

SCIENTIFIC JOURNAL OF WOOD TECHNOLOGY



ZNANSTVENI ČASOPIS ZA PITANJA DRVNE TEHNOLOGIJE

UDK 674.031.677.7 ISO: Drv. Ind. CODEN: DRINAT JCR: DRVNA IND ISSN 0012-6772 Populus spp.





SCIENTIFIC JOURNAL OF WOOD TECHNOLOGY

Znanstveni časopis za pitanja drvne tehnologije

PUBLISHER AND EDITORIAL OFFICE

Izdavač i uredništvo

Faculty of Forestry and Wood Technology University of Zagreb, Croatia Fakultet šumarstva i drvne tehnologije Sveučilišta u Zagrebu www.sumfak.unizg.hr

CO-PUBLISHER / Suizdavač

Hrvatska komora inženjera šumarstva i drvne tehnologije

FOUNDER / Osnivač Institut za drvnoindustrijska istraživanja, Zagreb

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Časopis Drvna industrija javno je znanstveno glasilo za objavu rezultata istraživanja građe, svojstava i zaštite drva i drvnih materijala, primjene drva i drvnih materijala, mehaničke i hidrotermičke obrade te kemijske prerade drva, svih aspekata proizvodnje drvnih materijala i proizvoda te trgovine drvom i drvnim proizvodima. Časopis izlazi četiri puta u godini uz financijsku potporu Ministarstva znanosti i obrazovanja Republike Hrvatske.

Contents Sadržaj

CIRCULATION: 400 pieces

INDEXED IN: Science Citation Index Expanded, Scopus, CAB Abstracts, Compendex, Environment Index, Veterinary Science Database, Geobase, DOAJ, Hrčak

MANUSCRIPTS ARE TO BE SUBMITTED by the link http://journal.sdewes.org/drvind

CONTACT WITH THE EDITORIAL e-mail: editordi@sumfak.hr

SUBSCRIPTION: Annual subscription is 55 EUR. For pupils, students and retired persons the subscription is 15 EUR. Subscription shall be paid to the IBAN HR0923600001101340148 with the indication "Drvna industrija".

PRINTED BY: DENONA d.o.o., Getaldićeva 1, Zagreb, www.denona.hr

DESIGN: Bernardić Studio

THE JOURNAL IS AVAILABLE ONLINE: https://drvnaindustrija.com

COVER: Cross section of Populus spp., xylotheca of Institute for Wood Science, Faculty of Forestry and Wood Technology University of Zagreb

DRVNA INDUSTRIJA · VOL. 74, 1 · P. 1-136 · SPRING 2023. · ZAGREB EDITORIAL COMPLETED 1. 3. 2023.

NAKLADA: 400 komada

ČASOPIS JE REFERIRAN U: Science Citation Index Expanded, Scopus, CAB Abstracts, Compendex, Environment Index, Veterinary Science Database, Geobase, DOAJ, Hrčak

ČLANKE TREBA SLATI putem poveznice http://journal.sdewes.org/drvind

KONTAKT S UREDNIŠTVOM: e-mail: editordi@sumfak.hr

PRETPLATA: Godišnja pretplata za pretplatnike u Hrvatskoj i inozemstvu iznosi 55 EUR. Za đake, studente i umirovljenike 15 EUR. Pretplata se plaća na IBAN HR0923600001101340148 s naznakom "Drvna industrija".

TISAK: DENONA d.o.o., Getaldićeva 1, Zagreb, www.denona.hr

DESIGN: Bernardić Studio

ČASOPIS JE DOSTUPAN NA INTERNETU: https://drvnaindustrija.com

NASLOVNICA: Presjek drva Populus spp., ksiloteka Zavoda za znanost o drvu, Fakultet šumarstva i drvne tehnologije Sveučilišta u Zagrebu

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Seid Hajdarevic¹, Murco Obucina¹, Manja Kitek Kuzman², Dick Sandberg³

Bending Moment of Mortise-and-Tenon Joints in a Crossed Chair Base

Moment savijanja spojeva s čepom i rupom na križnoj potkonstrukciji sjedala stolice

ORIGINAL SCIENTIFIC PAPER

Izvorni znanstveni rad Received – prispjelo: 21. 12. 2021. Accepted – prihvaćeno: 14. 5. 2022. UDK: 684.4.058; 684.412 https://doi.org/10.5552/drvind.2023.0004

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ABSTRACT • This paper investigated the bending moment of chair base joints. The ultimate bending moments (maximum moment), calculated on the base of the measured maximum applied loads (maximum force), were compared for the front leg and rear leg joints of a chair base. The joints had different angles between the stretcher and the leg (joint angle) as well different tenon lengths (30 mm and 32 mm). The results of the tests indicated that for different test specimen configurations but the same tenon-and-mortise geometry, the maximum force of joints with a smaller value of joint angle (front leg joints) was higher than the force values of joints with a larger angle (rear leg joints) for all tenon lengths. However, the results showed less difference among the calculated bending moments of the analysed sets of joint angle and the bending moments of joints with a smaller value of joint angle and the bending moments of joints with a larger angle for all tenon lengths. A significant difference was not revealed between the bending moments of joints with a smaller value of joint angle and the bending moments for the tenon length of 30 mm and tenon length of 32 mm was determined for rear leg joints but not for front leg joints. The presented approach of joint strength analysis through the testing of specimens with different shapes and dimensions are applicable to research and practice.

KEYWORDS: chair; mortise-and-tenon joint; bending moment; joint angle; tenon length

SAŽETAK • U radu je proučavan moment savijanja spojeva na križnoj potkonstrukciji sjedala stolice. Krajnji momenti savijanja (najveći moment), izračunani na temelju izmjerenih najvećih opterećenja (najveća sila), uspoređeni su za spojeve prednjih i stražnjih nogu križne potkonstrukcije sjedala stolice. Spojevi su imali različite kutove između poveznika i noge (kut spoja), kao i različitu duljinu čepova (30 i 32 mm). Rezultati ispitivanja pokazali su da je za različitu konfiguraciju ispitnih uzoraka, ali za istu geometriju čepa i rupe, najveća sila bila veća za spojeve s manjim kutom (spojevi prednjih nogu) nego za spojeve s većim kutom (spojevi stražnjih nogu), i to za čepove obiju duljina. Rezultati su također pokazali manju razliku između izračunanih momenata savijanja analiziranih grupa spojeva. Nije utvrđena značajna razlika momenata savijanja spojeva s manjim i većim kutom za čepove obiju duljina. Značajna razlika između momenata savijanja za čepove dužine 30 i 32 mm utvrđena je za spojeve stražnjih nogu, ali ne i za spojeve prednjih nogu stolice. Prikazani pristup analize čvrstoće spojeva ispitivanjem uzoraka različitih oblika i dimenzija primjenjiv je u istraživanju i u praksi.

KLJUČNE RIJEČI: stolica; spoj s čepom i rupom; moment savijanja; kut spoja; duljina čepa

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1 INTRODUCTION

1. UVOD

Different forms of mortise-and-tenon joints are still the most common solutions for connecting elements of wood chairs. The mechanical properties of tenon joints are widely researched in order to improve the quality of joints and chair structure, and hence different factors that affect the strength, stiffness and load-carrying capacity have been investigated.

The tenon size is well known to be the main factor affecting the moment capacity of a mortise-and-tenon joint. The bending-moment capacity is strongly affected by the length and width of the tenon, while the width has a slight lower influence (Kasal et al., 2015; Likos et al., 2012; Derikvand et al., 2014; Hajdarevic et al., 2020). Elek et al. (2020) investigated the effects of fitting for pairing of open full-width mortise-and-tenon-joint elements on the strength of joints, and this type of joint was found to be the strongest at a tight fit of 0.1 mm. The mortise-and-tenon joint, in combination with PVAc, provided the best strength for all investigated wood species (Vassiliou et al., 2016; Bardak et al., 2017), while PU glue has appropriate strength in comparison with PVAc, and can completely fill and cover the gaps between the elements of a joint (Hrovatin et al., 2013). The suitable construction type of chairs, constructed with defined tenon dimensions, has been determined based on the required load-carrying capacity (Kiliç et al., 2018; Hitka et al., 2018; Kasal et al., 2016; Ayrilmis et al., 2020). Different chair structures and chair joints were analysed with the finite-element method to obtain the stress and strain states under loading, and the results showed good agreement with experimental tests to determine the mechanical properties of such constructions and joints (Smardzewski, 2008; Horman et al., 2010; Hajdarevic and Martinovic, 2014; Hajdarevic and Busuladzic, 2015; Kasal et al., 2016).

The strength of the joints represents the strength of the entire system of a chair frame and, as the most critical parts of a chair, these should have adequate strength (Kiliç *et al.*, 2018). However, there is a problem in joint strength testing of real chair structures. The analysis of such joints should enable obtaining realistic and comparable values of joint strength regardless of the conditioned shape and dimensions of the tested sample, although the shape and dimensions of the tested samples depend on the shape and dimensions of the structure from which they were cut.

The aim of the study was, therefore, to explore the capabilities of the test method to obtain a comparable joint strength of two different sets of specimens that were cut from specific wooden frame configurations. The objective of the present study was to determine the effects of joint angle and length of the tenon in the joints of the crossed chair base in bending.

2 MATERIALS AND METHODS 2. MATERIJALI I METODE

This study investigated the bending moment of the crossed chair-base joints taken from an industrial manufacturing process. The front and rear leg joints of the crossed chair base had different angles, i.e. the positions of the stretcher and the front leg and the stretcher and the rear leg were different and asymmetric. In addition, all joints had the same geometry configuration with two different tenon lengths (30 mm and 32 mm).

Each crossed chair base was assembled from two frames and four corner blocks and supplied from furniture manufacturers (Figure 1a), and specimens for the bending test of the mortise-and-tenon joint were prepared using these chair bases by cutting the frames into two parts and shortening the leg length (Figure 1b-c). All parts of the crossed chair base were made of com-

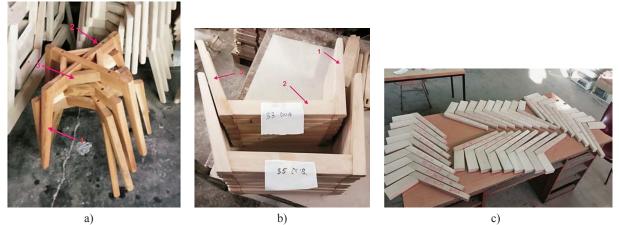
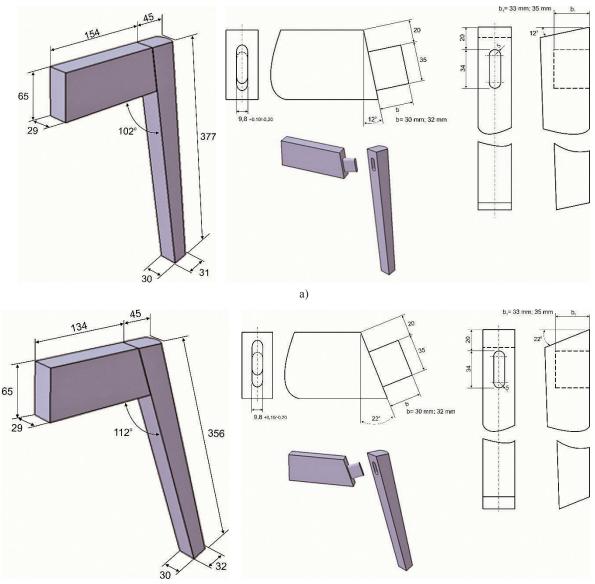


Figure 1 Crossed chair base: a) assembled structure (1 – leg; 2 – stretcher; 3 – corner block), b) frame of two chair legs (1 – front leg; 3 – rear leg) and stretcher (2), c) specimens prepared for bending test of mortise-and-tenon joint **Slika 1.** Križna potkonstrukcija sjedala stolice: a) sastavljena konstrukcija (1 – noga; 2 – poveznik; 3 – kutni poveznik); b) okvir dviju nogu stolice (1 – prednja noga; 3 – stražnja noga) i poveznik (2); c) uzorci spojeni čepom i rupom pripremljeni za ispitivanje



b)

Figure 2 Configuration of test specimens: a) front leg joint (left) and configuration of mortise-and-tenon (right); b) rear leg joint (left) and configuration of mortise-and-tenon (right)

Slika 2. Konfiguracija ispitnih uzoraka: a) spoj prednje noge (lijevo) i konfiguracija čepa i rupe (desno); b) spoj stražnje noge (lijevo) i konfiguracija čepa i rupe (desno)

mon oak wood (*Quercus robur* L.). The moisture content and density of oak wood were evaluated in accordance with the procedures described in ISO 13061-1 (2014) and ISO 13061-2 (2014) after testing. The average value of moisture content and density of the wood specimens were 12.1 % and 0.77 g/cm³, respectively.

Each frame was composed of two chair legs and a stretcher. PVA-c adhesive (Kleiberit 303.0, Klebchemie, Weingarten, Germany) was used to bond the frame elements together at ambient conditions and with applied pressure. The adhesive met the requirements of stress group D3 according to DIN/EN 204,

Specimen group No. Broj skupine uzoraka	Specimen group mark Oznaka skupine uzoraka	Position Pozicija	Angle between stretcher and leg, ° Kut između poveznika i noge, °	No. of specimens Broj uzoraka	Tenon length, mm <i>Duljina čepa,</i> mm
1	FL-T30	Front leg / prednja noga	102	9	30
2	FL-T32	Front leg / prednja noga	102	9	32
3	RL-T30	Rear leg / stražnja noga	112	9	30
4	RL-T32	Rear leg / stražnja noga	112	9	32

Table 1 Test set-up and description of specimens (see Figure 2)Tablica 1. Postavke ispitivanja i opis uzoraka (v. sl. 2.)

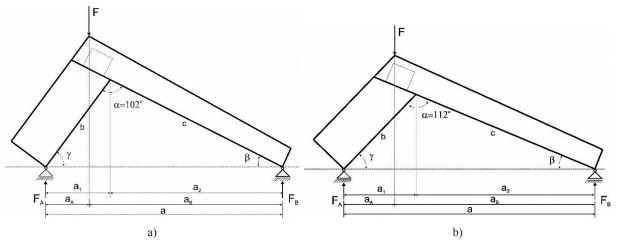


Figure 3 Diagram of joint loading: a) front leg joint, b) rear leg joint. Notation used: F - force (load), F_A and $F_B - \text{support}$ reactions, a_A and $a_B - \text{moment arms}$, i.e. perpendicular distances from support reaction line to force line, sides (a, b and c), angles (α , β and γ) and base distances to height (a_1 and a_2) of a scalene triangle

Slika 3. Dijagram opterećenja spoja: a) spoj prednje noge; b) spoj stražnje noge. Oznake: F - sila (opterećenje), F_A i F_B – reakcije oslonca, a_A i a_B – krakovi momenta, tj. okomiti razmaci od osi reakcije oslonca do osi sila, stranice (a, b i c), kutovi (α, β i γ) i udaljenosti baza do visine (a, i a₂) razmjernog trokuta.

with the density of 1.1 g/cm³, pH approx. 3 and viscosity at 20 °C of about 13.000 mPa·s. The stretcher joined and supported a front leg and a rear leg of the chair by mortise-and-tenon joints. All joints were of mortiseand-tenon type with rounded tenons.

Configurations of specimens of front and rear leg joints for the bending test of the mortise-and-tenon joint are shown in Figure 2. The geometry and dimensions of the specimens were different, i.e. the front leg joint had a smaller angle between the stretcher and leg (102°) than the test specimens of the rear leg joint (112°) . The test set-up is presented in Table 1.

The loading diagrams of the front leg joints and rear leg joints are shown in Figure 3. The supports were set up on the edges of the leg and stretcher cross-section. The load was applied to the joint in a manner that corresponded to the tension of the specimen. The test was carried out on a universal testing machine Zwick 1282 (Zwick Roell Group, Ulm, Germany), as shown in Figure 4. The force (load cell RSCC-C3/1t HBM, Darmstadt, Germany) and displacement (inductive displacement transducer WI10 HBM, Darmstadt, Germany) along the force line were measured simultaneously until a joint failure and large fall in the load occurred.

The applied load values and the corresponding displacement along the force line were ascertained using the data collected by the software. Working diagrams of the 36 tested specimens were then created.

The ultimate (maximum) bending moment of the joints, based on the ultimate applied load values, were calculated both for front leg joints and rear leg joints by:

$$M_{\rm max} = F_{\rm (A max)} \cdot a_{\rm A} = F_{\rm (B max)} \cdot a_{\rm B} \tag{1}$$

Where M_{max} is maximum bending moment, $F_{(A \text{ max})}$ and $F_{(B \text{ max})}$ are support reactions and, a_A and a_B

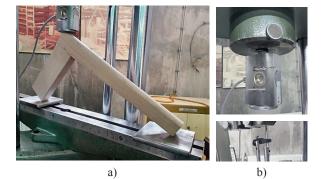


Figure 4 Joint testing: a) set-up of joints in testing machine, b) load cell and inductive displacement transducer Slika 4. Ispitivanje spoja: a) postavljanje spojeva u ispitni uređaj; b) davač sile i induktivni davač pomaka

are moment arms. Support reactions were obtained based on the trigonometric analysis and static conditions.

