amount of solid waste worldwide (The World Bank, 2020). In addition, awareness of waste recycling is quite high in these countries. The European Paper Recycling Council (EPRC) reported that in 2019, eight European Union countries had recovered 60 % and 15 countries 70 % of their waste paper. The total rate of recycled waste paper has been reported as reaching 72 % (EPRC, 2019).

Paper is most commonly used as a medium for writing and printing. In addition, it is used as a packaging material and actively used in the industrial and construction sectors. These and many other uses make it almost impossible to stop the production of paper. (Thaemngoen *et al.,* 2004). Waste paper is paper that meets production requirements and needs disposal. Waste paper is sourced from offices, homes, public facilities, commercial enterprises, and industrial production facilities (Oriyomi *et al.,* 2015). In particular, 90 % of office waste is recyclable (Okino *et al.,* 2000). The recycling of waste paper in Europe is an excellent example, as it provides economic circulation and significant environmental gains (Adu *et al.,* 2018).

Okino *et al.,* (2000) examined the physical and mechanical properties of low-density boards obtained by using three different types of waste paper (white office waste paper, newspaper, and mixed magazines) and two different thermosetting glues (urea formaldehyde and tannin paraformaldehyde). As a result of the study, the modulus of elasticity (*MOE*), modulus of rupture (*MOR*), and internal bond (*IB*) values of the 0.55 g/cm^3 density boards obtained using 100 % waste paper and urea formaldehyde glue were determined as 2.0 (GPa), 5.3 (MPa) and 137 (kPa), respectively. Moreover, in the same study, the boards obtained from the office paper showed the best thickness swelling properties.

Bringing the waste recovered through recycling into the economy is as important as recycling. Depending on the characteristics of the recycled materials, significant contributions to the economies of local governments and countries can be made by ensuring sustainability and producing high value-added products. The waste paper has advantages as a material in terms of both collection and transformation into different products using various production methods. Many industrial products have also been produced such as fiber-cement composites (Thaemngoen *et al.,* 2004), granular and sheet activated carbon from wood waste (Malikov *et al.,* 2007; Yorgun *et al.,* 2009), paperboard briquettes (Odusote *et al.,* 2016), pine tree bark insulation board (Ozlusoylu and Istek, 2019; Istek and Ozlusoylu, 2019), and particleboard produced from chainsaw dust (Istek *et al.,* 2020) and waste sweet bay wood (Yazici, 2020).

This study aimed to produce low-density board using office waste paper. For this purpose, office waste paper and chopped glass fiber were used in different proportions and the physical and mechanical properties of the boards were then compared with the relevant standards.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Material
2.1. Materijal

In this study, office waste paper (WP), wood particles (P) and chopped E-glass fiber (GF) were used as

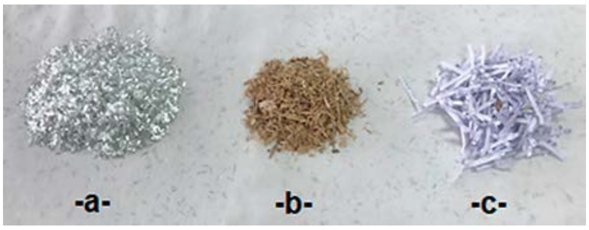

Figure 1 Materials used in the study: (a) chopped glass fiber, (b) wood chips, (c) office waste paper
Slika 1. Materijali upotrijebljeni u istraživanju: a) usitnjena staklena vlakna, b) drvno iverje, c) otpadni uredski papir

Table 1 Properties of urea formaldehyde glue used in the study
Tablica 1. Svojstva urea-formaldehidne smole primijenjene u istraživanju

Properties *Svojstvo*	**Values** *Vrijednost*	**Unit** *Jedinica*
pH / *pH vrijednost*	7.5 – 8.5	-
Solid matter ratio *udio suhe tvari*	54.00 – 56.00	%
Viscosity *viskoznost*	100 – 200	at 20 °C, cps
Density / *gustoća*	1.220 – 1.230	at 20 °C, kg/m³
Gel time *vrijeme želiranja*	15 – 25	at 100 °C, s

materials. Office waste paper, used on both sides, was recycled within the scope of the “zero waste” project of Bartın University (in Turkey). Before being used, the waste paper, weighing 80 g/m², was shredded using an office shredder.

Wood particles in the form of chips were used as the outer layers in the production of the particleboard. These chips were obtained from an industrial production company (Kronospan Forest Products Industry and Trade Inc., Kastamonu, Turkey) and consisted of 50 % coniferous (*Pinus sylvestris*) and 50 % deciduous (*Fagus orientalis* L.) wood chips that varied in size between 2 and 10 mm. The waste paper and wood chips were dried in an oven to a moisture content of 4 - 4.5 % before the boards were produced. During drying, it was observed that the moisture of the wood chips was reduced at a faster rate.

Chopped E-glass fibers were supplied by the Omnis Composite Industry and Trade Inc. (Istanbul, Turkey). As glass fibers do not hold moisture, they were not subjected to any drying process. Images of the materials used in the study are shown in Figure 1.

Urea formaldehyde glue (POLIURE 7455) supplied by Polisan Chemistry Trade Inc. (Istanbul, Turkey) was used. No hardening agent was added to the glue. The amount of glue (solid matter ratio) was 10 % of the total produced board weight. The properties of the urea formaldehyde glue, as listed in the MSDS (Material Safety Data Sheet) catalog of the manufacturer, are given in Table 1.

2.2 Methods
2.2. Metode

The boards produced in the study consisted of three layers: two surface layers and a middle layer. Wood chips alone were used in the surface layers of the boards, which constituted 50 % of the total board weight. Different proportions of waste paper and chopped glass fiber strands were used in the middle layer of the boards. In all variations, the board thickness produced was 12 mm. Table 2 shows the ratios and weights of the board variations created within the scope of the study.

2.3 Production of boards
2.3. Izrada ploča

The wood particles (P) and office waste paper (WP) were brought to 4-4.5 % moisture content in the oven and then held for 2 h to be cooled to room temperature. The production phase was carried out according to the variations given in Table 2. Wood particles alone were used in the production of the control samples. Therefore, the control boards consisted of a single layer. All other variations were composed of three layers, i.e., two surface layers and a middle layer.

Table 2 Ratios and weights of board variations in the study
Tablica 2. Udjeli i mase pojedinih sirovina od kojih su izrađene ispitivane ploče

Variations *Varijacije*	**Surface** *Vanjski sloj*		**Middle layer** *Središnji sloj*				**Surface** *Vanjski sloj*		**Code** *Oznaka*
	Wood chips (Particles) (P) *Drvno iverje (P)*		**Waste paper (WP)** *Otpadni papir (WP)*		**Glass fiber (G)** *Staklena vlakna (G)*		**Wood chips (Particles) (P)** *Drvno iverje (P)*		
	g	%	g	%	g	%	g	%	
P100 (Control)	500	50	-	-	-	-	500	50	P1
P50W50	250	25	500	50	-	-	250	25	P5W5
P50W45G5	250	25	450	45	50	5	250	25	P5W45G5
P50W40G10	250	25	400	40	100	10	250	25	P5W4G1
P50W30G20	250	25	300	30	200	20	250	25	P5W3G2

The wood particles (P) used in the surface layers of all boards except the control samples constituted half of the weight of the mat. The other half consisted of the chopped glass fiber (G) used with waste paper (WP). The production setup for the variations (excluding the controls) is shown in Figure 2. The materials, in their specified weights, for the two surface layers and one middle layer (Figure 2A) were processed in two different concrete mixers, respectively. The materials were glued using urea formaldehyde (solid matter rate, 55±1 %) (Figure 2B). A pressure paint gun was used and care was taken to ensure that the gluing was homogeneous. The glued materials then proceeded to the laying process.

A 400 mm × 400 mm mold was used in the preparation of the mat. This mold was placed on top of a thin metal plate covered by baking paper. In the laying process, first of all, wood particles (25 %) were spread homogeneously and carefully to form the substrate. The material/s (50 %) forming the middle layer was then spread over the substrate. Finally, the same (25 %) wood particles as those used in the lower layer were spread to form the upper layer. Finally, baking paper and a thin metal plate were placed on the mat, respectively. Pre-pressing at up to 10 % of the hot press pressure (3 kg/cm^2) was applied to form the mat in preparation for hot pressing (Figure 2C). The formed mat (Figure 3) was hot pressed for 6 min (Figure 2D). To ensure uniform board thickness, 12 mm-thick metal laths were used at the hot press stage (Figure 2D-e).

The production conditions of the boards are given in Table 3, and the appearance of the board before and after hot pressing is shown in Figure 3.

Table 3 Production conditions of boards
Tablica 3. Parametri izrade ploča

Characteristics *Svojstvo*	**Production conditions** *Parametar*	**Unit** *Jedinica*
Board thickness *debljina ploča*	12	mm
Board width – length *širina i dužina ploča*	400 × 400	mm
Target density *ciljana gustoća ploča*	0.52	g/cm^3
Middle layer material/s ratio *udio središnjeg sloja*	50	%
Surface layer chip ratio *udio iverja u vanjskim slojevima*	50	%
Press temperature *temperatura prešanja*	180	°C
Press pressure / *tlak prešanja*	30	kg/cm^2
Total press time *ukupno vrijeme prešanja*	180	s
Resin ratio / *nanos smole*	10	%

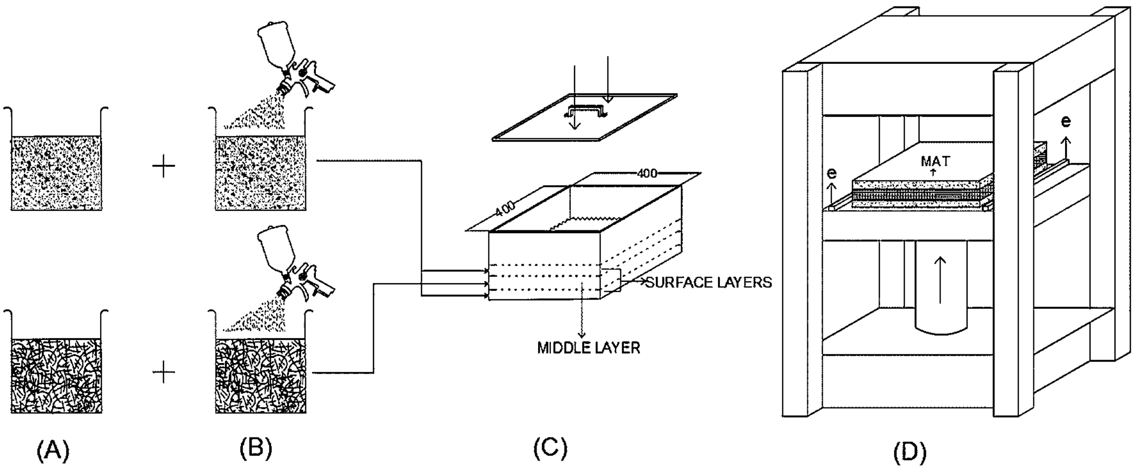

Figure 2 Production stages of boards
Slika 2. Faze izrade ploča

Figure 3 Hot-pressed board: (a) pre-hot pressing, (b) post hot pressing
Slika 3. Eksperimentalne ploče: a) prije vrućeg prešanja, b) nakon vrućeg prešanja

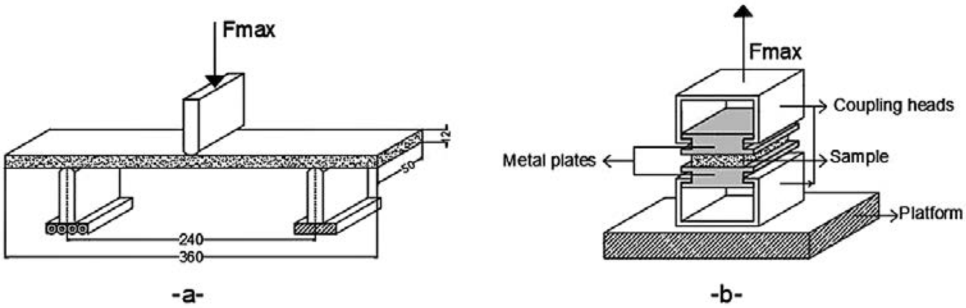

Figure 4 Test configuration: (a) bending strength, (b) internal bond strength
Slika 4. Način ispitivanja: a) savojne čvrstoće, b) vlačne čvrstoće

Boards taken from the hot press were left to cool at room temperature using 20 mm ×20 mm stacking laths. After 48 h, the board edges were evened using a circular saw.

2.4 Preparation of test samples
2.4. Priprema ispitnih uzoraka

Sample preparation processes began 10 days after the production of the boards. Samples were cut from different parts of the produced boards and investigations were carried out in accordance with the TS EN 326-1 (1999) standard. The oven-dry (D_0) and 12 % (air-dry) (D_{12}) density values of the samples were determined in accordance with the TS EN 323 (1999) standard. In order to determine the oven-dry density, the samples cut in accordance with the standards were kept at (103 ±2) °C until their weight was stabilized. Determination of thickness swelling after immersion in water (24 h) was carried out in accordance with TS EN 317 (1999). Modulus of elasticity in bending (MOE) and bending strength tests were carried out in accordance with the principles specified in TS EN 310 (1999). The loads in the elastic region (F2-F1) and the difference in the deformation amounts corresponding to these loads (d2-d1) were used to calculate the MOE. Care was taken to ensure that these values were between 10 % and 40 % of the maximum load.

2.5 Test method
2.5. Metoda ispitivanja

The setups for the bending strength and IB strength tests applied to the samples are shown in Figure 4a and Figure 4b, respectively. Samples in dimensions of 50 mm × 360 mm ×12 mm were prepared in accordance with the specified standard for bending strength tests. 50 mm × 50 mm ×12-mm samples were used to determine the IB strength (Figure 5). Ten replications were made for each variation of bending strength and internal bond tests. Silicon was used to bond metal plates to the surfaces of these samples. Internal bond strength tests were carried out by attaching these plates to the coupling heads.

Figure 5 Internal bond strength test samples
Slika 5. Uzorci za ispitivanje čvrstoće raslojavanja

Eqs. (1), (2), and (3) were used to calculate bending strength (σ_{B}), *MOE* (σ_{MOE}) and *IB* (σ_{IB}) strength, respectively.

$$\sigma_B = \frac{3}{2}\frac{F \cdot L_S}{b \cdot h^2} \tag{1}$$

$$\sigma_{MOE} = \frac{\Delta F \cdot L_S^3}{4 \cdot \Delta d \cdot b \cdot h^3} \tag{2}$$

$$\sigma_{IB} = \frac{F}{A} \tag{3}$$

Here, F is the maximum load value, L_S is the distance between the supports (240 mm), b is the width of the sample (50 mm), h is the height of the sample, ΔF is the difference of the two loads within the elastic limits, Δd is the deformation difference corresponding to the two loads within the elastic limits, and A expresses the surface area of the sample (50 mm × 50 mm = 2500 mm²).

2.6 Evaluation of data
2.6. Evaluacija podataka

The one-way analysis of variance (ANOVA) included in the Statistical Package for the Social Sciences (SPSS) software program was used to evaluate the data. Duncan's test was used to determine the differences between the groups.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Density
3.1. Gustoća

The density of the produced boards was measured at oven-dry (D_0) and air-dry (D_{12}) moisture con-

Table 4 Average density and standard deviation values of boards
Tablica 4. Prosječne gustoće ploča i standardne devijacije ispitivanih svojstava

Board codes *Oznake ploča*	**Density (D_{12})** *Gustoća (D_{12})*, g/cm³	**Standard deviation** *Standardna devijacija*	**Density (D_0)** *Gustoća (D_0)*, g/cm³	**Standard deviation** *Standardna devijacija*
P1	0.52	0.030	0.49	0.028
P5W5	0.52	0.050	0.49	0.048
P5W45G5	0.53	0.046	0.50	0.045
P5W4G1	0.53	0.049	0.50	0.050
P5W3G2	0.53	0.040	0.50	0.049

tent. To calculate the density of the boards, ten samples were used for each variation. No statistically significant difference was found among the board density values. The average density and standard deviation values of the boards are given in Table 4.

The air-dry density (D_{12}) of the boards was targeted as 0.52 g/cm³ at the production stage. The density measurements demonstrate that this goal was achieved. The variation of 0.01 in the P1 and P5W5 boards was thought to have been a result of chips lost during production. On the other hand, the standard deviation values show that the overlaying process had been carried out as homogeneously as possible. An average decrease of 6 % was observed in the oven-dry density (D_0) values. Istek and Sıradag (2013) stated that density changes of up to 10 % in particleboards do not significantly affect board properties. The decrease in density in the P5W4G1 and P5W3G2 variations containing glass fiber was less than in the other variations. This situation was thought to have been caused by the fact that the water absorption and water holding abilities of E-glass fiber are lower than those of wood chips and waste paper.