3 RESULTS AND DISCUSSION

3. REZULTATI I RASPRAVA

Figure 5 shows the force-displacement diagrams of the tested specimens. All joints had a wide defined elastic region, *i.e.* proportional zones. The initial wood or bond-line fractures occurred at load values higher than 4000 N for all specimens. There was a noticeable difference among the ultimate loads of the front and rear leg joints, although the differences were not apparent among the ultimate loads of the front or rear leg joints with different tenon lengths. The values of displacements at ultimate load ranged from 8 to 12 mm.

The results of maximum force (F_{max}) and maximum moment (M_{max}) of the front leg joints and rear

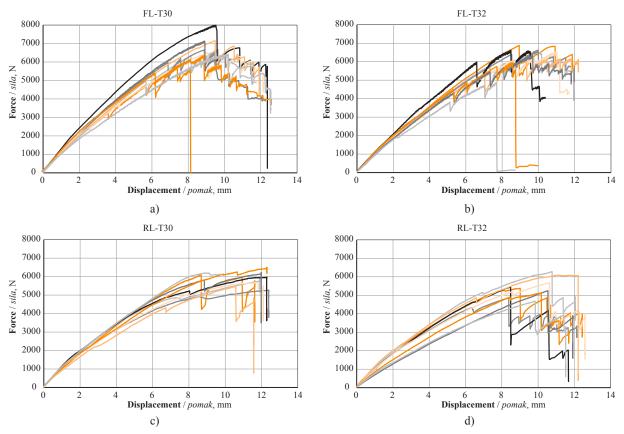


Figure 5 Force-displacement diagram of joint strength testing for front leg joints with tenon length of a) 30 mm and b) 32 mm, and rear leg joints with tenon length of c) 30 mm and d) 32 mm

Slika 5. Dijagram sila-pomak pri ispitivanju čvrstoće spojeva prednje noge s čepom duljine: a) 30 mm i b) 32 mm, te stražnje noge s čepom duljine: c) 30 mm i d) 32 mm

joints with two different tenon lengths are given in Tables 2 and 3, respectively.

The characteristic pattern of the ultimate fractures of joints is shown in Figure 6. The tenon started to take the load after the bond-line failed, and then the tenon was partially pulled out from the mortise, while fracture of mortise wood occurred. This typical fracture mode of L-shape mortise and tenon joints glued with PVA-c glue was also described in (Kasal *et al.*, 2015).

Front leg – joint angle 102° / Prednja noga – kut spoja 102°					
No. of specimenTenon length 30 mm / Duljina čepa 30 mm			Tenon length 32	mm / <i>Duljina čepa 32</i> mm	
Broj uzorka	Max force, N	Max moment, Nm	Max force, N	Max moment, Nm	
	<i>Najveća sila,</i> N	<i>Moment savijanja,</i> Nm	<i>Najveća sila,</i> N	<i>Moment savijanja,</i> Nm	
1	6421.1	359.0	4891.8	273.5	
2	6329.1	353.8	5940.1	332.1	
3	7136.9	399.0	6336.9	354.3	
4	7155.9	400.1	6165.2	344.7	
5 6511.0		364.0	6514.8	364.2	
6	7986.0	446.5	6622.2	370.2	
7	6647.7	371.7	6631.4	370.7	
8	6670.1	372.9	6496.1	363.2	
9	5736.1	320.7	6881.9	384.7	
Mean / srednja vrijednost	6732.7	376.4	6275.6	350.8	
Median / medijan	6647.7	371.6	6496.1	363.2	
Standard deviation	635.1	35.5	587.7	32.9	
standardna devijacija					
Coefficient of variation	9.4 %	9.4 %	9.4 %	9.4 %	
koeficijent varijacije					

Table 2 Results of forces and bending moments of front leg joints

 Tablica 2. Rezultati sila i momenata savijanja spojeva prednjih nogu

Rear leg – joint angle 112° / Stražnja noga – kut spoja 112°					
No. of specimen			Tenon length 32 mm		
Broj uzorka	Duljine	<i>a čepa 30</i> mm	Duljine	<i>a čepa 32</i> mm	
	Max force, N	Max moment, Nm	Max force, N	Max moment, Nm	
	<i>Najveća sila,</i> N	Moment savijanja, Nm	<i>Najveća sila,</i> N	Moment savijanja, Nm	
1	6194.4	386.0	4682.0	291.8	
2	6503.6	405.3	5131.5	319.8	
3	6222.6	387.8	5248.6	327.1	
4	5452.7	339.8	6088.4	379.4	
5	5670.8	5670.8 353.4		353.4	
6	5969.5	372.0	5451.6	339.7	
7	5261.7	327.9	4863.4	303.1	
8	5931.4	369.6	6282.1	391.5	
9	6080.2	378.9	5372.8	334.8	
Mean / srednja vrijednost	5920.8	369.0	5421.3	337.9	
Median / medijan	5969.5	372.0	5372.8	334.8	
Standard deviation	395.2	24.6	527.2	32.9	
standardna devijacija					
Coefficient of variation	6.7 %	6.7 %	9.7 %	9.7 %	
koeficijent varijacije					

Table 3 Results of forces and bending moments of rear leg joints
Tablica 3. Rezultati sila i momenata savijanja spojeva stražnjih nogu

Generally, the results indicated that the maximum force mean values of front leg joints with a smaller angle between the stretcher and leg were higher than the mean values of rear leg joints for all tenon lengths. The mean maximum force of the front leg joints (angle 102°) with a tenon length of 30 mm was 13.7 %, and with a tenon length of 32 mm 15.7 %, higher than the rear leg joints (angle 112°) with the same tenon length. The differences are partly the result of different configurations of test specimens of the front and rear leg joints, e.g. the different lengths of the specimen elements that were obtained during their construction.

Consequently, the values of maximum force were not comparable due to dimensional differences of these two groups of joints and, in this case, the maximum force cannot be an indicator of the joint strength.

Moreover, the results show differences among the mean values of the maximum force of joints constructed using two different tenon lengths (30 and 32 mm). The mean values of maximum force increased by 7.3 % (front leg joints – 102°) and 9.2 % (rear leg joints – 112°) as the tenon length decreased from 32 mm to 30 mm. These results contradict the data in the literature, which claim that joints became stronger as tenon



Figure 6 Characteristic joint failures Slika 6. Karakteristični lomovi spoja

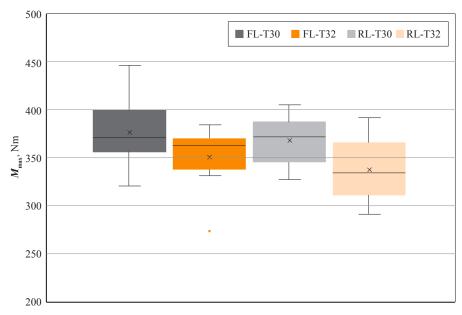


Figure 7 Box and whisker plot – distribution of maximum moment results of front leg joints (FL) and rear leg joints (RL) for tenon length of 30 mm and 32 mm (T30 and T32)

Slika 7. Dijagram raspodjele rezultata najvećeg momenta spojeva prednjih nogu (FL) i spojeva stražnjih nogu (RL) za duljinu čepa 30 i 32 mm (T30 i T32)

length increased. It is assumed that the results of the current work were influenced by a small difference between the examined tenon lengths (2 mm).

The left support reactions were $F_{Amax} = 0.842 \cdot F_{max}$ and $F_{Amax} = 0.802 \cdot F_{max}$ for front and rear leg joints, respectively. The values of the ultimate bending moment of the joints were calculated using the moment arm (1), i.e. the distance from the left support reaction to the force line, $a_A = 66.39$ mm for front leg joints and the distance $a_A = 77.66$ mm for rear leg joints.

Certainly, the percentages of the ratio of maximum moments of the joints with tenon lengths of 30 and 32 mm had the same values as the differences among the mean values of maximum force of the joints constructed using those two tenon lengths. However, the front leg joints (angle 102°) with a tenon length of 30 mm had only 2.0 %, and with tenon length of 32 mm only 3.8 %, higher mean maximum moments than the rear leg joints (angle 112°) with the same tenon length. The differences between the ratio of maximum force with two tenon lengths and the ratio of maximum moment with the same tenon lengths were the results of the specimen configuration of front and rear leg joints. Unlike maximum force, the values of maximum moment were comparable and, in this case, they can be an indicator of the strength of the front and rear leg joints.

The basic descriptive statistics of the maximum moment results of front and rear leg joints are presented in Tables 2 and 3. The coefficients of variation (CV) indicate the extent of variability in relation to the mean values of the maximum moment results of the four tested groups of joints. The front leg joints with tenon lengths of 30 mm (CV 9.4 %) and 32 mm (CV 9.4 %), as well the rear leg joints with a tenon length of 32 mm (CV 9.7 %), turned out to exhibit the highest and similar variability, whereas the rear leg joints with a tenon length of 30 mm (CV 6.7 %) were characterised by the lowest variability.

Comparative distributions of the maximum moment results of the tested groups of joints, i.e. the front and rear leg joints for two tenon lengths, are shown in a Box and Whisker plot (Figure 7). The median line of the RL – T32 box lies outside of all three other comparison boxes and indicates that there is a difference between this group of joints and the other tested joint groups. The interquartile range of the data group FL – T32 (with an outlier) is smaller compared to the other box lengths and indicates that the maximum moment results are less dispersed. Larger ranges for the group FL – T30 (the extreme values) indicate a wider distribution of the maximum moment results. The groups of maximum moment results show different kinds of asymmetry, i.e. skewness.

The t-test was used to determine if the means of two sets of maximum moment results were significantly different from each other. The *p*-values, shown in Table 4, are used to determine statistical significance in the hypothesis tests.

A significant difference (p = 0.1326) was not demonstrated with a 95 % level of confidence (α =0.05) between bending moments for the tenon lengths of 30 mm and 32 mm for the front leg joints, while a significant difference (p = 0.0371) was demonstrated between the bending moments for tenon lengths of 30 mm and 32 mm for the rear leg joints. According to the literature (Hajdarevic *et al.*, 2020), increasing the tenon **Table 4** P-values to determine statistical significance of difference between bending moments for front and rear leg joints with tenon lengths of 30 mm and 32 mm

Tablica 4. p-vrijednosti za određivanje statistički značajne razlike između momenata savijanja za spojeve prednjih i stražnjih	
nogu s čepovima duljine 30 mm i 32 mm	

Data source	Variable 1	Variable 2	<i>p</i> -value
Izvor podataka	Varijabla 1.	Varijabla 2.	p-vrijednost
Front leg	Tenon length 30 mm	Tenon length 30 mm Tenon length 32 mm	
prednja noga	<i>duljina čepa 30</i> mm	<i>duljina čepa 32</i> mm	0.1326
Rear leg	Tenon length 30 mm	Tenon length 32 mm	0.0371
stražnja noga	<i>duljina čepa 30</i> mm	mm duljina čepa 32 mm	
Tenon length 30 mm	Front leg joint	Rear leg joint	0.6137
<i>duljina čepa 30</i> mm	prednja noga	stražnja noga	0.0137
Tenon length 32 mm	Front leg joint	Rear leg joint	0.4138
<i>duljina čepa 32</i> mm	prednja noga	stražnja noga	0.4136

length increases the maximum moment and proportional moment of the mortise-and-tenon joints. The results in this paper indicate that the effect of tenon length is not always clear when there are small changes in it (the difference in tenon length was 2 mm).

A significant difference was not found between the bending moments for front and rear leg joints (different angles between the stretcher and leg) for the tenon length of 30 mm (p = 0.6137) or tenon length of 32 mm (p = 0.4138). In both cases, the bending moments of the front and rear leg joint specimens were analysed with the same tenon-and-mortise configuration of geometry.

The axial force and transversal force (internal, cross-section forces) of the front and rear leg joint specimens had significantly different values. However, these internal forces are not dominant, and in this case, it can be assumed that they had little effect on the stress state of loaded joints, i.e. joint strength.

4 CONCLUSIONS

4. ZAKLJUČAK

This paper analysed the joint strengths of two sets of specimens with different shapes and dimensions that were cut from real wooden frame constructions. The maximum applied load and bending moment of the front and rear leg joints of the crossed chair base were investigated. All specimens had the same tenonand-mortise geometry, while the difference in the joint angles was 10°.

The results showed that the joints with a smaller value of joint angle (front leg joints) had greater maximum force than those with a larger joint angle (rear leg joints). The results of maximum force were not completely comparable due to different lengths of specimen elements of front and rear joints. The maximum force increased less than 10 % as the tenon length decreased by 2 mm for both the front and rear leg joints.

The result of calculations showed that the joints with a smaller value of joint angle (front leg joints) had

a higher maximum bending moment than those with a larger joint angle (rear leg joints). However, the differences among the values of bending moment were only 2.0 % and 3.8 % for the tenon lengths of 30 mm and 32 mm, respectively. A significant difference was not revealed between the bending moments of joints with a smaller value of joint angle and the bending moments of joints with a larger angle for all tenon lengths. Significant differences between bending moments for the different tenon lengths were determined for rear leg joints, but not for front leg joints.

The approach presented here to determine and compare joint strength through testing different types of specimens is applicable to research and practice.

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University of Zagreb • Faculty of forestry and wood technology Testing laboratory for furniture and playground equipment Institute for furniture and wood in construction Svetošimunska cesta 23 HR-10000 Zagreb, Croatia Günay Özbay¹, Musa Atar², Ayhan Ozcifci¹

Synthesis of Phenolic Resin Reinforced with TiO₂ Nanoparticles and its Effect on Combustion Performance of Laminated Veneer Lumber (LVL)

Sinteza fenolne smole ojačane nanočesticama TiO₂ i njezin utjecaj na gorivost lamelirane drvne građe (LVL)

ORIGINAL SCIENTIFIC PAPER

Izvorni znanstveni rad Received – prispjelo: 30. 12. 2021. Accepted – prihvaćeno: 11. 5. 2022. UDK: 549.514.6; 630*83; 674.049.3 https://doi.org/10.5552/drvind.2023.0006 © 2023 by the author(s). Licensee Faculty of Forestry and Wood Technology, University of Zagreb. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license.

ABSTRACT • In this study, phenol-formaldehyde (PF) resin has been modified with titanium dioxide nanoparticles (nano-TiO₂) at a varying ratio from 0.05 wt.% to 1.5 wt.% to enhance the thermal properties and combustion performance of the resins. The effect of the nano-TiO₂ modification on the properties (chemical or thermal) of the resins was determined by Fourier to transform infrared (FT-IR) and thermal analysis (TGA) techniques. In addition, the combustion performance of laminated veneer lumber (LVL) samples bonded with the PF resin modified with nano-TiO₂ was tested. The result of the FT-IR analysis indicated that the modified PF resins had match peaks to the reference PF resin. These similarities of the peaks supported that the modified PF resins were successfully synthesise with phenol, formaldehyde, and nano-TiO₂. The PF resins modified by nano-TiO₂ demonstrated better thermal stability than the reference resin. The nano-TiO₂ modified PF resin exhibited a favourable influence on the combustion characteristics of LVL. In consequence, PF resin modified with nano-TiO₂ could be used as a combustion retardant adhesive in the wood industry.

KEYWORDS: adhesive; combustion; FT-IR; nano-chemicals; TiO, nanoparticles

SAŽETAK • U ovom je istraživanju fenol-formaldehidna smola (PF) modificirana nanočesticama titanijeva dioksida (nano-TiO₂) u različitim omjerima, od 0,05 tež.% do 1,5 tež.% kako bi se poboljšala njezina toplinska svojstva i svojstva gorivosti. Utjecaj modifikacije nanočesticama TiO₂ na svojstva smola (kemijska i toplinska) određen je Fourierovom transformiranom infracrvenom spektroskopijom (FT-IR) i termogravimetrijskom analizom (TGA). Osim toga, ispitana je gorivost uzoraka lamelirane drvne građe (LVL) lijepljene PF smolom modificiranom nano-

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česticama TiO₂. Rezultati FT-IR analize pokazali su da modificirane PF smole imaju jednake vrhove kao referentna PF smola. Te sličnosti vrhova potvrđuju da su modificirane PF smole uspješno sintetizirane s fenolom, formaldehidom i nanočesticama TiO₂. PF smole modificirane nanočesticama TiO₂ pokazale su bolju toplinsku stabilnost od referentne smole i povoljno su utjecale na gorivost LVL-a. Stoga zaključujemo da bi se PF smola modificirana nanočesticama TiO, mogla upotrebljavati kao ljepilo za usporavanje gorenja u drvnoj industriji.