Table 5 Minimum requirements of ANSI 208.1 and EN 16368/LP1standards
Tablica 5. Minimalni zahtjevi definirani normama ANSI 208.1 i EN 16368/LP1

Properties *Svojstvo*	**ANSI 208.1**	**EN 16368/LP1**
MOE, N/mm²	500	950
MOR, N/mm²	2.8	7
IB, N/mm²	0.14	0.28

3.2 Modulus of Rupture (*MOR*) and Modulus of Elasticity (*MOE*)
3.2. Modul loma (*MOR*) i modul elastičnosti (*MOE*)

The average *MOR* and *MOE* values of the boards are shown in Figure 6. The *MOR* values show that reducing the amount of chips forming the board to 50 % (in all variations except P1) was effective in reducing the bending strength. Decreases in the *MOE* were in parallel with the decrease in bending strength.

The physical and mechanical properties of the boards were compared with the relevant standards. As the density of these boards was less than 600 kg/m³, they were classified as low-density boards. Thus, the boards were comparable to the American National Standards Institute for particleboard (LD2) for interior application (ANSI 208.1, 2016) and the European standard for lightweight particleboard for interior application (EN 16368/LP1, 2014). Table 5 gives the minimum *MOE*, *MOR*, and *IB* requirements of the relevant standards.

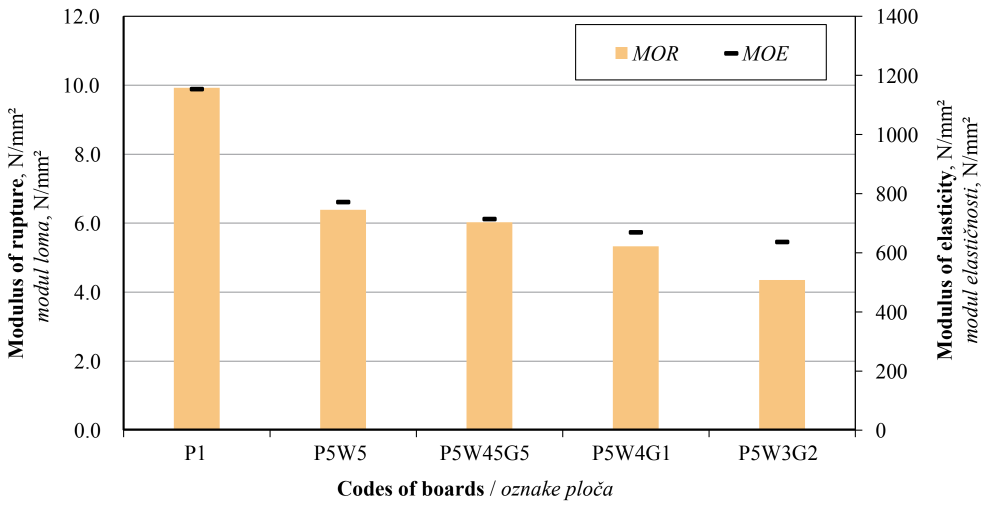

Figure 6 *MOR* and *MOE* values of boards
Slika 6. Vrijednosti modula loma i modula elastičnosti

The P1 variation showed the highest *MOR* (9.93 N/mm²) and *MOE* (1153 N/mm²) values. The use of chips alone in this variation was effective in increasing these values. The compatibility of the surface chips and glue used in particleboard production was an important factor. All of the produced variations met the minimum *MOE* value of 500 N/mm² specified in the ANSI A208.1 standard. On the other hand, except for P1, none of the other variations could reach the *MOE* value of 950 N/mm² specified in the EN 16368/LP1 standard.

The use of waste paper in the boards lowered the bending strength values. This decrease was 35 % in the P5W5 boards (which used the most waste paper) and 56 % in the P5W3G2 boards (which used the least waste paper). The waste paper shredded in an office shredder was a bulkier material than the glass fiber or wood chips. Therefore, it had a larger surface area. This was thought to have reduced the rate of glue per unit area during gluing. The distribution and amount of glue play an important role in board production. Okino *et al.* (2000) used 8 % and 12 % urea formaldehyde glue in low-density boards produced from office waste paper. The bending strength values of these boards were determined as 3.8 N/mm² and 5.3 N/mm², respectively.

The fillers such as $CaCO_3$ used in the production of white office paper were believed to be effective in the adhesion performance of the glue. Consequently, the weakening of the glue performance was effective in the decrease of the bending strength. The importance of glue-material compatibility was therefore revealed in the study. Although paper is manufactured from fibers (wood), additives and fillers cause significant changes in the structure of these fibers. In terms of adhesion performance, it was observed that, except for P1, the compatibility between the chips and the waste paper was not sufficiently achieved in the variations. Ellis *et al.* (1993), Massijaya and Okuma (1996), and Okino *et al.* (2000) reported that the mechanical properties in low-density boards produced using waste paper were boosted by an increase in the glue ratio.

The increase in the ratio of E-glass glass fiber used in the boards caused a decrease in the bending strength. The bending strength, which was 6.39 in the P5W5 board, was reduced to 6.03, 5.33, and 4.35 in the P5W45G5, P5W4G1, and P5W3G2 boards, respectively. The urea formaldehyde glue incompatibility observed with the waste paper was also seen with the E-glass glass fiber. In the literature, it is reported that, when glass fibers are chopped or in fabric form, they are compatible and can be used with epoxy. Valente *et al.* (2011) produced hybrid composites with thermoplastic polymers using wood flour and recycled chopped glass fiber. They reported that the chopped glass fiber increased the mechanical properties of the composites, and emphasized that the particle size and the hardness of the matrix and the bond area were effective in this situation. Various studies have stated that many factors can affect the mechanical properties in board production (Istek *et al.,* 2018; 2019).

Table 6 Duncan's test results of *MOR*
Tablica 6. Rezultati Duncanova testa za modul loma

Board codes *Oznake ploča*	**Number of samples** *Broj uzoraka*	**A**	**B**	**C**
P5W3G2	10	636.00		
P5W4G1	10	668.50	668.50	
P5W45G5	10	713.00	713.00	
P5W5	10		771.38	
P1	10			1152.9

A statistically significant difference in the bending strength among the variations was demonstrated by the ANOVA (F_{ratio} = 47.716; P_{value} = 0.000 < 0.05). Duncan's test was used to determine differences in *MOR* among the groups, as shown in Table 6.

3.3 Internal Bond (*IB*) Strength
3.3. Čvrstoća raslojavanja (*IB*)

Internal bond strength directly or indirectly affects the physical and mechanical properties of wood-based boards. The *IB* strength of the produced boards varied between 0.35 N/mm² and 0.04 N/mm². The *IB* strength and standard deviation values of the boards produced within the scope of the study are shown in Table 7.

In the *IB* strength tests, the highest value (0.35 N/mm²) was achieved by the P1 and the lowest (0.04 N/mm²) by the P5W3G2 variation. These values were in

Table 7 Internal bond strength and standard deviation values of boards
Tablica 7. Čvrstoća raslojavanja i standardne devijacije

Board codes *Oznake ploča*	**Number of samples** *Broj ispitanih uzoraka*	**F_{max} Average** *Prosjek maksimalne sile loma (F_{max})*	**Standard Deviation** *Standardna devijacija*	**IB Strength** Čvrstoća *unutarnje veze,* N/mm²
P1	10	904.2	±70.7	0.35
P5W5	10	184.8	±11.3	0.07
P5W45G5	10	172.2	±10.6	0.07
P5W4G1	10	146.2	±14.1	0.06
P5W3G2	10	101.4	±7.1	0.04

accordance with the 0.14 N/mm² and 0.28 N/mm² values specified in the ANSI 208.1 and EN 16368/LP1 standards, respectively. In fact, the P1 variation, which was produced totally from chips, reached a value close to the 0.4 N/mm² required in the TS EN 310 standard for particleboards. There was an 80 % decrease in the IB strength of the P5W5 and P5W45G5 variations. In these variations, the waste paper ratio constituted 50 % and 45 % of the board weight, respectively. The use of waste paper in the middle layer of the board was effective in the *IB* strength decreases. The increase in the amount of glass fiber was in parallel with the decrease in the *IB* strength of the boards. The urea formaldehyde glue used was compatible with the lignocellulosic materials (chips). However, it did not show the same compatibility with the waste paper, which is basically a lignocellulosic material. This was thought to have been caused by the fillers and additives used in the production of the office paper. The same incompatibility applied to the E-glass glass fiber.

Synthetic glass fibers have hydrophobic properties and therefore do not form adhesive bonds with wood. However, when these fibers are used with suitable resins, they give strength to the composite. These fibers are more commonly used with epoxy-based resins/glues (Ashori and Sheshmani, 2010; Gurunathan *et al.,* 2015; Wei and McDonald, 2016). When the failure types formed as a result of the IB strength tests, the ruptures were seen to occur from the middle layer (Figure 7a). Figure 7b shows the failure type resulting from the *IB* strength test.