KLJUČNE RIJEČI: ljepilo; gorenje; FT-IR; nanokemikalije; nanočestice TiO,

1 INTRODUCTION

1. UVOD

Adhesive plays a fundamental role in the strength and durability of wooden structures. Recently, adhesives reinforced by different types of chemicals have become a very significant area of research since they can be provided to improve dimensional stability and fire retardancy of wood constructions. In that case, phenolic resins are generally used as wood adhesives for outdoor applications. They are classified into two main types: novolac and resol. Novolac resins are synthesise by reacting to a molar excess of phenol and formaldehyde under acidic conditions. Resol resins are prepared by an alkaline reaction of phenol and formaldehyde. PF resol resin is generally preferred for the production of wood and wood-based composites (laminated veneer lumber (LVL), laminated strand lumber (LSL), oriented strand board (OSB), plywood, etc.), due to its faultless durability, strong bonding quality, water resistance, and mechanical strength. However, the resin has two main disadvantages. First, it is more expensive than other amino resins such as melamine-formaldehyde (MF) and ureaformaldehyde (UF) as the price and availability depend on petroleum. Secondly, the application of PF resin at high temperatures has poor thermal stability (Turunen et al., 2003; Pizzi, 1993; Kokten et al., 2020; Hassan et al., 2009; Farhaninejad et al., 2021; Park et al., 2002; Cheng et al., 2012; Lima García et al., 2018). Several studies around the world have shown that there are several methods to improve the combustion and thermal properties of the composites, such as reinforcing adhesive with micro and nano chemicals (Prasad et al., 2018; Hanumantharaya et al., 2021; Ozbay et al., 2021; Guerra et al., 2019). Kumar and co-workers studied the influence of nano-TiO₂ in enhancing the mechanical and thermal properties of the flax fibre reinforced epoxy composites. They reported that the nanofillers have specific characteristics that can improve the mechanical and thermal properties of the composite system (Kumar et al., 2016).

Nano-chemicals such as nano-SiO₂, nano-TiO₂, nano-AlO₃, and nano-ZnO have gained increasing interest due to their comprehensive industrial applications. In particular, these chemicals are widely used in different types of polymers synthesis for improving the desired properties (Mu et *al.*, 2011; Yuen *et al.*, 2009; Kavitha *et al.*, 2013; Zhang *et al.*, 2018; Prabhu *et al.*,

2021). There are many reports on adhesive modification with nano-chemicals including nano-SiO₂, nano-CaCO₃, nano-Al₂O₃, nano-TiO₂, and others to enhance the mechanical, physical, and chemical properties of adhesives in the literature (Zhai et *al.*, 2008; Zhang *et al.*, 2013; Ghosh *et al.*, 2016; Li *et al.*, 2016; Raghavendra *et al.*, 2021). However, there is less information about the effect of nano-chemicals on the thermal properties of adhesives (Pang *et al.*, 2018; Ataberk *et al.*, 2020; Gultekin *et al.*, 2021).

The purpose of this experimental study was to examine the effect of the addition of nano- TiO_2 on some properties of PF resins such as chemical, physical and thermal. The combustion behaviour of LVL bonded with modified PF resins was also identified by considering ASTM-E 160-50 Standard.

2 MATERIALS AND METHODS

2. MATERIJALI I METODE

2.1 Materials

2.1. Materijali

The chemicals applied in resin synthesis are analytical grades of NaOH (pellets) and nano-TiO₂ all purchased from Sigma Aldrich. The diameter of the nano-TiO₂ particle size was 50 nm. Phenol (liquid) and paraformaldehyde were supplied by GENTAŞ chemical industries (Turkey).

2.2 Preparation of test samples

2.2. Priprema ispitnih uzoraka

Laminated veneer lumbers (LVL) were produced from beech wood (Fagus Orientalis Lipsky) with a density of 0.58 g/cm³. Wood planks were purchased from a local supplier. Initially, LVL samples with the dimension of 25 mm x 25 mm x 500 mm were cut from planks and acclimatise at a temperature of (20 ± 2) °C and (65 ± 5) % relative humidity until they reached an equilibrium in moisture content. Then the resin was applied on one surface (200 g/m²) of the lamella using a hand brush. The pressure, temperature and duration were 0.2 N/mm², 130 °C, and 15 min, respectively. LVL samples were conditioned for 10 days in the climate room at (20 ± 2) °C and (65 ± 5) % relative humidity. Combustion test samples (Figure 1) dimensions of 13 mm x 13 mm x 76 mm were cut from the laminated lumbers according to ASTM E 160.

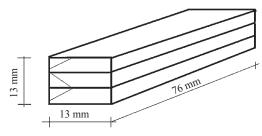


Figure 1 Combustion test samples Slika 1. Uzorci za ispitivanje gorivosti

2.3 PF resin synthesis procedure 2.3. Postupak sinteze PF smole

The PF resin was prepared in a glass-made reactor endowed with a mechanical stirrer, cooling condenser and a water bath. Control PF (unmodified) was synthesise in the reactor according to the following procedure: the reactor was charged with phenol (90 %), paraformaldehyde (46 %), and pure water. After heating the mixture to 40 °C, the first part of the NaOH solution (50 wt%) was added slowly. The temperature was slowly increased to 60 °C. The temperature of the components was increased to 90 °C and maintained at this temperature for 60 min. After that, the second proportion of NaOH solution (50 wt%) was put into the reactor to regulate the pH value of about 11 at 60 °C. When the reaction was completed, the temperature of the PF resin was cooled to near room temperature. Similar procedures as described above were carried out for the synthesis of the PF resin as the control PF resin and other nano-PF resins with varying nano-TiO₂ ratios (0.05 wt.%, 0.10 wt.%, and wt.% 0. 15). Before the synthesis process, various amounts of nano-TiO₂ were mixed with phenol, paraformaldehyde, and pure water using a mechanical stirrer at about 600 rpm until a homogeneous appearance was achieved at room temperature.

2.4 Resin characterisation methods 2.4. Metode karakterizacije smole

The effect of nano-TiO₂ addition on the physical properties such as pH value, gel time, dynamic viscosity, and the solid content of the resin was examined. The pH value was measured using a previously calibrated digital pH meter (TES-1380 pH meter). The viscosity was measured by Brookfield digital viscometer (model: Dv-I Prime), number spindle 1 at 2.09 rad/s (20 rpm) at 25 °C. This test method is described in ASTM D1084-08 standard. The solid content of resins was calculated according to ASTM D3529M-97. The liquid PF resin characterisation was subjected to Infrared spectrometry (FT-IR, model- Alpha) by direct transmittance using KBr pellet technique. Infrared spectra were scanned in a range wavenumber between 4000-400 cm⁻¹. The thermal stability of the liquid PF resins was analysed by thermo-gravimetric technique on a TGA (HITACHI STA 7300). Appx. 10 mg of the sample was used with a heating rate of 10 °C /min under nitrogen atmospheres flow rate of 30 ml/min. The curves were recorded by heating from 20 °C to 700 °C. Three replicates were used for each resin.

2.5 Combustion test

2.5. Test gorivosti

A combustion test was performed in a combustion test stand based on ASTM E 160-50 standards. In accordance with this standard, laminated samples were conditioned until they reached a moisture content of 7 % before the combustion test. In the combustion test, 24 pcs test samples were aligned as a rectangular prism and burned. 3 different tests were made for each combustion test and 72 pcs test samples were used for each of them (Figure 2).

Combustion tests were performed in three steps: combustion from the flame, self-combustion, and com-



Figure 2 Test samples and combustion test Slika 2. Ispitni uzorci i test gorivosti



bustion as an ember. Each sample batch was weighed before the test was put on a wire stand. The samples on the stand were placed vertically to the below and above stands. The fire distance from the lower maker type outlet was set as (25 ± 1.3) cm when the device was empty, and the gas pressure was set as 0.5 kg/cm^2 in the manometer. When the gas was burned, the temperature in the thermocouple funnel was tried to be fixed at (200 ± 8) °C. The flame source was centred at the bottom of the sample pile, and then the burning of the flame source was maintained for 3 minutes. The temperature change of combustion was also measured with a thermometer (ASTM E 160-50, 1975).

2.6 Evaluation of data

2.6. Evaluacija podataka

Multiple variance analysis was utilised to identify both the combustion properties and the effects of rate TiO_2 on combustion with flame sourced (FSc), flameless: self-burning (FLs) and combustion period within the context of fire test. Furthermore, the Duncan test was applied between groups at the end of the analysis in case the differences were recognised to test the homogeneity of the groups.

3 RESULTS AND DISCUSSION 3. REZULTATI I RASPRAVA

3.1 Resin properties

3.1. Svojstva smole

The basic physical properties of modified resins and control PF resin are given in Table 1. The solid content ranges from 43.14 % to 44.51 %. The solid content of the PF resin-modified with nano-TiO₂ varied depending on the additive ratio of nano-TiO₂. The pH value of PF resins ranged from 11.50 to 10.90. The viscosity of PF resins ranged from 294 to 334 cPs. The viscosity of PF resins modified with nano-TiO₂ was more viscous than that of the control PF resin, so it can be said that the addition of nano-TiO₂ up to 0.15 % did not reveal any significant trend in the fundamental physical properties of the PF resin.

PF resin modified with nano-TiO₂ was characterised by FT-IR spectral analysis to determine the effect of adding nano-TiO₂ on the chemical properties of the PF resin. The FT-IR spectra of the resins reveal that the functional groups observed were similar to each other, indicating structural similarity between PF and modified PF resins. (Fig. 3). Mainly, the bonds that appeared in the spectrum of PF were also observed in the spectra of modified PF resins. IR spectrum of all samples had a strong wideband between 3400 and 3200 cm⁻¹, assigned to OH stretching vibrations (1). The absorption peak between 2950 cm⁻¹ and 2800 cm⁻¹ was assigned to C-H stretching frequencies from methyl and methylene groups (2). The band at 1750-1700 cm⁻¹ showed the aromatic ring C=C stretching vibrations (3). The peaks at about 1600 and 1480 cm⁻¹ corresponded to aromatic ring stretching bands (4), and the peak at 1250 - 1200 cm⁻¹ was assigned to the C-O stretching in the aromatic ring for all samples (5). The peak was observed in the range of 800-750 cm⁻¹ presenting C-H out-of-plane bending vibrations of the aromatics (6). (Cui et al., 2017; Poljansek and Krajnc, 2005; Ibrahim et al., 2011; Horikawa et al., 2003; Wang et al., 2009; Wang et al., 2011). It was confirmed that the synthesis of nano-TiO, PF resin followed a reaction pathway similar to that of the control PF resin.

Thermal degradation and stability of the PF and PF resin modified with nano-TiO₂ were studied by TGA analysis. Figure 4 shows the TGA graphs of the PF resin and modified PF resins. The control PF resin was stable up to 250 °C. The maximum rate loss in control PF resins occurs at the temperature range of 300 - 450 °C. The PF resins modified with nano-TiO₂ were more thermally stable than the control PF resin in the range of 200 - 600 °C. The thermal stability of PF resin modified with nano-TiO₂ showed a rising trend with adding nano-TiO₂. TGA analysis results were consistent with previous studies on the thermal stability of adhesives modified with nano-chemicals (Yuan *et al.*, 2008; Lin *et al.*, 2009; Ekrem *et al.*, 2018; Sahoo *et al.*, 2017; Zheng *et al.*, 2021).

3.2 Combustion properties 3.2. Gorivost

The results of multiple variance analysis regarding the effect of combustion type and content of nano- TiO_2 on combustion temperature (°C) are given in Table 2.

The effects of combustion type and amount of TiO, on combustion temperature were statistically sig-

Table 1 Basic physical properties of PF resins modified with nano-TiO₂ **Tablica 1.** Osnovna fizička svojstva PF smola modificiranih nanočesticama TiO₂

	5 2			
Resin	Solid content, %	pH at 20 °C	Viscosity, cPs at 25 °C	
Smola	Sadržaj suhe tvari, %	<i>pH pri 20</i> °C	Viskoznost, cPs pri 25 °C	
Control PF	43.14	11.90	294	
0.05 % nano-TiO ₂ - PF	43.75	11.78	319	
0.10 % nano-TiO ₂ - PF	44.16	11.71	325	
0.15 % nano-TiO ₂ - PF	44.51	11.50	334	

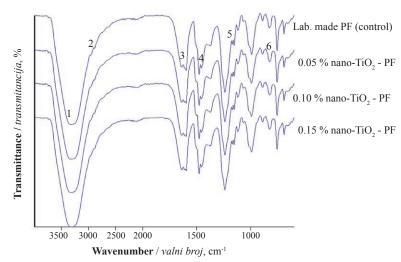


Figure 3 FT-IR spectra of resins (control PF and modified PF) **Slika 3.** FT-IR spektar kontrolne i modificirane PF smole

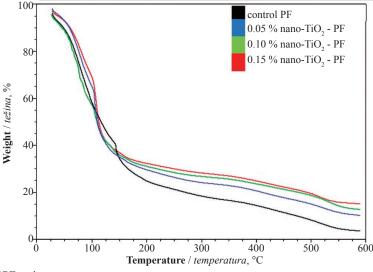


Figure 4 TGA curves of PF resins **Slika 4.** TGA krivulje PF smola

nificant (α =0.05). Duncan test was applied to evaluate the differences between the groups. The results of the Duncan test are presented in Table 3.

It may be seen in Table 3 that the highest temperature depending on the combustion type in flame sourced combustion was 536 °C, and the lowest in Cb was 260.5 °C; on the other hand, as for the amount of nano-TiO₂, the highest value was measured in 0.01 nano-TiO₂ – 389.1 °C, the lowest in 0.05 nano-TiO₂ – 389.1 °C. Lin and co-workers reported that nano-PF

resins were thermally stable up to a temperature of about 300 °C. This thermal stabilisation may be explained by new structures, which can be formed during resin synthesis (Lin *et al.*, 2006). With respect to combustion type and content of nano-TiO₂, the highest temperature value was measured in flameless+0.15 - 560.3 °C, and the lowest in charcoal burning+0.15 - 245 °C. According to these results, when the % amount of nano-TiO₂ was increased, the combustion temperature decreased.

Table 2 Multiple variance analysis results
Tablica 2. Rezultati analize višestruke varijance

Source	Degrees of freedom	Sum of squares	Mean square	F value	Prob.
Izvor	Stupnjevi slobode	Zbroj kvadrata	Srednji kvadrat	F-vrijednost	
Factor A	460898.604	460898.604	230449.302	749.0285	0.0000
Factor B	12328.539	12328.539	4109.513	13.3571	0.0000
AB	24913.756	24913.756	4152.293	13.4962	0.0000
Error	7383.943	7383.943	307.664		
Total	505524.842	505524.842			

Factor A – combustion type, Factor B – nano-TiO2+control, coefficient of variation: $4.50 \% / faktor A - vrsta izgaranja; faktor B - nano-TiO_2 + kontrola; koeficijent varijance: <math>4,50 \%$

Table 3 Duncan test results Tablica 3. Rezultati Duncanova testa

Combustion type* / Vrsta izgaranja*	<i>X</i> , ⁰C	HG
Flame sourced / s izvorom plamena (FSc)	536.0	Α
Flameless / bez plamena (FLs)	372.2	В
Charcoal burning / pougljenje (Cb)	260.5	С
Nano chemical ** / Nanokemikalije**		
Control / kontrolni uzorci (C)	419.3	Α
0.10% TiO ₂	389.1	В
0.15% TiO ₂	380.2	BC
0.05% TiO ₂	369.7	С
Type of process *** / Vrsta procesa ***		·
Flameless + 0.15 % TiO_2 bez plamena + 0,15 % TiO_2	560.3	А
Flameless + 0.10 % TiO ₂ bez plamena 0,10 % TiO ₂	538.3	AB
Flameless + C / $bez plamena + C$	527.7	В
Flameless + 0.05% TiO ₂ bez plamena + 0.05% TiO ₂	517.7	В
Flame sourced + C / s izvorom plamena + C	449.0	С
Flame sourced + $0.05 \% \text{ TiO}_2$ s izvorom plamena + $0.05 \% \text{ TiO}_2$	371.3	D
Flame sourced + 0.10 % TiO_2 s izvorom plamena + 0,10 % TiO_2	364.8	D
Flame sourced + $0.15 \% \text{ TiO}_2$ s izvorom plamena + $0,15 \% \text{ TiO}_2$	303.7	Е
Charcoal burning + C / $pougljenje + C$	281.3	EF
Charcoal burning + $0.10 \% \text{ TiO}_2$ pougljenje + $0,10 \% \text{ TiO}_2$	264.0	FG
Charcoal burning + 0.05 % TiO_2 pougljenje + 0,05 % TiO_2	251.7	FG
Charcoal burning + $0.15 \% \text{ TiO}_2$ pougljenje + $0.15 \% \text{ TiO}_2$	245.0	G

*LSD value = 14.54, ** LSD value = 16.79, *** LSD value = 29.07, X – Average, HG – Homogeneous group *LSD vrijednost = 14,54, ** LSD vrijednost = 16,79, *** LSD vri-

jednost = 29,07, X – srednja vrijednost, HG – homogene grupe

Multivariance analysis (Table 4) was applied to determine the effect of both the amount of nano-TiO, and combustion type on the length of burning time or combustion duration. Considering the significance of the Duncan test, each test group was compared with each other and with themselves (in Table 5).

The multivariance analysis was carried out to test the results obtained from the combustion tests. In accordance with the multivariance analysis, the effects of nanoTiO₂ and combustion type on combustion duration were statistically significant. The interactions between nano-TiO₂ and combustion type were statistically important $(P \leq 0.05)$. The means of variation sources that were found to be significant were compared using Duncan's test and the average values are summarised in Table 5.