During *IB* strength tests, if ruptures arise from the surface of the boards, the test is considered invalid. In the tests performed, all the ruptures, including those in the control sample, arose from the middle layer. The incompatibility of the waste paper with the urea formaldehyde glue bolstered the occurrence of breakouts from the middle layer. Okino *et al.* (2000) produced low-density boards using office waste paper, newsprint, and magazine paper. Using 8 % and 12 % urea formaldehyde glue, they determined the *IB* strength of the boards obtained from office paper to be 0.108 and 0.137 N/mm², respectively. They noted that these results could be explained by poor resin dispersion and/or low resin levels.

3.4 Water Absorption (*WA*) and Thickness Swelling (*TS*)
3.4. Upijanje vode (*WA*) i debljinsko bubrenje (*TS*)

The 24-h water absorption and thickness swelling values of the boards are shown in Figure 8.

The lowest *TS* rate (27 %) was that of the P1 board. No statistically significant difference was found among the *TS* ratios in the other variations. The reason that the P1 boards exhibited higher (negative) values than those specified in the standard was attributed to the fact that chips were not used in the middle layer. On the other hand, water was more effective among the chips distributed homogeneously within the outer layers. The 20 % increase in the E-glass fiber in the board content caused a 2 % decrease in the *TS* ratio. The water-resistant structure of the glass fiber was effective in this situation. Ranakoti *et al.* (2019) used epoxy resin as glue in boards composed of wood flour and glass fiber. As a result of the study, they reported a linear relationship between the wood flour ratio and *WA* rate.

When the *WA* samples were examined visually after the experiment, the majority of the *TS* were observed in a line between the layers. Figure 9 shows the separations between the layers after the *WA*-*TS* test.

Wood particles are hydrophilic because of the cellulose in their structure. This feature had a significant effect on the *WA* rate. In office paper formed of cellulosic fibers, additives and fillers, such as $CaCO_3$ incorporated at the production stage, caused changes in the properties of the cellulose. Thus, the *TS* ratios of the surface layers were higher than those of the middle layer.

Figure 7 Results of internal bond strength test: (a) middle layer, (b) failure type after rupture
Slika 7. Prikaz loma uzoraka pri ispitivanju čvrstoće raslojavanja: a) pukotina u središnjem sloju ploča, b) lomne površine nakon razdvajanja iskidanog uzorka

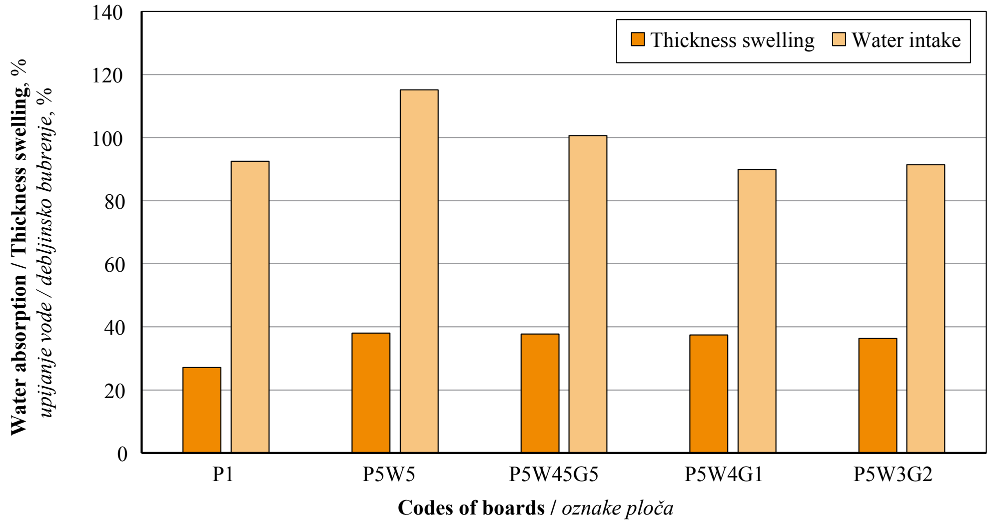

Figure 8 24-hour water absorption and thickness swelling values of boards
Slika 8. Vrijednosti upijanja vode i debljinskog bubrenja ploča nakon 24-satnog izlaganja djelovanju vode

Figure 9 Separation between layers after water absorption-thickness swelling test
Slika 9. Razdvajanje na granici srednjega i vanjskih slojeva ploča nakon izlaganja djelovanju vode

4 CONCLUSIONS
4. ZAKLJUČAK

In this study, in which the physical and mechanical properties of low-density boards obtained from wood particles, office waste paper and chopped glass fiber strands were examined, the following conclusions were reached.

It is possible to produce low-density boards using office wastepaper. Additives can be incorporated into these boards to give them different properties. However, the properties and compatibility of the materials used, as well as the areas of use, should be carefully determined.

The wood particles interacted better with office wastepaper, whereas E-glass, a synthetic fiber, did not exhibit sufficient interaction. The urea formaldehyde glue had a great impact on this situation. The use of resins or glues compatible with synthetic fibers would be especially effective in increasing mechanical properties.

All variations produced met the minimum *MOR* and *MOE* requirements specified in ANSI 208.1 standards (2016), while only variation P1 met the *IB* requirements.

In this study, low-density boards produced in a layered format may reveal physical and mechanical properties differing from boards produced in a homogeneous form.

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Physical and Mechanical Properties of Thermoplastic Composites Filled with Wood Flour of Underutilized Chaste Tree

Fizička i mehanička svojstva termoplastičnih kompozita punjenih drvnim brašnom rijetko upotrebljavane konopljike

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ABSTRACT • *The potential use of a lignocellulosic filler, Vitex agnus-castus plant (Chaste tree), which is a deciduous invasive shrub, in thermoplastic composites was investigated. The stems of chaste trees with a diameter of 5-10 cm from Mugla city, Western Turkey, were used for the study. The different amounts (0 to 50 wt%, by 10 % increments) of the wood flour passing through the screen openings of 0.237 mm were added to the polypropylene matrix. Premixed raw materials were put into the volumetric feeder of the twin-screw extruder. The extruder barrel temperature was gradually increased from 170 °C (feeding zone) to the die zone (190 °C) at a constant screw speed (40 rpm). Then, the dried granules were hot-pressed into the 4 mm thick WPC panels at 2 MPa and 190 °C for 5 min. 3 wt% of maleic anhydride grafted polypropylene (MAPP) was added as compatibilizer into the formulation. The WPCs showed an increase in the thickness swelling (0.58 to 5.68 %) as the amount of the filler increased from 10 to 50 wt% in the polypropylene. The bending strength of the polypropylene composites increased from 33.9 to 44.8 MPa as the amount of the chaste wood flour was increased to 30 wt%, but further increase caused the decrease in the tensile strength (25.7 MPa). As for the bending modulus, it increased from 815 to 3250 MPa when the wood content reached 50 wt%. The tensile modulus increased from 1690 to 2253 MPa when the wood content arised from 10 to 50 wt%. The tensile strength, tensile modulus, flexural strength and flexural modulus of the unfilled polypropylene were found to be 19.6 MPa, 1505 MPa, 30.2 MPa and 664 MPa, respectively. According to the test results, it was concluded that the 30-40 wt% of Vitex agnus-castus wood could be efficiently used in the polypropylene composites for the semi-building applications such as decking or siding. The evaluation of underused invasive chaste wood in the production of tWPC production may result in an effective way to utilize this resource.*

KEYWORDS: *Vitex agnus-castus; polypropylene; thermoplastic; composite; lignocellulosic filler*

SAŽETAK • *U radu je istražena potencijalna primjena lignoceluloznog punila u termoplastičnim kompozitima od biljke Vitex agnus-castus (konopljike), koja je listopadni invazivni grm. Stabljike konopljike promjera 5 – 10 cm nabavljene su iz grada Mugla, iz zapadne Turske. U polipropilensku matricu dodane su različite količine drvnog brašna (0 – 50 wt. %, uz povećanje od 10 %) koje je prolazilo kroz sito otvora 0,237 mm. Prethodno pomiješane*

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sirovine stavljene su u lijevak dvovijčanog ekstrudera. Temperatura spremnika ekstrudera postupno je povećavana od 170 °C (zona uvlačenja) do 190 °C (zona istiskivanja), uz konstantnu brzinu vijka (40 okr./min). Zatim su osušene pelete 5 min vruće prešane pri 2 MPa i 190 °C u oblik WPC ploče debljine 4 mm. Kao kompatibilizator formulaciji je dodan MAPP (3 wt. %). Kako se količina punila u polipropilenu povećavala s 10 na 50 wt. %, kompoziti su pokazivali povećanje debljinskog bubrenja (od 0,58 do 5,68 %). S povećanjem količine čistoga drvnog brašna na 30 wt. %, čvrstoća na savijanje polipropilenskih kompozita povećala se s 33,9 na 44,8 MPa, ali daljnje je povećanje dovelo do smanjenja vlačne čvrstoće (25,7 MPa). Kada je udio drva dosegnuo 50 wt. %, modul savijanja povećao se s 815 na 3250 MPa. Pri povećanju udjela drva s 10 na 50 wt%, modul elastičnosti pri vlačnom se naprezanju povećao s 1690 na 2253 MPa. Utvrđeno je da su vlačna čvrstoća, modul elastičnosti pri vlačnom naprezanju, čvrstoća na savijanje i modul na savijanje polipropilena bez punila redom 19,6 MPa, 1505 MPa, 30,2 MPa i 664 MPa. Prema rezultatima ispitivanja, zaključeno je da se 30 – 40 wt. % drva konopljike može učinkovito iskoristiti u polipropilenskim kompozitima za primjenu u graditeljstvu kao što su podne ili zidne obloge. Istraživanje slabo iskorištene konopljike u proizvodnji WPC-a može rezultirati učinkovitim načinom upotrebe te sirovine.