According to total combustion duration, in terms of the effect of combustion type on the combustion duration, the highest value was 40.58 min in charcoal burning, the lowest was 3.87 min. in Flameless. As for the amount of nano-TiO₂ (%), the highest value was 61.61 in 0.01 TiO₂, the lowest was $3.76 \text{ min in } 0.05 \text{ TiO}_2$. The interaction of combustion type and the amount of nano- TiO_{2} , total combustion duration +0.05 TiO₂ showed the longest combustion duration as 99.10 min, the shortest duration was obtained in flameless +0.15 nano-TiO₂ -3.867 min. The duration of combustion was reduced by the amount of nano-TiO₂. The nano-chemical was quite effective in flame sourced combustion, extinguishing the flame and decreasing the risk of fire enhancement. This situation positively affected especially the charcoal duration at the end of the combustion. Similar to other reported results, nano-chemicals improved the thermal stability of phenolic resin (Zheng et al., 2003; Ma et al., 2005; Wang et al., 2011; Özbay et al., 2021).

4 CONCLUSIONS

4. ZAKLJUČAK

The purpose of this study was to evaluate the influence of adding nano-TiO₂ on the chemical, physical and thermal characteristics of PF resins. Additionally, combustion properties of laminated veneer lumber (LVL) bonded with the PF resins modified with different content of nano-TiO₂ were determined. The following can be concluded:

The addition of nano-TiO, up to 0.15 % did not reveal any significant trend in the fundamental physical properties such as solid content, pH value and viscosity of the PF resin.

The synthesis of nano-TiO, PF resin followed a reaction pathway similar to that of the control PF resin as confirmed by FT-IR spectroscopy analysis.

TGA analysis indicated that the increase of nano-TiO₂ content in the resin formula enhanced the thermal stability of the PF resin.

Table 4 Results of multivariance analysis	
Tablica 4. Rezultati analize višestruke varija	ance

Source Izvor	Degrees of freedom Stupnjevi slobode	Sum of squares Zbroj kvadrata	Mean square Srednji kvadrat	F value <i>F-vrijednost</i>	Prob.
Factor A	2	15476.264	7738.132	521.0473	0.0000
Factor B	3	10437.310	3479.103	234.2655	0.0000
AB	6	5347.205	891.201	60.0090	0.0000
Error	24	356.427	14.851		
Total	35	31617.206			

Factor A – combustion type, Factor B – nano-TiO, + control, coefficient of variation: 4.40 % / faktor A – vrsta izgaranja; faktor B – nano-TiO, + kontrola; koeficijent varijance: 4,40 %

Table 5 Effects of nano-TiO₂ content (%) and combustion type on length of burning time, or combustion duration (min) **Tablica 5.** Utjecaj sadržaja nano-TiO₂ (%) i vrste izgaranja na duljinu gorenja odnosno na trajanje izgaranja (min)

Combustion type* / Vrsta izgaranja*	X	HG
Flameless / bez plamena	3.875	С
Charcoal burning / pougljenje	40.58	В
Total combustion duration	52.63	А
ukupno trajanje izgaranja	52.05	A
Nano-chemical** / Nanokemikalije**		
Control / kontrolni uzorci (C)	21.44	С
0.10% TiO ₂	61.62	Α
0.15% TiO ₂	26.02	В
0.05% TiO ₂	20.36	С
Type of process*** / Vrsta procesa***		
Total combustion duration + 0.15 TiO_2 <i>ukupno vrijeme izgaranja</i> + 0,15 TiO_2	99.10	А
Charcoal burning + 0.05 TiO ₂ / $pougljenje$ + 0,05 TiO ₂	82.00	В
Total combustion duration + 0.10 TiO_2 ukupno vrijeme izgaranja + 0,10 TiO_2	44.20	С
Total combustion duration / ukupno vrijeme izgaranja	35.00	D
Total combustion duration $+ 0.15 \text{ TiO}_2$ <i>ukupno vrijeme izgaranja</i> $+ 0,15 \text{ TiO}_2$	32.20	D
Charcoal burning $+ 0.10 \text{ TiO}_2$ pougljenje $+ 0.10 \text{ TiO}_2$	30.00	DE
Charcoal burning / pougljenje	25.33	Е
Charcoal burning $+ 0.15 \text{ TiO}_2$ pougljenje $+ 0.15 \text{ TiO}_2$	25.00	Е
Flameless – control / bez plamena – kontrola	4.000	F
Flameless + 0.15 TiO_2 bez plamena + 0,15 TiO_2	3.867	F
Flameless + 0.10 TiO_2 bez plamena + 0,10 TiO_2	3.865	F
Flameless + 0.05 TiO_2 bez plamena + 0,05 TiO_2	3.767	F

*LSD value = 3.069, ** LSD value = 3.688, *** LSD value= 6.388, X – Average, HG – Homogeneous group

*LSD vrijednost = 3,069, ** LSD vrijednost = 3,688, *** LSD vrijednost = 6,388, X – srednja vrijednost, HG – homogene grupe

LVL samples bonded with nano-TiO₂ modified PF resin exhibited better fire resistance as compared to LVL bonded with PF resin (control). The PF resin modified with nano-TiO₂ can be used as a combustion resistance adhesive in the wood industry.

 $Nano-TiO_2$, due to its availability and remarkable thermal properties, can be used as reinforcement in phenolic resin production.

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Structural and Design Values of Solid Timber Beams and Glued Laminated Timber Beams of *Dipteryx panamensis* and *Hieronyma alchorneoides* Wood from Fast-Growth Plantation

Strukturne i projektirane vrijednosti lameliranih greda i greda od cjelovitog drva *Dipteryx panamensis* i *Hieronyma alchorneoides* s brzorastućih plantaža

ORIGINAL SCIENTIFIC PAPER

Izvorni znanstveni rad Received – prispjelo: 9. 1. 2022. Accepted – prihvaćeno: 11. 5. 2022. UDK: 630*83; 674.06 https://doi.org/10.5552/drvind.2023.0007 © 2023 by the author(s). Licensee Faculty of Forestry and Wood Technology, University of Zagreb. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license.

ABSTRACT • <u>Dipteryx panamensis</u> and <u>Hieronyma alchorneoides</u> are two species of high specific gravity used in reforestation programs in Costa Rica, but they lack products with structural value for commercialization. In order to introduce the wood of these two species in the market, the objective was established to study the behavior of solid timber beams and glued laminated timber beams of two cross sections ($2 \text{ cm} \times 10 \text{ cm}$ and $2 \text{ cm} \times 15 \text{ cm}$) and establish the design values in bending test. The results showed that the bending design values (f_b) ranged from 2 to 26 MPa in glued laminated timber beams, while in solid timber beams, fb ranged from 6 to 15 MPa. In the shear design values (f_y), the variation was from 0.29 to 0.67 MPa in glue laminated timber beams and from 1.80 to 2.58 MPa in solid timber beams. It was also found that the <u>D. panamensis</u> beams showed higher values than <u>H.</u> <u>alchornoides</u> beams. Finally, it was established that glued laminated timber beams when used in floor and roof construction.

KEYWORDS: *tropical species; bending; shear; mezzanine; glulam; mass timber*

SAŽETAK • <u>Dipteryx panamensis</u> i <u>Hieronyma alchorneoides</u> dvije su vrste drva velike specifične gustoće koje su uvrštene u program pošumljavanja u Kostarici, ali se ne iskorištavaju za strukturne proizvode kako bi se komercijalizirale. Da bi se te dvije vrste drva plasirale na tržište, istraženo je ponašanje lameliranih greda i greda od cjelovitog drva s dva različita poprečna presjeka (2 cm × 10 cm i 2 cm × 15 cm) te su ispitivanjem savijanja

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utvrđene projektirane vrijednosti. Rezultati su pokazali da su se projektirane vrijednosti savijanja (f_b) kretale u rasponu od 2 do 26 MPa za lamelirane grede te od 6 do 15 MPa za grede od cjelovitog drva. Projektirane vrijednosti smicanja varirale su od 0,29 do 0,67 MPa za lamelirane grede te od 1,8 do 2,58 MPa za grede od cjelovitog drva. Za grede od drva <u>D. panamensis</u> izmjerene su veće vrijednosti ispitivanih svojstava nego za grede od drva <u>H. alchorneoides</u>. Konačno, utvrđeno je da su lamelirane grede pokazale bolja svojstva savijanja i veće projektirane vrijednosti od greda od cjelovitog drva, što rezultira većim vrijednostima raspona kada se rabe na podu ili krovu.

KLJUČNE RIJEČI: tropske vrste; savijanje; smicanje; mezanin; lamelirana greda; cjelovito drvo

1 INTRODUCTION

1. UVOD

Costa Rica is a small Central American country currently characterized by the conservation of its natural resources, especially natural forests (Allen and Vásquez, 2017). However, the country has gone through several stages in the harvesting of natural forests, and the most critical point occurred in the 1980s, when forest cover decreased to critical levels (Stan and Sanchez-Azofeifa, 2019). The selective harvesting of natural forests resulted in the production of bigger logs (Johns, 1988), where large cross section lumber was obtained (Bello, 2020). In the market, lumber for furniture fabrication could be find with dimensions of 2.5 cm thickness, over 40 cm in width and length over 3.36 meters, while in the case of timber, the dimensions could be 10 cm in thickness, over 15 cm in width and length of up to 5.5 m. Thus, before the 1980s, buildings (houses and buildings) were characterized by the use of wood in all elements of high structural demand, such as beams and columns (Serrano-Montero and Moya, 2011).

After this critical period, the government encouraged reforestation programs through forestry incentives, with the aim to reduce the pressure on natural forests and to increase a source of sawlog supply for the country (Quesada-Mateo and Solis-Rivera, 1990). These programs were maintained for several years, resulting in the fact that commercial plantations currently supply 60 % of the volume of sawlogs consumed in Costa Rica (Ugalde, 2021). Throughout this process, about 20 forest species have shown adequate results in terms of growth and production. So, farmers have accepted many species to establish commercial plantations (Nichols and Vanclay, 2012). Fast-growing species, with rotation periods of less than 25 years, such as Dipteryx panamensis, Terminalia amazonia, Vochysia guatemalensis, Cordia al*liodora* and *Hieronyma alchorneoides* (native species) and Tectona grandis, Cupressus lusitanica, Acacia mangium and Gmelina arborea (exotic species), have been extensively studied (Petit and Montagnini, 2006) and have shown excellent results in forest plantations in Costa Rica (Petit and Montagnini, 2006; Nichols and Vanclay, 2012). However, it has been observed that most of the above species are concentrated in moderate density wood, used as lumber for furniture manufacturing (Moya et al., 2021).

D. panamensis (almendro) and H. alchorneoides (pilon) are two species used in reforestation programs due to their high specific gravity and therefore their high values in structural properties. However, three main problems have been identified in wood from forest plantations: (i) presence of warp during sawmilling process, (ii) high incidence of drying defects in the drying process and (iii) lack of product development for marketing. The first two have been recently addressed by Moya et al. (2021) and Moya and Tenorio (2021). The lack of products for the commercialization of these species is associated with two aspects: (i) they have a density greater than 0.5 g/cm^3 , so they are classified as high-density timber, and consequently they cannot be used in the manufacture of furniture and pallets fabrication, which are the main markets for plantation sawlog, and (ii) the other most influential aspect is that the heartwood color of plantation timber has lighter color in relation to natural forest wood (Moya and Tenorio, 2021; Moya et al., 2021).

D. panamensis and *H. alchornoides* are classified as high wood density considering their characteristics of trees from natural forests in Costa Rica (Moya *et al.*, 2021). The wood from these trees has an established market and is used for the construction of trusses, floors and columns to support walls, bridges, and truck, among others, that is, in uses where there is an important demand for structural strength (Moya and Tenorio, 2021). However, wood from trees growing in fast growth plantations showed a decreasing trend in specific gravity in relation to wood from natural forests (Senft *et al.*, 1985). However, this decrease should not be a problem for plantation wood to be introduced in the same market sector established for wood from trees from natural forest (Moya *et al.*, 2021).

One way to mitigate the limitations of these species is to develop new processing options and not only to minimize problems during the sawing or drying process (Moya *et al.*, 2021, Moya and Tenorio, 2021 and 2022). It is also vital to develop highly engineered products that would enable wood from these species growing in fast growth plantation to competitively enter new markets (Zobel, 1981). A good example is the fabrication of glued laminated timber beams (Moody and Hernandez, 1997). This type of product is manufactured with laminates of limited thickness, with a certain degree of structural grading of its layers, and glued with structural adhesives (Kitek Kuzman *et al.*, 2010), but may present some differences in structural values in relation to solid wood (Falk and Colling, 1995). For example, elements in compression are of higher strength, but in bending test show a higher modulus of elasticity and a lower shear strength than solid timber beams (Ndong Bidzo *et al.*, 2021).

Although glued laminated timber beams and solid timber beams are produced from wood of relatively uniform quality, variations are always present (Moody and Hernandez, 1997), so they need to be standardized in order to establish different design values (Morin-Bernard *et al.*, 2021). Thus, the present work aims to study the behavior of solid timber beams and glued laminated timber beams made of *D. panamensis* and *H. alchorneoides* wood from fast growth plantations in two cross sections (2 cm \times 10 cm and 2 cm \times 15 cm) and stablish the design values in bending test.

2 MATERIALS AND METHODS

2. MATERIJALI I METODE

2.1 Site and characteristics of the plantation

2.1. Položaj i obilježja plantaže

Two fast-growth plantations were sampled: one of *H. alchorneoides* and another of *D. panamensis*. At the time of sampling, the *H. alchorneoides* plantation was 12 years old and had a density of 450 trees/ha, while the *D. panamensis* plantation was 16 years old and had a density of 550 trees/ha. More details on the characteristics of the plantations can be found in Moya and Tenorio (2021) and Moya *et al.* (2021).

2.2 Sampling and sawing trees

2.2. Uzorkovanje i piljenje stabala

Approximately 70 trees of each species were sampled, close to the average diameter of each plantation, 19.7 cm for *H. alchorneoides* and 17.9 cm for *D. panamensis*. Half of the logs were sawn using a typical cutting pattern for timber production in Costa Rica (Serrano-Montero and Moya, 2011), where a semi-log was obtained, which was sawn into 2.5 cm thick timber (Figure 1a). From the other half of the logs, a 6.2 cm thick central block was obtained across the width of the log (Figure 1b). Logs were sawn using a band saw and a single-cut resaw. The 2.5 cm and 6.2 cm timber were dried using a drying schedule proposed by Moya and Tenorio (2022).

2.3 Timber dimension and beam fabrication2.3. Dimenzije drvene građe i izrada greda

The timber was dried using the drying schedule proposed by Moya and Tenorio (2022) with a target moisture content of 12 %. Extensive details of the drying process for the two species can be found in Moya and Tenorio (2022). After drying, timber of 2.5 cm in thickness was planed to 2 cm × 6.5 cm × 270 cm, while timber of 6.2 cm was planed to 5.0 cm. Two type of timber beams (solid and glued laminated) were prepared with length of 270 cm of two different cross dimensions: (i) 5 cm × 10 cm and (ii) 5 cm × 15 cm. For solid timber beams, the dried timber with 5 cm in thickness was cut to the width of 10 cm and 15 cm. From each cross-section, 15 solid timber beams were fabricated in two cross sections and a length of 270 cm: (i) 5 cm × 10 cm

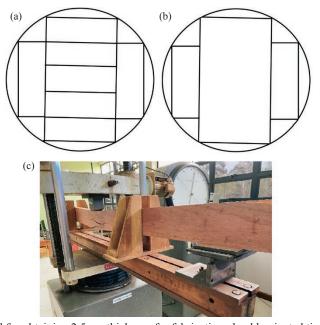


Figure 1 Sawing pattern used for obtaining 2.5 cm thickness for fabricating glued laminated timber beams (a), sawing patter used for obtaining 6.2 cm thick solid timber beams (b) and three-point bending test (c) **Slika 1.** Način piljenja za dobivanje 2,5 cm debelih elemenata za izradu lameliranih greda (a), način piljenja za dobivanje 6,2 cm debelih elemenata za izradu greda od cjelovitog drva (b) i ispitivanje savijanja u tri točke (c) with 6 lamellas of 20 cm in thickness and (ii) 5 cm \times 15 cm with 8 lamellas of 20 mm in thickness. A total of 15 glued laminated timber beams were constructed for each cross section, for a total of 60 beams (2 different types of beams \times 2 cross-sections \times 15 beams). Advantage EP-950A® isocyanate polymer emulsion (EPI)+catalyst 200 Franklin® (isocyanate polymer) adhesive system (Franklin Adhesives and Polymers, OHIO, USA) was used. The adhesive was applied at a weight of 200 g/m² on one side of lamellas. The lamella was placed on a balance and the required amount of adhesive was applied with micropore rollers. The pressing was performed with an ITALPRESSE PL/9/SCF/8 hydraulic press (Italpresse S.A., Bergamo, Italy), at a pressure of 8.0 MPa for 3600 s.

2.4 Bending test

2.4. Ispitivanje savijanja

The solid and glued laminated timber beams were tested by three-point static bending test over a span of 2.10 m as shown in Figure 1c. Testing was conducted with a Tinus Olsen Super L universal testing machine, with the capacity of 60 tons. The test conditions, load speed and deflection determination were compliant with ASTM D198-21a (ASTM, 2021a). A displacement sensor (LVDT) was placed at the center of the beam under the king post, to measure the vertical displacement at the time of load application, then load and displacement were measured each 30 μ s. From the results of these tests, the modulus of elasticity (*MOE*) and modulus of rupture (*MOR*) were determined using Eq. 1 and 2, respectively. The shear stress (τ) in bending was determined according to Eq. 3.

$$MOE = \frac{0.852 \cdot F_{\rm LP} \cdot L^3}{48 \cdot I \cdot y} \,({\rm GPa}) \tag{1}$$

$$MOR = \frac{F_{\max} \cdot L \cdot \frac{H}{2}}{6 \cdot I} \cdot 0.0981 \,(\text{MPa})$$
(2)

$$\tau(\text{MPa}) = \frac{3 \cdot F_{\text{max}}}{4 \cdot b \cdot H} \cdot 0.0981$$
(3)

Where: $F_{\rm LP}$ – load at proportional limit (kgf), $F_{\rm max}$ = rupture load (kgf), L – span (cm), I – moment of inertia (cm⁴) for rectangular form, y – deflection (cm), b – beam width, H – beam depth (cm), 0.0000981 conversion units from kgf/cm² to GPa and 0.0981 – conversion units from kgf/cm² to MPa, and 0.852 is derived from general equation for *MOE* for one load (Eq. 3).

In addition, the type of failure that occurred in the two types of beams was determined. For glued laminated timber beams, the type of failure was first categorized according to the location of the failure in the lamina number, with L1 being the lamina where the load is applied and Ln being the lamina farthest away from the load; it was L6 for the 10 cm high beams and L8 for the 15 cm high beams (Figure 2a-b). Second, it was determined if the failure was due to delamination (Figure 2c), which refers to the separation of the glueline between two lamellae, or if any lamellae that make up the beam failed; this type of failure can be of two types: tension and shear (Figure 2d-e). Meanwhile, for solid timber beams, 5 types of failure were established according to ASTM D-143 standards (ASTM, 2021b): simple tension (Figure 2f), cross-grain tension (Figure 2g), splintering tension (Figure 2h), brash tension (Figure 2i) and horizontal sheer (Figure 2j).

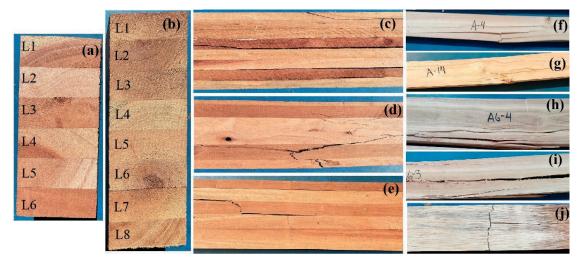


Figure 2 Number of laminates in glued laminated timber beams of 5 cm \times 10 cm (a) and 5 cm \times 15 cm (b), types of failures in glued laminated timber beams: delamination of glued line (c) and failure in lamina by tension (d) or shear (e), and failure in solid timber beams: simple tension (f), cross-grain tension (g), splintering tension (h), brash tension (i), and horizontal sheer (j) in static bending

Slika 2. Broj lamela u lameliranim gredama presjeka 5 cm \times 10 cm (a) i 5 cm \times 15 cm (b). Vrste lomova u lameliranim gredama: lom po ljepilu (c) i lom po lameli zbog napetosti (d) ili smicanja (e); vrsta loma u gredama od cjelovitog drva: jednostavna napetost (f), napetost u poprečnom presjeku (g), cijepanje (h), trzajna napetost (i) i horizontalni pomak pri statičkom savijanju (j).

2.5 Density, moisture content and stress in shear parallel to grain

2.5. Gustoća, sadržaj vode i smično naprezanje u smjeru vlakanaca drva

Following bending tests, a 5-cm cross-section was extracted from each beam. Its volume and weight were measured, and density (kg/m³) was calculated. Then, the sample was oven-dried at 103 °C for 24 hours, and moisture content was determined. Density value was later used to calculate the total weight of the beam. Glue line test was used for determining stress in shear parallel to the grain in glued laminated timber beam and shear resistance of solid timber beam. A sample of approximately 20 cm length was taken from each beam, from a section away from the failure area. From this, two samples of 6.5 cm in length were extracted (60 samples in total) and tested according to ASTM D905 (ASTM, 2021c) for glued laminated timber beam (30 samples) and ASTM D143 (2021b) for solid timber beam (30 samples).

2.6 Derivation of design values

2.6. Deriviranje projektiranih vrijednosti

Design values were derived from *MOE* and *MOR* values of glued laminated timber and solid timber beams tested in bending test, and its applicability will be shown as beam in flexion applications. In the derivation of design values, the beam was structurally analyzed in two different ways: (i) bending capacity, and (ii) deflection in the span. For bending capacity, the superimposed load in different spans was determined, considering only maximum stress of the transverse section.

For the derivation of the design values, a normal distribution of the *MOR* and *MOE* obtained for each type of beam was assumed. Then using the t-student statistical distribution function, the basic stress LRFD is obtained according to Eq. 4 and transformed into ASD stress by applying Eq. 5. This step was not performed for the *MOE* because it was not necessary. Next, the nominal bending strength (ΦMn), nominal shear strength (ΦVn) and bending stiffness (*EI*) were calculated, considering Eqs. 6, 7 and 8, respectively.

$$F_{\rm LRFD} = \overline{X} \cdot \left(1 - t_{\rm f} \cdot \frac{CV}{100} \right) \tag{4}$$

$$F_{\rm ASD} = \frac{F_{\rm LRFD}}{K_F \cdot \phi} \tag{5}$$

$$\Phi Mn = f_{\rm b} \cdot S \cdot K_{\rm F} \cdot \phi \tag{6}$$

$$\Phi Vn = \frac{2}{3} \cdot f_{v} \cdot A \cdot K_{F} \cdot \phi \tag{7}$$

$$EI = MOE I$$
 (8)

Where: \overline{X} – average value of data, CV – coefficient of variation of data (percentage), t_f – *t*-Student factor for a 95 % exclusion level according to sample size, $K_{\rm F}$ – format conversion factor, according to NDS 2018 (American Wood Council, 2018), 2.54 for flexure and 2.

88 for shear, Φ - strength reduction factor, according to Seismic Code of Costa Rica 2010 revision 2014 (CIFA, 2011; INTECO, 2011), which in this case was 0.85 for bending and 0.75 for shear, f_b – bending design values, S– elastic section modulus, f_v – shear design values, A – cross-sectional area, E - MOE determined in Eq. 1, I – moment of inertia and EI – bending stiffness.

Subsequently, the maximum capacity in terms of overload (distributed load per unit area, w) in supported flexure was calculated, varying span length (L) and the separation between beams (*sep*) using Eqs. 9, 10 and 11. From these three calculated values, the lowest one was selected by safety factor, which is the one that determines the design values.

$$v = \frac{8 \cdot \Phi M n}{sep \cdot L^2} \tag{9}$$

$$w = \frac{384 \cdot Y \cdot EI}{5 \cdot sep \cdot L^4} \tag{10}$$

$$w = \frac{2 \cdot \Phi V n}{sep \cdot L} \tag{11}$$

Where: ΦMn – nominal bending strength, *Y* – maximum allowable deflection (*L*/240), *EI* – product of modulus of elasticity and the second moment of area and ΦVn – nominal shear strength.

2.7 Statistical analysis

2.7. Statistička analiza

One-way ANOVA was applied to mechanical parameters in flexure test (load and deflection at proportional limit, maximum load, *MOR* and *MOE*) and maximum stress in shear test - the physical parameters (density, *MC* and *WA*). The Tukey test was used to test the mean difference at a level of significance of p<0.01 per species and each type of beams. The SAS 8.1 statistics program for Windows (SAS Institute Inc., Cary, N.C., USA) was used to carry out the analyses.

3 RESULTS AND DISCUSSION

3. REZULTATI I RASPRAVA

3.1 Density and moisture content

3.1. Gustoća i sadržaj vode

Figure 3 presents the *MC* and density values obtained for the two species by cross section and type of beam. The *MC* was higher in the glued laminated timber beams for both species and cross section of beams, except for the 5 cm \times 10 cm cross section of *D. panamensis* (Figure 3a). Wood density of the solid beams of both species presented the highest values, except for 5 cm \times 10 cm cross section of *D. panamensis*, which showed no differences between the two types of beams. In addition, the beams manufactured with *D. panamensis* timber (solid and laminated) had higher wood density in relation to the beams of *H. alchorneoides* (Figure 3b).

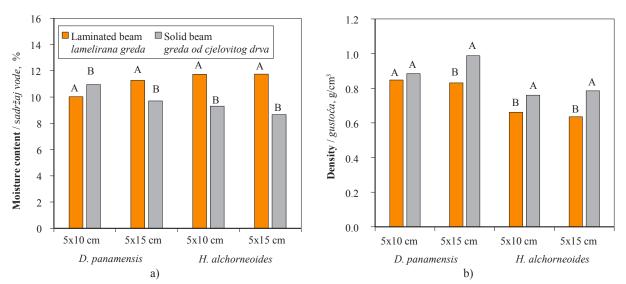


Figure 3 Moisture content (a) and density (b) of laminated and solid beams made of *D. panamensis* and *H. alchorneoides* timber **Slika 3.** Sadržaj vode (a) i gustoća (b) lameliranih greda i greda od cjelovitog drva *D. panamensis* i *H. alchorneoides*

3.2 Bending and shear test

3.2. Ispitivanje na savijanje i smicanje

In the bending test for *D. panamensis*, no statistical differences were observed between laminated timber and solid timber beams in 5 cm \times 10 cm cross section in terms of load at LP and deflection, but statistical differences were observed in terms of maximum load, with the glued laminated timber beams showed the highest values (Table 1). For cross sections of 5 cm \times

15 cm of the same species, it was observed that there were statistical differences between the two types of beams, in the three parameters evaluated, where the solid timber beams showed the highest values (Table 1). In the case of *H. alchorneoides*, for 5 cm \times 10 cm in cross section, differences were observed in load at *LP* and deflection at *LP*, where the solid timber beams showed the highest value; in the case of maximum load, no statistical differences were observed between

Table 1 Statistical values obtained in bending test for glued laminated timber and solid timber beams made of *D. panamensis* and *H. alchorneoides* timber

Tablica 1. Statističke vrijednosti dobivene ispitivanjem na savijanje lameliranih greda i greda od cjelovitog drva *D. panamensis* i *H. alchorneoides*

	Parameter / Parametar		Type of beam / Vrsta grede					
Specie Vrsta			5 cm × 10 cm cro	oss section	5 cm × 15 cm cross section			
			Presjek 5 cm \times 10 cm		Presjek 5 cm \times 15 cm			
			Glued laminated timber Lamelirano drvo	Solid timber Cjelovito drvo	Glued laminated timber Lamelirano drvo	Solid timber Cjelovito drvo		
	Load at PL	Average (kN)	8.38 ^A	9.04 ^A	10.84 ^B	32.43 ^A		
		SD (kN)	2.84	4.17	4.61	14.73		
sis	opterećenje pri PL	<i>CV</i> (%)	33.82	46.16	42.47	45.42		
panamensis	D. C. H. DI	Average (mm)	35.20 ^A	43.73 ^A	20.58 ^B	64.86 ^A		
nan	Deflection at PL <i>deformacija pri PL</i>	SD (mm)	7.55	15.46	7.29	14.40		
pan		<i>CV</i> (%)	30.74	35.36	35.41	22.20		
D.	Maximum load maksimalno opterećenje	Average (kN)	17.81 ^A	12.83 ^B	24.96 ^B	35.69 ^A		
		SD (kN)	4.73	4.99	7.63	13.19		
		<i>CV</i> (%)	26.56	38.91	30.56	36.97		
	Load at PL opterećenje pri PL	Average (kN)	5.47 ^B	12.67 ^A	7.38 ^B	30.08 ^A		
		SD (kN)	2.27	4.16	4.02	6.64		
ides		<i>CV</i> (%)	41.49	32.80	54.49	22.06		
ioəi	Deflection at PL deformacija pri PL	Average (mm)	20.48 ^B	56.90 ^A	17.28 ^B	63.00 ^A		
non		SD (mm)	7.54	11.97	8.18	9.49		
alchorneoides		<i>CV</i> (%)	36.83	21.03	47.36	15.06		
Н. с	Maximum load	Average (kN)	11.03 ^A	14.18 ^A	16.27 ^в	31.95 ^A		
	maksimalno	SD (kN)	4.05	4.57	7.70	7.62		
	opterećenje	<i>CV</i> (%)	36.75	32.24	47.33	23.84		

Note: *PL* – proportional limit / granica proporcionalnosti, SD – standard deviation / standardna devijacija, CV – coefficient of variation / koeficijent varijacije

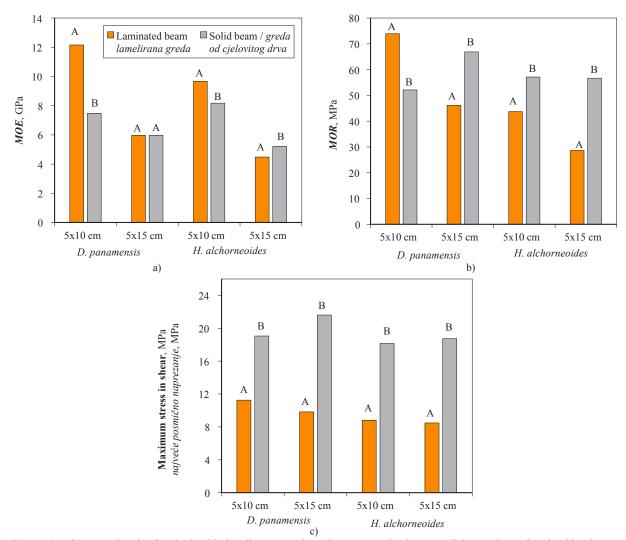


Figure 4 *MOE* (a) and *MOR* (b) obtained in bending test and maximum stress in shear parallel to grain (c) for glued laminated timber and solid timber beams made of *D. panamensis* and *H. alchorneoides* timber **Slika 4.** *MOE* (a) i *MOR* (b) dobiveni pri ispitivanju savijanja i najvećega posmičnog naprezanja u smjeru drvnih vlakanaca (c) lameliranih greda i greda od cjelovitog drva *D. panamensis* i *H. alchorneoides*

the two types of beams (Table 1). For the 5 cm \times 15 cm in cross section, there were differences in the three parameters evaluated, where the solid timber beams showed the highest values in relation to the glued laminated timber beams (Table 1).

Figure 4 presents the *MOE* and *MOR* values obtained for the bending test for the beams of the two species. For *D. panamensis* in cross section of 5 cm × 10 cm, the glued laminated timber beams showed the highest *MOE* value, while for 5 cm × 15 cm in cross section there were no statistical differences in *MOE* (Figure 4a). For *H. alchorneoides*, the glued laminated timber beams showed the highest *MOE* in cross section of 5 cm × 10 cm, while the lowest value of MOE was observed in the cross section of 5 cm × 15 cm (Figure 4a). As for the *MOE*, the solid timber beams showed the highest values for the two species and cross sections of beams, except for the 5 cm × 10 cm cross section of *D. panamensis*, where the glued laminated timber beams showed the highest value (Figure 4b). In the load vs. deflection curves obtained in the bending test, it is observed that for both species at the same deflection value, the 5 cm \times 15 cm cross section beams for solid wood and glulam beams show the highest load value, followed by the 5 cm \times 10 cm cross section beams for glued laminated timber and solid wood beams (Figure 5). In addition, it is observed that the beams made of *D. panamensis* wood show higher load values in relation to the beams made of *H. alchorneoides* for the same deflection (Figure 5). Regarding the maximum stress in the shear test parallel to the grain, it was observed that the solid timber beams showed the highest values for the beams of two species and two cross sections used (Figure 4c).

3.3 Types of failures

3.3. Vrste lomova

Due to the different configurations of the two types of beams, the types of failures were different (Table 2). In the case of the solid timber beams, the type of

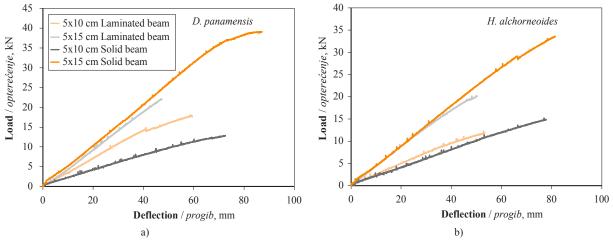


Figure 5 Average load versus deflection plots obtained in bending test for glued laminated timber and solid timber beams made of *D. panamensis* and *H. alchorneoides* wood

Slika 5. Grafički prikazi prosječnog opterećenja s obzirom na progib, dobiveni ispitivanjem na savijanje lameliranih greda i greda od ejelovitog drva *D. panamensis* i *H. alchorneoides*

failure was different by species and cross section (Table 2). Horizontal shear was the most frequent type of failure in the solid timber beams made of *D. panamensis* of 5 cm × 10 cm in cross section, followed by simple failure and cross-grain tension, while in the 5 cm × 15 cm beams, horizontal shear and splintering tension showed the highest percentages. In the solid timber beams made of *H. alchorneoides*, cross-grain and splintering tension failure were the most frequent in the 5×10 cm dimension, while the splintering tension failures were the most frequent in the beams of 5×15 cm in cross section, followed by cross-grain and simple tension failure (Table 2).

Typical failures of solid timber beams are those involving tension (cross-grain, splintering or simple

tension) (Nadir and Nagarajan, 2014), as occurred in two species and two cross sections of beams tested (Table 2). According to Conrad *et al.* (2003), the lack of ductility between fibers in solid timber beams causes the beam to fail in tension, as evidenced by the results of the solid timber beams in the present study.