KLJUČNE RIJEČI: *Vitex agnus-castus; polipropilen; termoplastični kompozit; lignocelulozna vlakna*

1 INTRODUCTION
1. UVOD

Chaste tree (*Vitex agnus-castus*), which is a deciduous shrub L., belongs to the *Lamiaceae* family and is naturally grown in tropical and sub-tropical regions, Mediterranean area, Asia, and North Africa. It reaches up to 3 m, with pleasingly aromatic foliage. It was listed in the invasive plant atlas of the United States according to the U.S Forest Service (Reichard, 1994). Chaste tree is one of the most used medicinal plants with beneficial effects on human health. It also attracts butterflies and bees, and it makes an excellent honey plan. The flowers, leaves and seeds of the Chaste tree are used for herbal and medical applications. Branches of Chaste tree are used to make baskets and molds (kelter), especially to store or carry fruits (Souto *et al.*, 2020). Chaste wood is not evaluated in the group of high value-added products, such as thermoplastic composites, or wood-based panels, such as particleboards, although it is a broad spreading tree species.

Table 1 Chemical properties of wood of *Vitex agnus-castus* (Ceviz, 2016)
Tablica 1. Kemijska svojstva drva konopljike (Ceviz, 2016.)

Constituent *Sastavnica*	**Value, %** *Vrijednost, %*
Holocellulose / *holoceluloza*	74.64
Cellulose / *celuloza*	53.58
α-cellulose / *α-celuloza*	43.68
Lignin / *lignin*	21.27
Extractives / *ekstraktivi*	2.53
Ash / *pepeo*	2.79
Cold water solubility *topljivost u hladnoj vodi*	11.47
Hot water solubility *topljivost u vrućoj vodi*	12.42
Solubility in %1 NaOH solution *topljivost u 1 %-tnoj otopini NaOH*	24.33

Lignocellulosic materials contain different amounts of cellulose, hemicellulose, lignin, and extractives. The density, fiber properties (length, cell wall diameter, etc.), chemical composition, holocellulose, and lignin content of wood and non-wood species range widely according to age, soil properties, climate conditions, and geographic locations. These characteristics significantly influence technological properties, thermal, and acoustic properties of wood (Rowell *et al.,* 2012). The chemical properties of the chaste wood are given in Table 1. The cellulose and lignin contents of the chaste wood were found to be 53.5 % and 21.2 %, respectively (Ceviz, 2016). The cellulose and lignin are the structural components of wood and significantly affect the mechanical properties of wood, hence they have an impact on the quality properties of wood plastic composites (WPCs). The characteristics of lignocellulosic materials considerably affect the quality properties of WPCs (Mu *et al.,* 2018). Thus, it is necessary to understand physical and mechanical behavior of wood and non-wood materias before using it in thermoplastics. There is a number of experimental studies on the use of wood such as western red cedar wood (Clemons and Stark, 2009), paulownia wood (Ayrilmis and Kaymakci, 2013a), poplar wood (Nourbakhsh and Ashori, 2008); non-wood such as flax and sisal f.bers (Bax *et al.,* 2008), bamboo fibers (Lee and Wang, 2006); and agricultural waste such as rice husk (Wang and He, 2019), walnut shell (Ayrilmis *et al.*, 2013b), sunflower stalk (Kaymakci *et al.*, 2013) as lignocellulosic filler in thermoplastics. These studies showed that the use of lignocellulosic fillers in thermoplastics, such as polypropylene and polyethylene, could contribute to economic growth and environmental sustainability.

In recent years, the number of WPC manufacturers has increased due to great advantages of lignocellulosic fillers such as low-cost, easy-supply, abundance, recycling, renewability, low-abrasion to machine tools, biodegradability (Španić *et al.*, 2010;

Ayrilmis *et al.*, 2013b; Ayrilmis *et al.*, 2021). In general, lignocellulosic fillers have higher tensile modulus than many thermoplastics, thereby improving the stiffness of the thermoplastic composites (Shahzad, 2012). Particularly, lignocellulosic materials used as fillers are preferred in the commercial production of thermoplastic composites as they reduce the cost of the composite and provide the use of sustainable green materials. The addition of lignocellulosic fillers into the thermoplastics also decreases the amount of the plastics in the WPCs. Similary, lignocellulosic materials are alternative to some synthetic fiber, such as glass fibers, for some specific applications such as automotive industry. Furthermore, recently sustainable bio-based materials have been preferred for housing applications.

The wood of chaste tree, as an underutilized invasive species, is a promising sustainable raw material source for the WPCs. Its high cellulose content makes it an attractive lignocellulosic filler for thermoplastic composites. Unfortunately, the chaste wood has not been industrially used in the production of wood composites yet. Industrial use of chaste wood may result in a substantial economic impact on the WPC industry. The addition of chaste wood into thermopolypropylene may considerably decrease the cost of WPC due to relatively high cost of polypropylene. According to our extensive search, no study has yet been reported on the evaluation of chaste wood as filler in the production of WPC. The use of chaste wood as filler in the WPC manufacture could be one of the most efficient uses in high value-added composites. In this study, the impact of the use of chaste wood on the physical and mechanical properties of WPC were investigated.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

The stems of chaste trees with a diameter of 5-10 cm were obtained from Mugla city, Western Turkey (Figure 1). The stems were cut to the small chips by a laboratory chipper with three knives. The wood chips

Figure 1 *Vitex agnus-castus* plant (Chaste tree) samples
Slika 1. Uzorci biljke *Vitex agnus-castus* (konopljike)

were first air-dried in normal atmospheric temperature, and then dried to the 2-3 % moisture content in a dryer. The chips were ground into wood particles using a laboratory type grinder. The wood particles were screened for 10 min and the wood flour passing through the screen openings of 0.237 mm was used in the experiments. The wood flour were dried to less than 1 % moisture content at 95 °C for 3 h. The moisture content of the wood has an impact influence on the physical and mechanical properties of the WPC because the moisture makes irregularities in the pellets such as microvoids during the extrusion process. This problem can also be observed in the injection molded WPCs.

The virgin polypropylene granules were obtained from a local seller in Turkey. The melt flow index and density of polypropylene were 12 g/10 min (2.16 kg/230 °C) and 0.90 g/cm^3, respectively. Maleic anhydride modified homopolymer polypropylene (MAPP; Code: optim P-425, melt flow index with 110 g/10 min (2.16 kg/190 °C, and density: 0.91 g/cm^3)) granules, obtained from Pluss Polymers Pvt. Ltd. in Gurgaon city, India, were used as a compatibilizer to improve interfacial bond between polypropylene and wood.

2.2 Production WPC panels
2.2. Proizvodnja WPC ploča

The raw materials were weighed based on each formulation given in Table 2 and then pre-mixed. The mixture was put into volumetric feeder of the twin-screw extruder (co-rotating). The extruder barrel temperature was gradually increased from 170 °C (feeding

Table 2 Description of resulting WPC codes
Tablica 2. Opis oznaka dobivenih WPC uzoraka

WPC specimen code *Oznaka WPC-a*	**Wood flour content (wt%)** *Sadržaj drvnog brašna (wt. %)*	**Polypropylene content (wt%)** *Sadržaj polipropilena (wt. %)*	**MAPP content (compatibilizer) (wt%)** *Sadržaj MAPP-a (kompatibilizator) (wt. %)*
A	0	100	0
B	10	87	3
C	20	77	3
D	30	67	3
E	40	57	3
F	50	47	3

a) WPC mat / *tepih WPC-a*   b) Hot pressing of WPC mat *vruće prešanje tepiha WPC-a*   c) WPC samples *uzorak WPC-a*

Figure 2 Hot-press molding of WPC granules and resulting WPC specimens
Slika 2. Vruće prešanje WPC peleta i dobiveni WPC uzorci

zone) to the die zone (190 °C) at a constant screw speed (40 rpm). The compound filaments were put in water bath for cooling and then granulated using the granulation process. Before the injection molding process, the moisture content of the granules was decreased to about 1 % in an oven with fan.