In the case of glued laminated timber beams, delamination was the most frequent failure in the beams made of *D. panamensis* of two cross sections tested and in cross section of 5 cm \times 15 cm of *H. alchorneoides*, followed by tensile failure in these dimensions. In cross section of 5 cm \times 10 cm of beams made of *H. alchorneoides*, tension failure was the most frequent failure (Table 2). This behavior was different from that found by Nadir and Nagarajan (2014) for

Table 2 Percentage of presence of different types of failure for glued laminated timber and solid timber beams of *D. panamensis* and *H. alchornoides*

Type of		D. panamensis beam Greda od D. panamensis		<i>H. alchorneoides</i> beam Greda od H. alchorneoides		
beam Vrsta grede	Type of failure, % <i>Vrsta loma,</i> %	5 cm×10 cm cross section Presjek 5 cm×10 cm	5 cm×15 cm cross section Presjek 5 cm×15 cm	5 cm×10 cm cross section Presjek 5 cm×10 cm	5 cm×15 cm cross section Presjek 5 cm×15 cm	
	Cross-grain tension napetost u poprečnom presjeku	14.3	20.0	46.7	26.7	
Solid greda od	Horizontal shear horizontalni pomak	57.1	40.0	0.0	6.7	
cjelovitog	Simple tension / jednostavna napetost	28.6	0.0	6.7	26.7	
drva	Brash tension / trzajna napetost	0.0	0.0	6.7	0.0	
	Splintering tension / cijepanje	0.0	40.0	40.0	40.0	
	Delamination / delaminacija	58.3	90.9	0.0	64.3	
Glued laminated	Delamination and shear <i>delaminacija i pomak</i>	0.0	0.0	0.0	7.1	
lame- lirana	Shear / pomak	0.0	9.3	14.3	0.0	
greda	Tension / napetost	41.7	0.0	85.7	14.3	
	Tension and shear / napetost i pomak	0.0	0.0	0.0	14.3	

Tablica 2. Postotak različitih vrsta loma za lamelirane grede i grede od cjelovitog drva D. panamensis i H. alchorneoides

glued laminated timber beams of rubber wood tested in flexure, where tensile was the most frequent type of failure, while delamination was the least frequent. In this regard, these authors explain the different types of failures that occur in glued laminated timber beams tested in flexure and this may explain the results of the present study:

Due to the different configurations of the two types of beams, the types of failures were different (Table 2). In the case of solid timber beams, the type of failure was different by species and cross sections (Table 2). Horizontal shear was the most frequent type of failure in the solid timber beams of *D. panamensis* of 5 cm \times 10 cm cross section, followed by simple failure and cross-grain tension, while in the 5 cm \times 15 cm beams, horizontal shear and splintering tension were the most frequent failures. In the solid timber beams of *H. alchorneoides*, cross-grain and splintering tension failure were the most frequent failures in the 5 cm \times 10 cm dimension and splintering tension failures were the most frequent in the beams of 5 cm \times 15 cm cross sections, followed by cross-grain and simple tension failure (Table 2).

Typical failures of solid timber beams are those involving tension (cross-grain, splintering or simple tension) (Nadir and Nagarajan, 2014), as occurred in two species and two cross sections of beams tested (Table 2). According to Conrad *et al.* (2003), the lack of ductility between fibers in solid timber beams causes the beam to fail in tension, as evidenced by the results of the solid timber beams in the present study.

In the case of glued laminated timber beams, delamination is the most frequent type of failures in the beams of *D. panamensis* of two cross sections tested and in cross section of 5 cm \times 15 cm of *H. alchorneoides*, followed by tensile failure in these dimensions. In the cross section of 5 cm \times 10 cm of *H. alchorneoides*, tension was the most frequent failure (Table 2). This behavior is different from that found by Nadir and Nagarajan (2014) for glued laminated timber beams of rubber wood tested in flexure, where tensile was the most frequent type of failure, while delamination was the least frequent. In this regard, these authors explain the different types of failures that occur in glued laminated timber beams tested in flexure and this may explain the results of the present study:

- i. A delamination failure in glued laminated timber beams can occur when there is a defectively glued area within the lamella, as then the cracks start to propagate through the adhesive surface. However, when a very strong glued area appears, it passes through the wood and another type of failure occurs (Nadir and Nagarajan, 2014). Thus, the results of high occurrence of delamination failure (Table 2) in the beams of *D. panamensis* of two cross sections tested and in *H. alchorneoides* beams of 5 cm × 15 cm cross section indicate that these lamellae had problems of adhesion between the lamellae.
- ii. Tensile failure is the type of failure that is expected to occur and occurs when the joints are perfectly bonded and there are few defects in the timber that could have an adverse effect on the laminates. This results in failures starting from bottom lamina and spreading to the top (Nadir and Nagarajan, 2014). Finally, the *H. alchorneoides* beams of 5 cm \times 10

Species / Vrsta drva	Type of beam / Vrsta grede		$f_{\rm v}$, MPa	$f_{\rm b}$, MPa	MOE, GPa
D. panamensis	$5 \text{ cm} \times 10 \text{ cm}$	Glued laminated timber / lamelirano drvo	0.67	17.61	5.41
		Solid timber / cjelovito drvo	2.58	6.24	4.37
	5 cm × 15 cm	Glued laminated timber / lamelirano drvo	0.29	9.83	4.99
		Solid timber / cjelovito drvo	1.81	6.22	2.14
H. alchorneoides	$5 \text{ cm} \times 10 \text{ cm}$	Glued laminated timber / lamelirano drvo	0.34	6.94	7.27
		Solid timber / cjelovito drvo	2.43	11.07	5.81
	5 cm × 15 cm	Glued laminated timber / lamelirano drvo	0.39	2.16	3.58
		Solid timber / cjelovito drvo	1.80	15.42	4.36

Table 3 Design values for glued laminated timber and solid timber beams made of *D. panamensis* and *H. alchorneoides* wood**Tablica 3.** Projektirane vrijednosti za lamelirane grede i grede od cjelovitog drva *D. panamensis* i *H. alchorneoides*

Table 4 Design values for glulam and solid wood beams made of *D. panamensis* and *H. alchorneoides***Tablica 4.** Projektirane vrijednosti lameliranih greda i greda od cjelovitog drva *D. panamensis* i *H. alchorneoides*

Species / Vrsta drva	Type of beam / Vrsta grede		ΦMn , kN·m	ΦVn, kN	<i>EI</i> , $kN \cdot m^2$
D .	$5 \text{ cm} \times 10 \text{ cm}$	Glued laminated timber / lamelirano drvo	3.17	4.85	22.56
		Solid timber / cjelovito drvo	1.12	18.55	18.22
D. panamensis	5 cm × 15 cm	Glued laminated timber / lamelirano drvo	3.98	3.13	70.16
		Solid timber / cjelovito drvo	2.52	19.57	30.13
H. alchorneoides	$5 \text{ cm} \times 10 \text{ cm}$	Glued laminated timber / lamelirano drvo	1.25	2.46	30.28
		Solid timber / cjelovito drvo	1.99	17.48	24.20
	5 cm × 15 cm	Glued laminated timber / lamelirano drvo	0.87	4.24	50.38
		Solid timber / cjelovito drvo	6.24	19.44	61.29

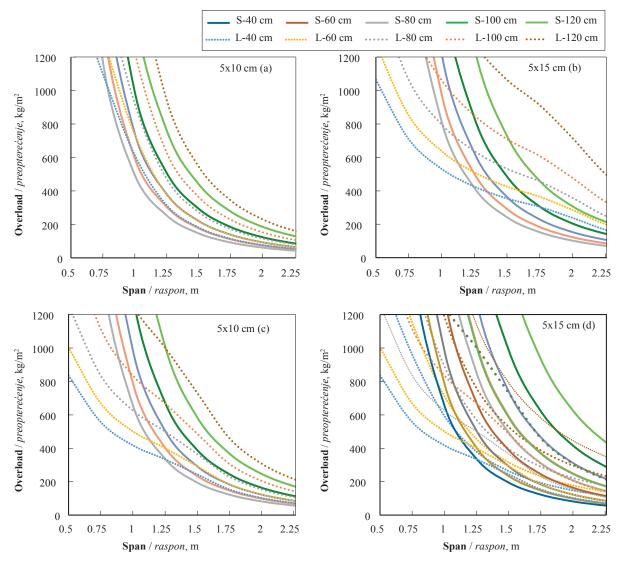


Figure 6 Overload vrs span for glued laminated timber and solid timber beams made of *D. panamensis* (a and b) and *H. alchorneoides* (c and d) wood

Slika 6. Preopterećenje u odnosu prema rasponu za lamelirane grede i grede od cjelovitog drva *D. panamensis* (a i b) te *H. alchorneoides* (c i d)

cm cross section, where tension was the most frequent type of failure (Table 2), was the type of beam made ofout problems of laminate adhesion and therefore only normal failures occurred.

3.4 Derivation of design values

3.4. Deriviranje projektiranih vrijednosti

Table 3 shows the f_v, f_b and *MOE* parameters derived from the bending tests of the two types of beams. These values were used to determine the maximum allowable length for each beam at ΦMn , ΦVn and *EI*, respectively. The results obtained first show that the two types of beams made of *D. panamensis* timber have higher values than the beams made of *H. alchorneoides* timber in some parameters, while some other parameters are lower than those of the beams made of *H. alchorneoides* timber (Table 3 and 4), meaning that both species have similar structural properties. It is also observed that solid timber beams

have higher values of fv than those of glued laminated timber beams (Table 3). However, the fb parameter is higher in glued laminated timber beams than in solid timber beams. In the case of the MOE parameter, there is a tendency of higher values for glued laminated timber beams, except for the 5 cm \times 15 cm cross section.

With respect to the nominal bending strength (ΦMn) , the two species and different cross sections of the beams show different behavior (Table 4). In the beams of *D. panamensis*, the glued laminated timber beams showed higher values than the solid timber beams. However, the behavior of *H. alchorneoides* beams was quite the opposite as the solid timber beams showed higher values of ΦMn . As to the nominal shear strength (ΦVn) , the solid timber beams of the two tested species showed higher values than the glued laminated timber beams. The bending stiffness (*EI*) was higher in the glued laminated timber beams of *D. pana*-

mensis in two cross sections, while in the beams of *H. alchorneoides*, there is no clear trend; the glued laminated timber beams of 5×10 cm cross section showed the highest value of *EI*, while 5 cm \times 15 cm cross section showed the lowest (Table 4).

Figure 6 represents the maximum allowable lengths and overload for the use of beams as floors with spans of 40, 60, 80, 100 and 120 cm for the two species and two cross sections of beams, commonly used in Costa Rica. For example, in solid timber beams of D. panamensis of 5×10 cm cross sections with a spacing of 60 cm and a load of 500 kg/m², it is necessary to use a span of approximately 1.2 m, but if the beam is fabricated in laminated form, the span increases to 1.35 m (Figure 6a). On the other hand, if the cross section of D. panamensis beams is 5×15 cm, the span for solid timber beams is 1.45 m and 1.95 m for glued laminated timber beams (Figure 6b). The same behavior was observed in the beams of H. alchorneoides, with the difference that the span was slightly smaller in the two cross sections of each type of beam (Figure 6c-d).

According to the classification proposed by the Andean Group for the tropical woods of South American countries, Group D can include species with basic specific gravity from 0.56 to 0.70 (Keenan et al., 1987). Thus, the two species studied can be classified in this species group. For the species classified in this group, design values in bending of 15 MPa for fb are presented, which in most cases is higher than the obtained results, so they are categorized in group C, since they present values close to 10 MPa, which is the range proposed by the classification for the Andean Group. This behavior and the presented overload results (Figure 6) show that glued laminated timber beams, as expected, show a better behavior in bending parameters and therefore in design values, resulting in wider span values than those used for solid timber beams (Ndong Bidzo et al., 2021).

4 CONCLUSIONS

4. ZAKLJUČAK

Although problems have been reported for its commercialization due to the lack of products of higher engineering value, Costa Rican plantation timbers such as *D. panamensis* and *H. alchorneoides*, whose characteristic is a high basic specific gravity, are a viable option for commercialization and fabrication of glue laminated timber beams. Such beams can become an engineered product with adequate bending design values when compared to solid timber beams or to design values used for tropical timbers of similar densities. Therefore, it is possible to reach wider spans when the proposed glued laminated timber beams are used as floors.

Other conclusions can also be derived from the present study: (1) the glued laminated timber beams made of *D. panamensis* wood show similar bending parameters as the glued laminated timber beams made of *H. alchorneoides* wood and (2) two cross sections studied for glued laminated timber beams show different values in the bending strength parameters, so for each one it is necessary to establish the design values separately.

Acknowledgements – Zahvala

The authors wish to thank the Vicerrectoría de Investigación y Extensión, Instituto Tecnológico de Costa Rica (ITCR, Cartago, Costa Rica) for the financial support of the project. We would also like to thank the company Cinco Ceibas Rainforest Reserve and Adventure Park for providing material and financial support for this research.

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Shear Force Capacities of H-Type Furniture Joints Constructed of Various Heat-Treated Wood Species

Kapaciteti posmične sile spojeva namještaja H-tipa izrađenoga od različitih toplinski tretiranih vrsta drva

ORIGINAL SCIENTIFIC PAPER

Izvorni znanstveni rad Received – prispjelo: 26. 1. 2022. Accepted – prihvaćeno: 18. 3. 2022. UDK: 684.4.058 https://doi.org/10.5552/drvind.2023.0010 © 2023 by the author(s). Licensee Faculty of Forestry and Wood Technology, University of Zagreb. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license.

ABSTRACT • The aim of this study was to investigate the effect of wood species, heat treatment, adhesive type and joint technique on shear force capacity of H-type furniture joints. For this purpose, an experimental design that consisted of 3 wood species, 2 treatment processes (untreated, heat-treated), 2 adhesive types (polyurethane (PUR), polyvinyl acetate (PVAc)) and 2 joint techniques (dowel, mortise-tenon (MT)) and 5 replications for each group were prepared, and accordingly, a total of 120 specimens were tested under static shear loads. Siberian pine (<u>Pinus sibirica</u>), Iroko (<u>Chlorophora excelsa</u>), and common ash (<u>Fraxinus excelsior</u>), which are commonly used in furniture constructions, were used as wood species. In general, iroko showed the highest shear force capacity between the wood species. The specimens constructed of heat-treated wood species showed lower shear force capacity by approximately 15 % in comparison to the same untreated specimens. MT joints showed better performance than dowel joints higher by approximately 21 %. PVAc adhesive gave higher values than PU adhesive by around 5 %. According to the results of four-way interactions, highest shear force capacities of H-type joints were obtained from "Common ash-PVAc-MT" combination in groups of untreated specimens and from "Iroko-PU-MT" combination in groups of untreated specimens and from "Iroko-PU-MT"

KEYWORDS: heat-treated wood; *H*-type joint; mortise and tenon joint; dowel joint; shear force capacity

SAŽETAK • Cilj ovog istraživanja bio je ispitati utjecaj vrste drva, njegove toplinske obrade, vrste ljepila i tehnike spajanja na kapacitet posmične sile spojeva namještaja H-tipa. Za tu je namjenu pripremljen eksperiment s ovim parametrima: tri vrste drva, dvije obrade (netretirani i toplinski tretiran namještaj), dvije vrste ljepila (poliuretan – PUR, polivinilacetat – PVAc) i dvije tehnike spajanja (moždanikom te čepom i rupom – MT). Za svaki sustav pripremljeno je pet uzoraka te je pri statičkom posmičnom opterećenju ispitano ukupno 120 uzoraka. Odabrane su vrste drva koje se često upotrebljavaju u konstrukcijama namještaja: sibirski bor (<u>Pinus sibirica</u>), iroko (<u>Chlorophora excelsa</u>) i jasen (<u>Fraxinus excelsior</u>). Prema rezultatima istraživanja, drvo iroka pokazalo je najveći kapacitet posmične sile od svih ostalih vrsta drva obuhvaćenih eksperimentom. Uzorci izrađeni od toplinski obrađenog drva imali su oko 15 % manje vrijednosti kapaciteta posmične sile od netretiranih uzoraka. MT spojevi pokazali su za oko 21 % veće vrijednosti od spojeva s moždanicima. Uz upotrebu PVAc ljepila vrijednosti su bile

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za oko 5 % veće od vrijednosti s PUR ljepilom. Prema rezultatima četverosmjernih interakcija, najveći kapaciteti posmične sile spojeva H-tipa dobiveni su za kombinaciju jasenovina – PVAc – MT u netretiranim uzorcima i za kombinaciju drvo iroka – PUR – MT u toplinski tretiranim uzorcima.

KLJUČNE RIJEČI: toplinski obrađeno drvo; spoj H-tipa; spoj čepom i rupom; spoj moždanikom; kapacitet posmične sile

1 INTRODUCTION

1. UVOD

Wood is a natural, sustainable and renewable structural material that has been used by humankind throughout the centuries. However, due to its organic composition, wood can be subjected to several types of degradation factors under adverse climate conditions that could negatively affect physical and mechanical properties of wood. Heat treatment is becoming popular in recent years to decrease the negative effects of those factors.

Heat treatment of wood is a kind of modification method that improves the properties of wood resulting in a new material that is more durable against environmental hazards as compared to the unmodified wood (Hill, 2011). During the heat treatment process, wood is subjected to high temperatures ranging from 160 to 280 °C for several hours in an environment with low oxygen content (Chanrion and Scheiber, 2002; Candelier et al., 2016). As a result of heat treatment process, dimensional stability and biological durability of wood are increased (Korkut et al., 2012; Kasemsiri et al., 2012; Romagnoli, 2015) but some physical and mechanical properties may decrease depending on conditions and intensity of the heat treatment (Viitaniemi 2000; Syrjanen 2001; Candelier et al., 2013; Romagnoli et al., 2015).

Heat-treated wood is an eco-friendly alternative to chemically treated wood and has a wide range of utilization within indoor and especially outdoor furniture applications. Garden furniture, such as tables, chairs, benches, etc., are some of those exposed to and affected by exterior weather conditions. Generally, frame construction technique, which usually consists of three types of joint combinations, namely, L-type (front leg to side rail), T-type (back leg to side rail), and H-type (back/front leg to side rail) joints is used to build this kind of furniture. Mortise and tenon (MT) joints and dowel joints are the most popular joint techniques to connect wooden frame members. The joints should be designed to resist the loads to which they will be exposed during the service life, since the joint strength is the most critical part of the whole frame construction. Typical loading conditions of joints of a simple sitting furniture are shown in Figure 1.

When a sitting furniture frame is exposed to seat loading, the joints shown in circles in Figure 1, which are connected to side, front, back and mid-rails with

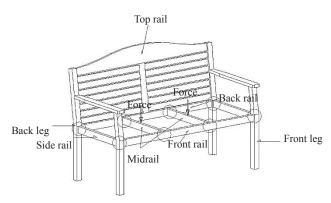


Figure 1 Typical loading conditions on a sitting furniture frame construction **Slika 1.** Tipično opterećenje okvirne konstrukcije

namještaja za sjedenje

back, front legs and rails, are subjected to coercive shear forces. The shear force capacities of the mentioned joints are quite important for the strength of the complete furniture frame.

Many factors, including wood species, adhesive type, tenon size, tenon shape, thickness of glue line, shape of fit, and moisture content (MC), etc., may affect the strength of the joints (Smardzewski, 2002; Dzincic and Skakic, 2012; Dzincic and Zivanic, 2014). Kasal et al. (2013) suggested that joints became stronger and stiffer as either tenon width or length increased. Prekrat and Smardzewski (2010) reported in their study that the shape of the glue line has a definite influence on the strength of the tenon joint. Najafi (2013) studied the withdrawal and shear strengths of dowel joints. Results showed that the withdrawal strength reduced up to 15 % due to moisture content conditions; furthermore, embedment diameter strongly affected withdrawal strength. Kasal et al. (2013) studied ultimate shear force capacity of various dowelled frame type furniture joints. According to the results, they reported that narrower dowel spacing provided greater shear force capacity. They also developed predictive expressions to estimate average shear force capacity of dowel joints. Stress and strain analysis of double-dowel case-type furniture corner joint was made by Hajdarevic and Martinovic (2016). Results of the study indicated that dowel spacing and distance from the edge of board have considerable effect on the stress state of the face and edge member; joints became stiffer when the distance between the dowels and board edge was rationally defined. Diler et al. (2017) studied the effect of wood species, heat treatment, adhesive type and of the study indicated that heat-treated specimens showed lower performance (by 25 %) than untreated specimens and all the factors mentioned above had significant effect on the withdrawal force capacity of T-type joints.

Previous studies show that numerical analysis theory is an alternative method to calculate the strength requirements for MT joints. In this context, an estimation formula was recommended by Erdil *et al.* (2005), which considers the effect of wood species, adhesive type, and joint geometry on the strength of MT joints. Kasal *et al.* (2016) compared the results of empirical tests and numerical analyses for various sizes of mortise and tenon joints. The results of the comparison showed that the numerical analyses gave reasonable estimates of mechanical behaviour of joint strength. Eckelman *et al.* (2017) improved a statistical technique that uses the same data to determine reduction factors and impact of the selection of any given confidenceproportion levels on design values for MT joints.

In addition, recent studies have shown that, with the development of computer technology, finite element method (FEM) has become widespread for the analysis of furniture systems and connections as well as complex structures. By the contribution of many researchers, finite element method (FEM) has been confirmed as an effective method commonly used in wood engineering (Kasal et al., 2016a; Kilic et al., 2018; Hu et al., 2019; Xi et al., 2020; Ceylan et al. 2021). Previous studies also proved that the FEM can be used to analyse some furniture joint techniques such as MT joint (Colakoglu et al., 2012; Smardzewski, 2016; Zhou, 2018; Kilic et al., 2018; Chen, 2019; Zhang, 2021). By use of an advanced finite element software, it is possible to perform accurate simulations of the behaviour of furniture constructions under loading conditions.

It is believed that the use of the heat-treated wood in furniture products will increase by expanding the knowledge regarding its strength properties. Although the physical and mechanical properties of heat-treated wood materials have been investigated in many studies, the information on strength performance of furniture joints made of heat-treated wood is very limited. Thus, it is the aim of this study to investigate the effect of wood species, heat treatment, joint types, and adhesive types on shear force capacities of H-type furniture joints.

2 MATERIALS AND METHODS 2. MATERIJALI I METODE

2.1 Materials

2.1. Materijali

In this study, three wood species, namely, Siberian pine (*Pinus sibirica*), iroko (*Chlorophora excelsa*) and common ash (*Fraxinus excelsior*) were used as wood materials. Since all these wood materials were common-

ly used in the wood products industry, they are potential wood species for industrial scale heat treatment. All heat-treated and untreated wood materials were provided by Novawood Company in Gerede, Turkey.

According to information gathered from, company, heat treatment process was applied according to that described in Finnish ThermoWood Handbook (Finnish Thermowood Association, 2003). The total heat treatment time was 63h, while the time of exposure to the highest temperature was 3h. The heat treatment operation was performed slowly because of the risk of cracks and drying defects. The specimens were prepared from selected defect free materials after heat treatment. Untreated planks of the same species, as control specimens, were dried in industrial drying kilns at approximately 70 °C and 65 % relative humidity (RH), until they reached an equilibrium moisture content. Care was given to select defect-free wood materials for preparing all test specimens. All the prepared specimens were conditioned at (20±2) °C and (65±3) % RH until an equilibrium was achieved before testing.

Moisture content (MC) of control specimens and heat-treated specimens was measured during testing in a range of 6-8 % and 3-5 %, respectively. MC and density (δ) of wood materials were measured according to procedures described in ASTM D 4442-92 (2001) and ASTM D 2395-14 (2015), respectively. In addition, tensile strength and compression strength in parallel to the grain and ultimate bending strength of wood materials were determined according to the test procedures described in ASTM D 143-94 (2000).

Results of some physical and mechanical properties of wood species used in this study are given in Table 1 and Table 2, respectively. According to the results, density of heat-treated wood materials was lower than that of the same untreated species by 7.9 %, 5.2 %, and 6.8% for Siberian pine, iroko, and common ash, respectively. In general, Siberian pine had the lowest density, while common ash had the highest. In addition, the equilibrium MC values of heat-treated wood specimens were lower than those of the untreated specimens.

According to Table 2, all heat-treated wood species yielded lower values of tensile strength as compared to the same untreated specimens. Siberian pine decreased by approximately 16.7 %, iroko by 11 % and common ash by 3 %. On the other hand, results of compression strength were contrary to expectations and common literature (Unsal and Ayrilmis, 2005; Korkut *et al.*, 2008); heat-treated specimens yielded higher values than untreated specimens by about 8 %, 19 %, 10 %, for Siberian pine, iroko, and common ash respectively. This could be explained by differences in moisture content between heat-treated and untreated wood specimens. In bending strength, heat-treated Siberian pine decreased by approximately 20 %, while

Wood species Vrsta drva	Heat treatment Toplinska obrada	Test moisture content (MC), % Sadržaj vode (MC), %		<i>COV</i> *, %	$\begin{array}{c} \textbf{Test } \textit{MC density} \\ (\delta_{\textit{MC}}), \textit{g/cm}^3 \\ \textit{Gustoća pri ispitiva-} \\ \textit{nom sadržaju vode} \\ (\delta_{\textit{MC}}), \textit{g/cm}^3 \end{array}$	<i>COV</i> *, %
Siberian pine	Heat-treated / toplinski tretirana	4.50	0.35	2.12	0.36	2.78
sibirska borovina	Untreated / netretirana	6.77	0.38	4.02	0.40	4.54
Iroko	Heat-treated / toplinski tretirano	3.71	0.54	2.48	0.56	2.28
drvo iroka	Untreated / netretirano	7.54	0.57	3.01	0.61	2.59
Common ash	Heat-treated / toplinski tretirana	4.24	0.55	2.26	0.57	2.24
jasenovina	Untreated / netretirana	7.04	0.59	3.65	0.63	3.86

Table 1 Physical properties of wood materials (Demirci *et al.*, 2016) Tablica 1. Fizička svojstva drvnog materijala (Demirci *et al.*, 2016.)

*COV: Coefficients of variation / koeficijent varijacije

 Table 2 Mechanical properties of wood materials (Demirci *et al.*, 2016)

 Tablica 2. Mehanička svojstva drvnog materijala (Demirci *et al.*, 2016.)

Wood species Vrsta drva	Heat treatment Toplinska obrada	Tensile strength parallel to grain, N/mm ² Čvrstoća na vlak u smjeru drvnih vlakanaca, N/mm ²	<i>COV</i> , %	Compression strength parallel to grain, N/mm ² Čvrstoća na tlak u smjeru drvnih vlakanaca, N/mm ²	<i>COV</i> , %	Bending strength, N/mm ² Čvrstoća na savijanje, N/mm ²	<i>COV</i> , %
Siberian pine sibirska	Heat-treated / toplinski tretirana	36.49	10.05	51.72	9.33	68.01	8.92
sibirska borovina	Untreated / netretirana	43.81	9.27	47.64	3.92	85.31	9.33
Iroko	Heat-treated / toplinski tretirano	51.54	10.07	69.72	8.38	88.29	8.58
drvo iroka	Untreated / netretirano	57.78	5.66	56.70	6.27	87.51	8.21
Common ash	Heat-treated / toplinski tretirana	69.25	9.30	77.73	2.69	137.67	6.18
jasenovina	Untreated/ netretirana	71.21	9.26	69.71	3.68	138.44	7.49

there was no significant difference between heat-treated and untreated specimens of iroko and common ash.

Polyvinyl acetate (PVAc) and polyurethane (PUR) adhesives were used to assemble the test specimens in this study. According to specifications in data sheet of the suppliers, viscosity of PVAc was 160 cps to 200 cps at 25 °C with a density of 1.09 g/cm³, 50 % solids content, liquid form and water resistance, viscosity of PUR was 3300 cps to 4000 cps at 25 °C with a density of 1.11 g/cm³ and one component. The adhesives were applied at (150 \pm 10) g/m² in accordance with suppliers' recommendation.

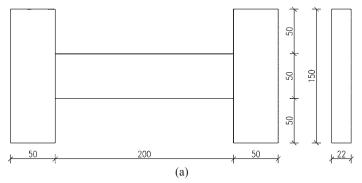
2.2 General configuration and construction of test specimens

2.2. Opća konfiguracija i konstrukcija ispitnih uzoraka

In this study, a total of 120 H-type joint specimens, constructed of 3 different wood species (WS), 2 heat treatments (HT), 2 adhesive types (AT), and 2 joint techniques (JT) with 5 replications for each group, were prepared and tested. Each test specimen was constructed with three structural elements, a post (200 mm \times 50 mm \times 22 mm) and two rail (150 mm \times 50 mm \times 22 mm) members. Technical drawing of the specimen and an assembled specimen are shown in Figure 2a and 2b, respectively.

In the MT joint specimens, mortise and tenon connections were produced with a mortising and tenoning machine. The dimensions of tenons measured were $35 \text{ mm} \times 40 \text{ mm} \times 8 \text{ mm}$ (length×width×thickness), and a snug fit (average mortise-tenon clearance of (0.076±0.025 mm) was obtained between tenons and mortises. Adhesive of (150±10) g/m² was applied approximately to all tenon and mortise faces.

Dowel joints were constructed according to TS 4539 (1985). In the dowel joint specimens, multigroove beech (Fagus orientalis L.) dowels with 8 mm in diameter and 36 mm in length were used. Two dowels were used in each joint with a 26 mm centreline distance. Dowels were embedded at the depth of 20 mm in the rail and 16 mm in the post members. Clearances of dowel-hole were not measured, but all dowels fit snugly into the holes. Adhesive was spread over the sides of the holes and all faces of the dowels. Wax paper was used to prevent adhering between the specimen members. All specimens were assembled manually one by one with a clamp under the pressure specified in the adhesive data sheet. Prior the test, all the test specimens were allowed to cure for minimum one month in an environmentally controlled conditioning room that was set to (65 ± 3) % relative humidity and (20±2) °C. Technical details of the MT and dowel joints are given in Figure 3a and 3b, respectively.





(b)

Figure 2 Test specimen dimensions (a) and real picture (b) (dimensions in mm) **Slika 2.** Dimenzije ispitnog uzorka (a) i stvarna slika uzorka (b) (dimenzije u mm)

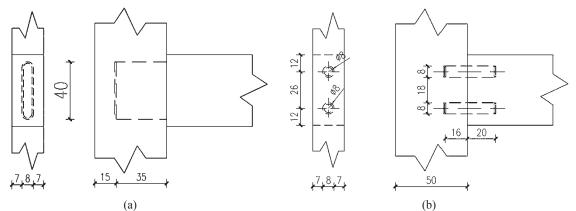


Figure 3 MT (a) and dowel (b) joints details (dimensions in mm) Slika 3. Detalji spoja čepom i rupom (a) i spoja moždanikom (b) (dimenzije u mm)

2.3 Testing method

2.3. Metoda ispitivanja

All the static tests and shear force capacity tests were conducted on a 50-kN capacity screw type universal-testing machine (Mares 2007, Turkey) under 6 mm/min static loading rate in the Physical and Mechanical Tests Laboratory of Wood Science and Industrial Engineering Department of Mugla Sitki Kocman University. Tensile and compression strength parallel to the grain and bending strength of the wood materials were determined according to the test procedures described in ASTM D 143-94 (2000). Shear force capacity tests were conducted based on the methods accepted in previous studies (Ors and Efe 1998; Dizel 2005; Yildirim et al. 2020, Balikci 2015). For the static tests, samples were loaded to ultimate failure; however, for the shear force capacity test samples, loading was continued until separation occurred on the intersecting surfaces. The test set-up used for shear force capacity tests is illustrated in Fig. 4. The ultimate force monitored on H-type joint specimens was recorded as the shear force in Newton (N). The maximum force for H-type joint elements was recorded as shear force capacity and calculated by following equation:

$$F = \frac{F_{\text{max}}}{2} \tag{1}$$

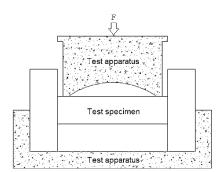


Figure 4 Shear force capacity test set-up Slika 4. Postav za ispitivanje kapaciteta posmične sile

F – Shear force capacity of a joint (N)

 F_{max} – Maximum force (N),

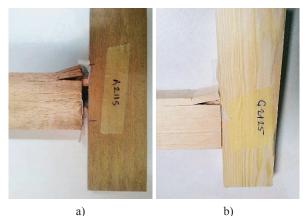
2 – Number of joints.

3 RESULTS AND DISCUSSION 3. REZULTATI I RASPRAVA

3.1 Experimental results for shear force capacity of H-type joints

3.1. Eksperimentalni rezultati kapaciteta posmične sile spojeva H-tipa

In shear force capacity tests, specimens reached ultimate values within 60-90 seconds. Commonly, all untreated specimen combinations failed due to glue



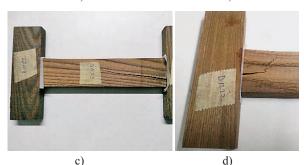


Figure 5 Typical failure modes of untreated (a) and heat-treated (b) specimens (Force directions are all from the top of photographs)

Slika 5. Tipični načini loma netretiranih (a) i toplinski tretiranih (b) uzoraka (sile su usmjerene od vrha fotografija prema dolje)

line fractures and cracking from upper edge of rail members as shown in Figure 5a. However, in the case of heat-treated specimens, generally, joints fractured from glue line and split from the middle or toward upper edge of rail members as shown in Figure 5b.

Table 3 Results of multiple variance analysis Tablica 3. Rezultati analize višestruke varijance

A four-way analysis of variance (MANOVA) general linear model procedure was performed to analyse the main effects and interactions on the shear force capacity. Statistically significant results were further analysed by the least significant difference (LSD) multiple comparisons procedure at 5 % significance level to determine the mean differences of shear force capacity values of H-type joints tested considering the wood species, heat treatment, adhesive type, joint technique, and their interactions. MSTAT-C statistical software (Michigan State University, USA) was used for statistical evaluations. Multiple variance analysis results on the effect of wood species (WS), treatment process (HT), adhesive types (AT) and joint types (JT) on the shear force capacity of H-type joints are given in Table 3.