First, the granules were placed in the metal frame and then transported to the hot-press (Figure 1a). Wax paper was used between the mat and the meal caul so that the mat did not stick to the metal cauls. The hot-press platens contacted the surface of the mat for melting the pellets at 190 °C for 10 min and then the compression was applied to the mat at 2 MPa and 190 °C for 5 min (Fig. 2b). The resulting WPC panels with dimensions of 200 mm × 200 mm × 4 mm were taken out from the hot press and then metal weights were immediately put on WPC panels for cooling.

2.3 Physical and mechanical characterization of WPCs
2.3. Fizička i mehanička karakterizacija WPC uzoraka

Water resistance of the WPC specimens, water absorption and thickness swelling after immersion in normal water for 24 h at room temperature were measured according to ISO 62 (2008) standard. The bending strength (MOR) and bending modulus (MOE) of the WPCs were carried out on the universal testing machine (Lyold instruments Ltd, West Sussex, UK) with a testing speed of 5 mm/min specified in ISO 178 (2010) standard. The tensile strength and modulus of the specimens were determined with a constant crosshead speed (2 mm/min) according to ISO 527 (2012) standard. The notched izod impact strength of the specimens was determined based on the impact bending equipment with a hammer of 2 J (Devotrans Company, Istanbul, Turkey) according to ISO 180 (2019) standard. The six specimens were used for determining the mechanical properties and four specimens for determining the physical properties. The specimens were conditioned according to ISO 291 (2008) standard prior to tests.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Physical properties of WPCs
3.1. Fizička svojstva WPC uzoraka

The density, thickness swelling, and water absorption of the WPC specimens are given in Table 3. The unfilled polypropylene showed negligible thickness swelling (0.08 %) and water absorption (0.05 %). The water resistance of the WPC specimens considerably decreased when the filler content increased in the polypropylene. Especially, when the filler content increased from 40 to 50 wt%, the water absorption of the WPCs sharply decreased, from 2.01 to 7.09 %. Similarly, when the filler content increased from 20 to 30 wt%, the thickness swelling increased from 0.90 to 3.74 %. Due to the hydrophilic property of wood, the water absorption and thickness swelling of the WPCs are negatively affected by the increased amount of the chaste wood flour. The WPCs having a higher amount of wood flour absorbed more water due to the increasing number of microcavities in the WPC as shown in the SEM image (Figure 3) and high amount of free hydroxyl groups. As known, holocelluloses in wood, cellulose, and hemicelluloses, contain free-hydroxyl groups reacting with the water molecules. The holocellulose content of wood flour (74.64 %) is significantly higher than that of hardwoods and softwoods (65-70 %) (Pettersen, 1984).

Polypropylene has a hydrophobic character, and its water absorption is quite negligible because it does not contain any functional polar group. There are several reasons for the lower water resistance of the WPCs such as the decrease in the amount of the polymer matrix as binder, the anotomy and chemical structure of wood, filler content, microcavities, microcracks, and poor interfacial bond in the WPC (Gardner *et al.*, 2015; Ayrilmis and Ashori, 2015; Özdemir *et al.*, 2017). Furthermore, the increase in the water absorption of the WPCs may be explained by the tortuous path formed. The tortuous diffusion paths enable the penetrating of

Table 3 Physical properties of WPCs
Tablica 3. Fizička svojstva WPC uzoraka

WPC specimen code, % *Oznaka WPC-a, %*	**Density, g/cm³** *Gustoća,* g/cm³	**Water absorption, %** *Apsorpcija vode, %*	**Thickness swelling, %** *Debljinsko bubrenje, %*
A	0.91 (0.02)*	0.05 (0.01)	0.08 (0.01)
B	0.92 (0.03)	0.25 (0.09)	0.58 (0.12)
C	0.84 (0.03)	1.08 (0.034)	0.90 (0.66)
D	0.92 (0.02)	1.20 (0.22)	3.74 (0.95)
E	0.98 (0.07)	2.01 (0.24)	4.78 (1.09)
F	1.02 (0.03)	7.09 (0.21)	5.68 (2.11)

*Standard deviation / *standardna devijacija*

water into the WPC. As a result, the increase in the filler content may results in the increased tortuousity in the WPC (Ayrilmis *et al*., 2013b). Typical SEM image of the tensile fracture surface of the WPC containing 50 wt% wood flour is presented in Figure 3. As shown in the SEM image, the number of cavities that take the water can be observed in the specimens with higher content of wood flour.

3.2 Mechanical properties of WPCs
3.2. Mehanička svojstva WPC uzoraka

The results of mechanical tests of the WPCs are summarized in Table 4. The MOR and MOE of the unfilled polypropylene specimens were determined as 30.2 MPa and 664 MPa, respectively. When the filler content increased from 10 to 30 wt%, the MOR increased from 33.9 to 44.8 MPa, and then decreased to 33 MPa. The lowest MOR (25.7 MPa) was found in the specimens with 50 wt% wood flour. The MOE of the WPCs was positively affected by the increased filler content. When the wood flour content increased from 10 to 50 wt%, the MOE increased from 815 to 3250 MPa. The increment in the MOE was not high when the addition of wood flour was increased from 40 to 50 wt%.

The tensile strength and modulus of unfilled polypropylene was determined as 19.6 MPa and 1505 MPa, respectively. The tensile strength of the WPCs was considerably lower than that of the unfilled polypropylene. The tensile modulus of the WPCs showed an increasing trend (11.7 MPa to 14.1 MPa) as the amount of the filler increased from 10 to 40 wt. Nevertheless, a further increase in the filler content reduced the tensile strength (11.4 MPa). When compared to the unfilled polypropylene, the lower tensile strength of the WPCs with high filler content may primarily be explained by the decrease in the polypropylene content, which acted as a binder for the filler in the composite. Moreover, higher loading levels of the wood filler, such as 50 wt%, caused a high degree agglomeration of wood particles in the polypropylene, which formed the zones of stress concentration and negatively affected the tensile strength. The tensile strength results showed that interfacial bonding was adversely influenced by the increased wood content when the wood content was beyond the optimum value of 40 wt%. This ratio was found to be 30 wt% wood flour for the bending strength (Table 4). As shown in the SEM image in Figure 3, most microvoids were observed in the specimens with the highest filler content (50 wt%), which decreased the tensile strength and bending strength. The pulled-out wood fibers and the resulting microcavities can be seen in the SEM image marked with yellow circles (Figure 3). Although the coupling agent (MAPP) was used to improve interfacial bond (ester bond) between the wood and polypropylene, the reason for fiber pullout at high filler content (50 wt%) may be explained by the agglomeration of wood particles and increasing microcavities.

Table 4 Mechanical properties of WPCs
Tablica 4. Mehanička svojstva WPC uzoraka

WPC specimen code *Oznaka WPC-a*	**Bending strength, MPa** *Čvrstoća na savijanje,* MPa	**Bending modulus, MPa** *Modul savijanja,* MPa	**Tensile strength, MPa** *Vlačna čvrstoća,* MPa	**Tensile modulus, MPa** *Modul elastičnosti pri vlačnom naprezanju,* MPa	**Notched izod impact strength, kJ/m²** *Otpornost na udarce,* kJ/m²
A	30.2 (1.6)*	664 (58)	19.6 (1.15)	1505 (187)	5.88 (0.45)
B	33.9 (4.1)	815 (89)	11.7 (1.64)	1690 (296)	4.93 (1.0)
C	35.0 (2.9)	1197 (105)	10.4 (2.16)	1838 (267)	4.83 (0.33)
D	44.8 (3.1)	1538 (112)	12.2 (1.25)	2034 (223)	4.44 (0.64)
E	33.0 (3.8)	3041 (132)	14.1 (1.20)	2241 (131)	3.92 (0.42)
F	25.7 (1.2)	3250 (119)	11.4 (0.92)	2253 (233)	3.52 (5.85)

*Standard deviation / *standardna devijacija*

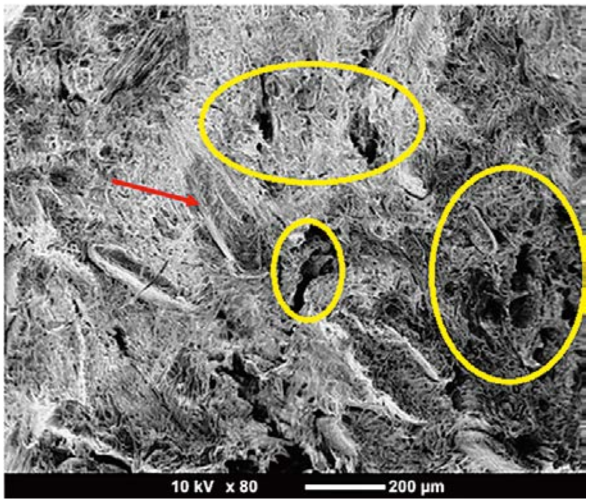

Figure 3 SEM image of tensile fracture surface of WPC containing 50 wt% chaste wood flour (red arrow shows wood fiber failure; yellow circles show pulled-out wood fibers and microcavities)
Slika 3. SEM slika površine vlačnog loma WPC-a koji sadržava 50 wt. % drvnog brašna konopljike (crvena strelica pokazuje lom drvnih vlakana, a žuti krugovi označuju izvučena drvna vlakna i mikrošupljine)

The results of bending modulus clearly showed that the wood flour efficiently contributed to the stress transfer from polypropylene to wood particles. The modulus of wood is higher than that of polypropylene (Shahzad, 2012). The addition of wood flour into the polymer matrix makes it stiffer, which results in the reduced failure strain (Dányádi *et al.*, 2006). In previous studies, this behavior was also described by the decrease in the mobility of the polymer chains when the lignocellulosic filler was added to the polymer matrix (Hinestroza and Netravali, 2014; Dolza *et al.*, 2021; Aljnaid and Banat, 2021).

The results of the notched izot impact strength of the WPCs are given in Table 4. As expected, the impact strength of the specimens decreased with increasing filler content. The impact bending strength of the unfilled polypropylene was 5.88 kJ/m^2. As the wood flour content increased from 10 to 50 wt%, the impact strength decreased from 4.93 to 3.52 kJ/m^2. The impact strength of the WPC specimens decreased gradually as the filler content was increased (Table 4). This can be explained by the decrease in the energy absorption of polypropylene with the addition of wood flour. The impact strength of thermoplastics is higher than that of lignocellulosic fillers because thermoplastics absorb more energy than rigid materials such as wood due to their elastic property (Ayrilmis, 2013a). Thus, the polypropylene filled with wood flour showed brittle fracture behavior. Furthermore, the microcavities and weak interfacial bond between polymer matrix and filler at higher filler contents form the zones of stress-concentration that need lower energy to start cracks in the WPC (Yadav *et al.*, 2021).

4 CONCLUSIONS
4. ZAKLJUČAK

The physical and mechanical properties of the WPCs were influenced by the loading level of the chaste wood flour. Although the water resistance of the specimens was negatively affected by the increased filler content, particularly above 20 wt%, the tensile and bending modulus values improved. When compared to the unfilled polypropylene, the bending modulus greatly improved with the increased filler content. Although the tensile strength of the unfilled polypropylene was higher than that of the WPCs, an increasing trend was observed in the tensile strength values up to 40 wt% filler content; however further increase in the filler content negatively affected the tensile strength. The evaluation of the wood of underutilized invasive chaste tree in the WPC production may result in an economically effective way to use this resource. The low specific flexural modulus of polypropylene limits its use in semi-building applications. The incorporation of chaste wood into the polypropylene could make of it a sustainable natural filler for semi-building applications such as decking, fencing, siding. According to the results, it was concluded that the 30-40 wt% incorporation of the chaste wood flour can be used in the production of the WPC.

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HRVATSKA KOMORA INŽENJERA ŠUMARSTVA I DRVNE TEHNOLOGIJE

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Drvo obične američke duglazije *Pseudotsuga menziesii* (Mirb.) Franco

MIKROSKOPSKA OBILJEŽJA

Drvo duglazije karakterizira oštar prijelaz ranog drva u kasno, s vidljivim smolenicama. Epitel uzdužnih i radijalnih smolenica je debelostijeni. Volumni udio aksijalnih traheida iznosi oko 93 %. Raspored aksijalnih traheida na poprečnom je presjeku pravilan i radijalan. Traheide ranog drva su tankostijene (2 – 4 µm), promjera 37 do 54 µm. Traheide kasnog drva su debelostijene (4 – 8 µm) i tangentno splоštene, promjera od 24 do 33 µm. Traheide imaju gusta spiralna zadebljanja. Duljina traheida kreće se od 2,5 do 5,6 mm. Aksijalni je parenhim terminalan, oskudan ili nedostaje te ima beznačajan volumni udio. Drvni su traci nepravilno raspoređeni, heterocelularni, s traheidama trakova koje imaju spiralna zadebljanja na stijenkama. Visina drvnih trakova je do 16 stanica, a širina do 5 stanica (onih sa smolenicama). Volumni udio drvnih trakova je oko 7 %.

RELEVANTNE SPOZNAJE O VARIJACIJAMA DRVA DUGLAZIJE UNUTAR GODA

Gustoću drva duglazije karakterizira znatna neujednačenost unutar i između godova (Vonnet i dr., 1985.). Wimmer je (1995.) osmislio geometrijski model za istraživanje utjecaja svojstava stanica drva na gustoću godova. Zaključio je da u drvu četinjača radijalni promjer stanica i debljina stanične stijenke traheida kasnog drva uvelike utječu na gustoću godova.

Razlikovanje ranoga i kasnog drva četinjača općenito se temelji na odnosu stijenka – lumen stanica (Park i dr., 2006.). U anatomiji drva dobro je poznata Morkova definicija (Mork, 1928.). Različiti autori objasnili su to na sljedeći način (Denne, 1988.; Schweingruber, 1983.; Park i dr., 2006.): a) Mork je predložio podjelu na rano i kasno drvo prema određenom položaju unutar goda, pri čemu je vrijednost indeksa (= 2 × dvostruka debljina stanične stijenke/staničnog lumena) veća od 1; b) takvo načelo primijenjeno je za određivanje gustoće drva, svojstva koje ovisi o omjeru stijenke i lumena stanica; c) u idealnom slučaju god počinje stanicama ranog drva i kada vrijednost indeksa (gustoće) prijeđe zadanu vri-

MICROSCOPIC CHARACTERISTICS

Douglas-fir wood is characterized by abrupt transition from earlywood to latewood, and resin canals normally occur and are visible. The epithelial cells of longitudinal and radial resin canals are thick-walled. The volume fraction of axial tracheids is about 93 %. The arrangement of axial tracheids on the cross-section is regular radial. Earlywood tracheids are thin-walled (2 to 4 µm), 37 to 54 µm in diameter. Latewood tracheids are thick-walled (4 to 8 µm) and tangentially flattened with a diameter of 24 to 33 µm. Tracheids are characterized by dense spiral thickenings. Tracheid length ranges from 2.5 to 5.6 mm. Axial parenchyma is terminal, scarce or absent and has an insignificant volume fraction. Wood rays are irregularly distributed, heterocellular with ray tracheids that have spiral thickenings on cell walls. The height of wood rays is up to 16 cells, and the width is up to 5 cells (those with resin canals). The volume fraction of wood rays is about 7 %.

RELEVANT INFORMATION ABOUT DOUGLAS-FIR INTRA-TREE-RING VARIATIONS

The wood density of Douglas-fir is characterized by a strong inter- and intra-tree-ring heterogeneity (Vonnet et al., 1985). Wimmer (1995) designed a geometric model to investigate the influence of wood cell characteristics on tree-ring density. He concluded that, for conifers, the radial cell diameter and the cell wall thickness of latewood tracheids greatly influenced the tree-ring density.

Earlywood and latewood demarcation for conifers is generally based on the wall-lumen relation of cells (Park et al., 2006). Mork's definition is well known in wood anatomy (Mork, 1928). The following was explained by different authors (Denne, 1988; Schweingruber, 1983; Park et al., 2006): (a) Mork suggested the divison of the earlywood and latewood into a fixed intra-ring position, where the index value (= 2 × double cell-wall thickness/cell lumen) exceeds 1; (b) such principle has been used in determining wood density, a characteristics related to the wall/lumen relation of cells; (c) in an ideal case, a tree ring begins with jednost, sve proizvedene stanice ksilema smatraju se stanicama kasnog drva.

Pregled istraživanja varijabilnosti unutar godova drva duglazije donosi spoznaje: (a) o najvećoj gustoći drva koja je uglavnom utvrđena prema kraju goda, a prijelaz iz ranoga u kasno drvo varirao je od postupnoga do nagloga (de Kort i dr., 1991.); (b) o sklonosti brzom rastu koji se povezuje s formiranjem traheida tankih stijenki u ranom drvu, ali i traheida debelih stijenki u kasnom drvu, kao i o blagom trendu koji pokazuje da stabla s većom gustoćom ranog drva imaju nešto manju gustoću kasnog drva (Abdel-Gadir i dr., 1993.); (c) o omjeru stijenke i lumena stanica koji se eksponencijalno povećao prema kraju redova stanica (Park i dr., 2006.); (d) o unutargodišnjoj staničnoj morfogenezi potaknutoj klimatskim uvjetima tijekom zimskog razdoblja, što upućuje na mogućnost razmatranja staničnih strukturnih obilježja u rekonstrukciji klimatskih varijabiliteta prethodnih godina (Balanzategui i dr., 2021.).

Detaljno istraživanje o povezivanju gustoće drva unutar goda i anatomskih obilježja traheida u drvu duglazije koje su proveli Rathgeber i dr. (2006.) govori: a) o smanjenju promjera stanica prijelazom iz ranog drva u kasno (radijalni promjeri stanica smanjili su se na polovicu svoje veličine, dok su tangentni promjeri stanica izgubili samo 15 % svojih dimenzija); b) o većem smanjenju promjera lumena stanica u usporedbi s promjerom stanica kroz god u radijalnom smjeru; c) o blagom smanjenju debljine stanične stijenke kroz god u radijalnom smjeru; d) o vrlo dobroj korelaciji između gustoće drva i udjela staničnih stijenki.

earlywood cells and once the index (density) value exceeds the given threshold, all the xylem cells produced are latewood cells.

A review of research on the intra-tree-ring variability in Douglas-fir provides the following information: (a) maximum density was frequently determined near the ring boundary, and transition from the earlywood to latewood varied from gradual to abrupt (de Kort et al. 1991); (b) the tendency for fast growth to be associated with thin-walled tracheids in the earlywood, but with thick-walled tracheids in the latewood, as well as a slight trend indicating that trees with higher earlywood density have slightly lower latewood density (Abdel-Gadir et al., 1993); (c) the cell wall/lumen ratio increased exponentially towards the end of the cell lines (Park et al., 2006); (d) intra-annual cell morphogenesis was driven by the climatic conditions during the winter period, demonstrating the potential of cell structural characteristics for reconstruction of the past climatic variability (Balanzategui et al., 2021).

A detailed investigation conducted by Rathgeber et al. (2006) on linking the wood intra-tree-ring density and tracheid anatomical characteristics in Douglas-fir concluded as follows: (a) a decline in cell diameters from the earlywood to latewood (radial cell diameters went down to half their size, while tangential cell diametres lost only 15% of their size); (b) a greater lumen diameter reduction, in the radial direction throughout the annual growth ring, in comparison to the cell diameter; (c) a slight decrease in the cell-wall thickness in the radial direction throughout the annual growth ring; (d) a very good correlation between the wood density and cell-wall proportion.

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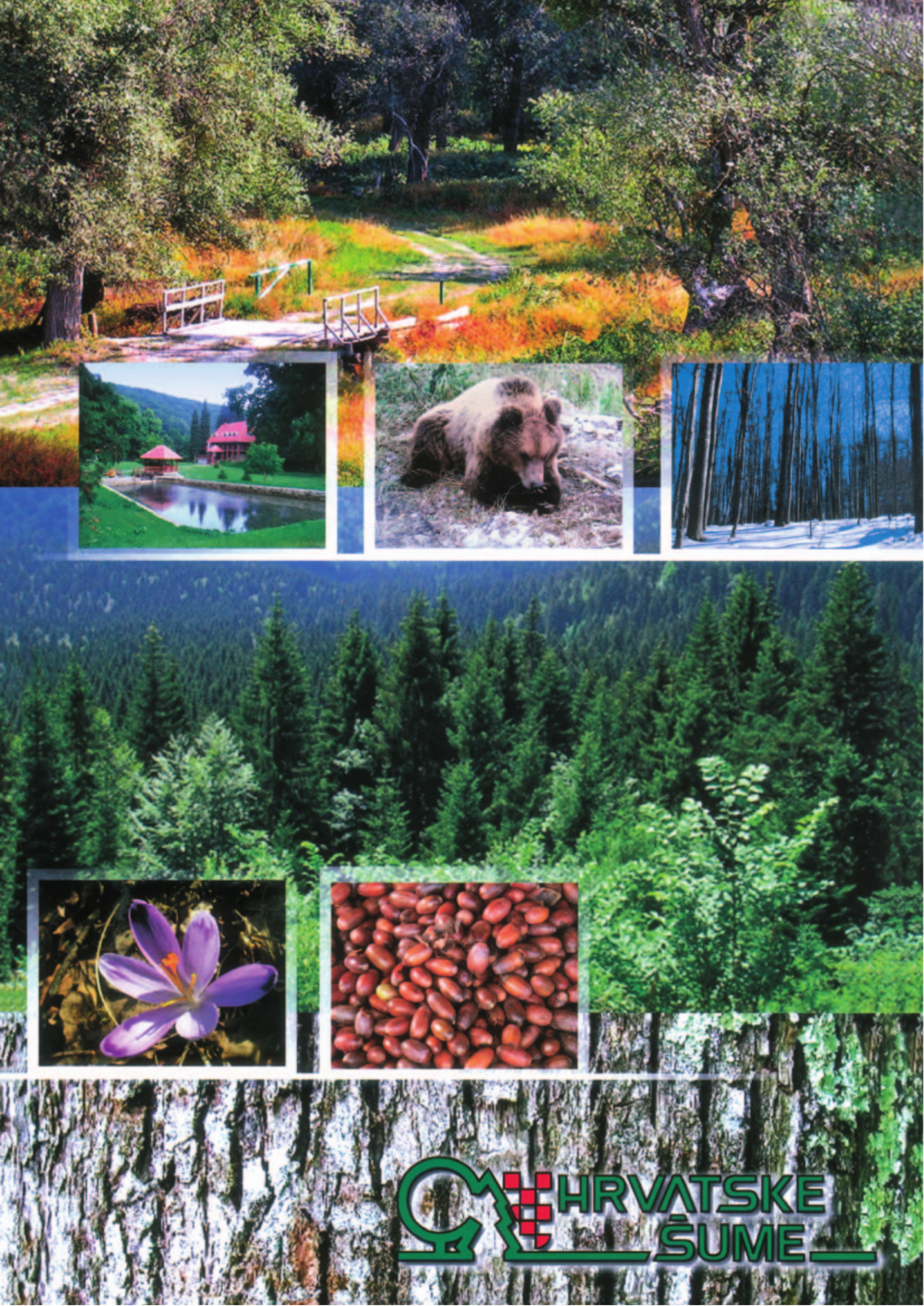
HRVATSKE
ŠUME

Projekt *Šume u rukama žena* - Fem4Forest

Projekt "Šume u rukama žena" („*Fem4Forest*") želi ojačati sektor baziran na šumama na lokalnoj, regionalnoj i međuregionalnoj razini kroz povećanu uključenost i jačanje sposobnosti žena, podržavajući njihovu jednaku prisutnost i kompetencije na tržištu.

Projekt se provodi od 01.07.2020 - 21.12.2022. godine u sklopu *Interreg Danube* transnacionalnog programa te je sufinanciran iz fondova EU (ERDF, IPA, ENI) i Vlade RH (Ured za udruge).

Na projektu sudjeluje 14 partnera iz 10 država Dunavske regije. Hrvatski partneri su Hrvatska komora inženjera šumarstva i drvne tehnologije i Hrvatski savez udruga privatnih šumovlasnika.

Na temelju provedenih upitnika i intervjua, zaključeno je kako su osobni razvoj i cjeloživotno učenje put za uspješnu karijeru svake žene u svim sektorima, pa tako i šumarskom, a na tom putu potrebno je:

- unaprijediti organizaciju poslovanja,
- učiti o tehnikama upravljanja kolektivom,
- vježbati komunikacijske vještine te
- identificirati uzore u sektoru i njihova iskustva.

Provedeni su razgovori sa ženama uzorima u sektoru, čija dugogodišnja i raznolika karijera, bogato iskustvo te pozitivan stav prema radu mogu poslužiti kao primjer mladim ženama na njihovu putu i profesionalnom razvoju.

U sklopu projekta uskoro počinje provedba trening radionica koje će se fokusirati na vještine vođenja, prezentacijske i komunikacijske vještine te osobne vještine za podizanje motivacije i samopouzdanja.

Za više informacija pratite web stranice projektnih partnera (www.hkisdt.hr, www.hsups.hr).


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