According to the results given in Table 3, all single and multiple interactions, except for "WS - HT -AT" and "WS - AT - JT", were statistically significant at 5 % significance level. For the main factors and fourway interactions, least significant difference (LSD) multiple comparisons at 5 % significance level were performed to determine mean differences in shear capacity of H-type joints.

Mean comparison results of the effect of wood species on the shear force capacity of the joints are shown in Table 4. The single LSD value was calculated as 270.7 N based on the error mean square of the full model.

According to Table 4, the effect of wood species on the shear force capacity of the H-type joints was statistically significant at 5 % significant level. Iroko yielded the highest shear force capacity. Accordingly, the results of common ash and Siberian pine were lower than

F-value

F-vrijednost

1739

Probability (p<0.05)

Vjerojatnost (p < 0,05)

0.0000

	5		
Variation sources	Degrees of freedom	Sum of squares	Mean square
Izvori varijacija	Stupnjevi slobode	Zbroj kvadrata	Srednji kvadrat
WS	2	63743904.9	31871952.4
HT	1	10887157.118	10887157.118
WS - HT	2	7237462.381	3618731.191
AT	1	1188764.315	1188764.315
WS - AT	2	2880596.895	1440298.448
	1		

0.0000 59.4184 19.7498 0.0000 0.0125 6.4879 0.0007 7.8607 HT - AT 1 4442058.017 4442058.017 24.2433 0.0000 WS - HT - AT 2 347795.884 695591.768 1.8982 NS JT 1 21249549.778 21249549.778 115.9729 0.0000 WS - JT 2 4273179.314 2136589.657 0.0000 11.6608 HT - JT 1 4764828.825 4764828.825 26.0048 0.0000 2 WS - HT - JT 7672005.539 3836002.770 20.9356 0.0000 7789169.147 7789169.147 42.5107 0.0000 AT - JT 1 WS - AT - JT 2 15635.711 7817.855 NS 0.0427 HT - AT - JT 3477986.129 1 3477986.129 18.9817 0.0000 WS - HT - AT - JT 2 1279682.618 639841.309 3.4920 0.0344 Error 96 17589946.561 183228.610 Total 119 159187518.977

NS - Not significant / nije značajno, WS - Wood species / vrsta drva, HT - Heat treatment / toplinska obrada, AT - Adhesive type / vrsta ljepila, JT - Joint technique / vrsta spoja

 Table 4 Mean comparisons of effect of wood species on shear force capacity

Tablica 4. Usporedbe srednjih vrijednosti utjecaja vrste drva na kapacitet posmične sile

Wood species Vrsta drva	Shear force capacity , N <i>Kapacitet posmične sile</i> , N			
	X	HG		
Siberian pine / sibirski bor	2653	С		
Iroko / drvo iroka	4304	A		
Common ash / jasenovina	4067	В		

LSD± 270.7 N, HG - Homogeneity group / homogene grupe

 Table 5 Mean comparisons for heat treatment effect on shear force capacity

Tablica 5. Usporedbe srednjih vrijednosti utjecaja toplinskog tretmana na kapacitet posmične sile

	Shear force		
Heat treatment	Kapacitet posmične sile,		
Toplinska obrada	N		
	X	HG	
Heat-treated / toplinski tretirano	3374	В	
Untreated / netretirano	3976	А	
LSD± 155.1 N			

those of iroko by approximately 5.5 % and 38 %, respectively, whereas the results of common ash were higher than those of Siberian pine by approximately 35 %.

The mean comparison values of the effect of heat treatment on shear force capacity of tested H-type joints are presented in Table 5. The single LSD value was calculated as 155.1 N based on the error mean square of the full model.

The specimens constructed of heat-treated wood species performed lower than the untreated specimens in terms of shear force capacity. The performance of the joints decreased by approximately 15 % as compared to the untreated specimens. This could be due to the fact that heat treatment processing causes the physical changes in the cellular structure of wood. The negative effects of the heat treatment process on the mechanical properties of wood materials have been well researched in the previous studies (Esteves and Pereira, 2009). As a result of thermal process, strength loss associated with thermal degradation and mass loss due to applied temperature may occur (Rusche, 1973; Zaman et al., 2000; Mazela et al., 2003). Mitchel (1998) indicated that irreversible degradation of the mechanical and technological properties of wood are caused by thermal degradation.

The mean comparison values of the effect of adhesives on shear force capacity of tested H-type joints are given in Table 6. The single LSD value was calculated as 155.1 N based on the error mean square of the full model.

In this study, PVAc adhesive performed better than PU adhesive by approximately 5.3 % higher values in terms of the shear force capacity of H-type
 Table 6 Mean comparisons for effect of adhesive type on shear force capacity

Tablica 6. Usporedbe srednjih vrijednosti utjecaja vrste ljepila na kapacitet posmične sile

Adhesive type Vrsta ljepila	Shear force capacity , N <i>Kapacitet posmične sile</i> , N			
vrsia ijepila	X	HG		
PUR	3575	В		
PVAc	3774	А		

LSD± 155.1 N

 Table 7 Mean comparisons for effect of joint technique on shear force capacity

Tablica 7. Usporedbe srednjih vrijednosti utjecaja tehnike

 spajanja na kapacitet posmične sile

Joint technique	Shear force capacity , N <i>Kapacitet posmične sile</i> , N				
Tehnika spajanja	X	HG			
Dowel / moždanik	3254	В			
MT	4096	А			
LSD± 155.1 N					

joints. PU and PVAc are structurally different adhesives. PUR is a thermosetting adhesive that results in a rigid material after curing, while PVAc is a thermoplastic adhesive with more elastic behaviour after curing. It is deduced that these properties of adhesives may affect the results and that PVAc might perform better in terms of mechanical adhesion of these kind of joints.

The mean comparison values for the effect of joint technique on the shear force capacity of tested H-type joints are presented in Table 7. The single LSD value was calculated as 155.1 N based on the error mean square of the full model.

According to Table 7, MT joints performed better than dowel joints by approximately 21 % in terms of shear force capacity. The results suggest that MT joints perform better where shear force capacity is needed. This may be explained by the fact that the bonding surface area of MT joints is larger than that of dowel joints.

The mean comparison values of the effect of four-way interactions on shear force capacity of tested H-type joints are given in Table 8. LSD was calculated as 537.4 N.

According to Table 8, in general, all combinations made of untreated wood species yielded higher values than combinations made of heat treated species except for "Iroko-PUR-MT combination. "Common ash–Untreated–PVAc–MT" combination showed the highest shear force capacity in this study. Among combinations made of heat-treated wood species, the highest performance was obtained with "Iroko-PUR-MT" and "iroko-PVAc-MT" combinations, and there was no significant difference between the two combinations,

 Table 8 Mean comparison results of effect of four-way interactions with coefficient variations on shear force capacity

 Tablica 8. Usporedba rezultata srednjih vrijednosti četverosmjernih interakcija s koeficijentima varijacije na kapacitet posmične sile

Wood species Vrsta drva	Heat treatment Toplinska obrada	Adhesive type Vrsta ljepila	Joint type Vrsta spoja	Shear force Kapacitet sn		<i>COV</i> , %	
visia aiva			vrsiu spoju	Х	HG		
		PUR	Dowel	2826	Н	11.06	
	Heat-treated	FUK	MT	2446	HI	7.88	
	toplinski tretiran	PVAc	Dowel	2051	Ι	6.55	
Siberian pine		IVAC	MT	2510	HI	11.65	
sibirski bor		PUR	Dowel	2832	Н	9.28	
	Untreated	FUK	MT	2880	GH	8.65	
	netretiran	PVAc	Dowel	2273	Ι	13.30	
		r vAC	MT	3407	FG	16.21	
	Heat-treated toplinski tretirano	PUR	Dowel	3763	DEF	13.14	
			MT	4989	С	9.18	
		PVAc	Dowel	3378	FG	10.63	
Iroko			MT	4814	С	7.58	
drvo iroka		PUR	Dowel	3964	DE	13.06	
	Untreated		MT	4080	D	19.89	
	netretirano	PVAc	Dowel	3749	DEF	5.95	
			MT	5695	В	13.32	
		PUR	Dowel	3399	FG	20.53	
	Heat-treated	PUK	MT	3376	FG	8.21	
	toplinski tretirana	DVA -	Dowel	3495	EF	15.15	
Common ash		PVAc	MT	3436	EF	12.35	
jasenovina		PUR	Dowel	3671	DEF	7.51	
	Untreated	PUK	MT	4677	С	9.26	
	netretirana	DVA -	Dowel	3646	DEF	6.83	
		PVAc	MT	6838	А	6.17	

 $\text{LSD}{\pm}~537.4~\text{N}$

while "Siberian pine-HT-PVAc-Dowel" combination showed the lowest values.

There was no significant difference between "Siberian pine-HT-MT" combinations constructed with both PUR and PVAc adhesives. A similar situation occurred when both MT and Dowel joints were constructed with a the combination of "iroko-HT-PUR" and "iroko-HT-PVAc". In addition, when comparing their combinations, no significant difference was found when both MT and Dowel joints were constructed with combinations of "Common ash-HT-PUR" and "Common ash-HT-PVAc".

It is believed that these results could provide economic and technical benefits for furniture designers, engineers, and manufacturers.

4 CONCLUSIONS

4. ZAKLJUČAK

In this study, shear force capacity of H-type joints made of heat-treated Siberian pine, Iroko and Common Ash were investigated. From results of the study, it can be concluded that wood species, heat treatment, adhesive type, and joint type had significant effects on the shear force capacity of H-type joints. In terms of adhesive type, PVAc could be considered as a better alternative, while the MT joint type could be preferred where high strength was necessary.

In terms of wood species, iroko showed higher shear force capacity values than common ash and Siberian pine. Shear force capacity of the specimens made of heat-treated wood was by approximately 15 % lower than that of the untreated specimens. As stated in many previous studies, the present study also confirmed that heat treatment has negative effects on the mechanical properties of wood. In terms of the adhesive type, the joints glued with PVAc had approximately 5 % higher shear force strength than those glued with PUR. In terms of the joint type, MT joints performed approximately 21 % better than dowel joints. For the specimens made of heat-treated wood materials, the best results were obtained from the iroko-PUR-MT and iroko- PVAc- MT combinations, and there was no significant difference between the two combinations.

It is believed that the results of this study will help design more durable furniture frame constructions, especially for sitting furniture, since most of their joints are exposed to shear effect under applied forces. The results of the study clearly indicate that designers/engineers should select the joint type, wood species, and adhesive, when using heat treated materials, in order to reduce the effects of strength reductions caused by heat treatment. In future studies, different wood species and joint types could be evaluated in order to make a wider range of engineering decisions. Such wide range of studies could also provide opportunity to create predictions for optimum strength of joints designed with heat treated materials.

Acknowledgements - Zahvala

This article is a part of MSc. thesis of second author (Balikci, 2015). It may contain some common data and similarities with the papers published by Diler et al. (2017) and Demirci et al. (2016) which are also a part of the same MSc. thesis. This study was supported by Scientific Research Project Office of Mugla Sitki Kocman University with project no 14/023. A part of this article was presented verbally in Proceedings of 28th International Conference Research for Furniture Industry in Poznan/Poland in 2017.

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Boron Compounds-Added Kraft Pulping from Scots Pine

Kraft celuloza od borovine s dodatkom spojeva bora

ORIGINAL SCIENTIFIC PAPER

Izvorni znanstveni rad Received – prispjelo: 29. 1. 2022. Accepted – prihvaćeno: 15. 3. 2022. UDK: 676.15 https://doi.org/10.5552/drvind.2023.0014 © 2023 by the author(s). Licensee Faculty of Forestry and Wood Technology, University of Zagreb. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license.

ABSTRACT • In this study, effects of KBH_4 (PB), $NaBH_4$ (SB), Etibor-48 (E48), Etidot-67 (E67), and colemanite (Col) on kraft pulp and paper properties of Scots pine (<u>Pinus sylvestris</u> L.) wood were evaluated. The control and boron compound-added kraft pulps were obtained under constant cooking conditions. The boron compounds were used as digester additives in different ratios (2 % and 4 %). The addition of boron compounds to kraft cooking liquor resulted in increases screened and total yield of pulps. The highest screened yield (52.05 %) and total yield (55.09 %) were obtained from PB-4 pulp. The lowest reject ratio (0.61 %) and kappa number (34.60) were determined from PB-2 pulp. Furthermore, the highest tensile properties of handsheets were obtained from E67-4 pulp. Also, E48-4 pulp had the highest burst index and tear index values. E48, E67, and Col are cheaper than PB and SB. From these boron compounds, pulps with relatively low pulp yield but stronger can be obtained.

KEYWORDS: *Pinus sylvestris*; pulp yield; boron compounds; pulp strength

SAŽETAK • U ovom je istraživanju proučavan utjecaj KBH₄ (PB), NaBH₄ (SB), Etibor-48 (E48), Etidot-67 (E67) i kolemanita (Col) na kraft celulozu i svojstva papira od borovine (<u>Pinus sylvestris</u> L.). Kontrolna i kraft celuloza s dodatkom spojeva bora dobivene su pri konstantnim uvjetima kuhanja. Spojevi bora upotrijebljeni su kao dodatci za poboljšanje digestije u različitim omjerima (2 i 4 %). Dodavanje spojeva bora tekućini za kuhanje rezultiralo je povećanjem prinosa nakon prosijavanja i ukupnog prinosa celuloze. Najveći prinos nakon prosijavanja (52,05 %) i najveći ukupni prinos (55,09 %) dobiveni su za celulozu PB-4. Najmanji omjer odbacivanja (0,61 %) i kappa broj (34,60) utvrđeni su za celulozu PB-2. Nadalje, najveća vlačna svojstva listova papira dobivena su za celulozu E67-4. Također, celuloza E48-4 imala je najveće vrijednosti indeksa probijanja i vlačnog indeksa. E48, E67 i kolemanit jeftiniji su od PB i SB. Od tih spojeva bora može se dobiti čvršća celuloza, ali s relativno malim prinosom celuloze.

KLJUČNE RIJEČI: Pinus sylvestris; prinos celuloze; spojevi bora; čvrstoća celuloze

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1 INTRODUCTION

1. UVOD

Scots pine is a significant trading tree species that is extensively planted for conservation and industrial activities such as live snow hedges and soil erosion protection (Şevik and Topaçoğlu, 2015). Scots pine forests cover 28 million hectares in Europe (Bußkamp *et al.*, 2020). Scots pine covers 1.41 million hectares of Türkiye forests and has a 6.15 % share of the Turkish forested area (OGM, 2021).

Türkiye has 73 % of the world's boron reserves based on B_2O_3 content. It is also the world's largest producer of boron compounds (57 % of the boron market). Türkiye's annual boron compound production is 1.73 million tons. The main boron minerals in Türkiye are ulexite (NaCaB₅O₉. 8H₂O) colemanite (Ca₂B₆O₁₁. 5H₂O), and tincal (Na,B₄O₇. 10H₂O) (Eti Mine, 2021).

The pulp industry has commercially used chemical or mechanical pulping methods, or a combination of the two, to obtain pulps with desirable properties. Chemical pulp accounts for approximately 75 % of the world's wood pulp production. Among the chemical pulp production methods, kraft pulping is the most widely used method and the strength properties of kraft pulp are higher compared to other methods. This process produces very high-strength papers. However, the process also has some negative aspects such as giving relatively low yields, being capital and energy-intensive, producing relatively low-value by-products, and generating problematic wastes (Das and Houtman, 2004).

In 2020, the world's paper and paperboard production was 401 million metric tons. The global chemical pulp production volume in 2020 was 148 million metric tons (FAO, 2021). Due to the rapid increase in paper consumption and demand, chemical pulp mills are aiming to find new lignocellulosic raw materials for pulp production (Kaur et al., 2018) or to increase pulp yield with various cooking liquor additives. In the chemical pulping process, cooking chemicals (NaOH, Na₂S, etc.) combined with heat break down the lignin. During pulping, carbohydrate losses and thus the pulp yield losses also occur. The oxidation or reduction of carbohydrate reducing end can prevent these losses. For this purpose, SB was extensively studied as cooking liquor additive in the several lignocellulosic biomass such as Pinus radiata (Meller, 1963; Meller and Ritman, 1964), Pinus brutia (Akgul et al., 2007; Copur and Tozluoglu, 2008; Tutus et al., 2012; Saracbasi et al., 2016), Pinus pinaster (İstek and Gonteki, 2009), Pinus pinea (Gümüşkaya et al., 2011), Abies bornmulleriana (Akgul and Temiz, 2006), Picea orientalis (Tutus et al., 2010a; Erişir et al., 2015), Populus tremula (İstek and Ozkan, 2008), Diospyros kaki (Tutuş et al., 2014), Prunus armeniaca (Tutuş et al., 2016), Castanea sativa and Corylus colurna (Aytekin, 2011), Eucalyptus camaldulensis and Eucalyptus grandis (Ayata, 2008), Rhododendron luteum and Rhododendron ponticum (Birinci et al., 2020), Acer saccharum (Letho et al., 2021), Cyperus papyrus (Gabir and Khristov, 1973), Gossypium hirsutum (Tutus et al., 2010b), Brassica napus (Akgül et al., 2018), poppy stems (Tutuş et al., 2011), and wheat straw (Tutus and Alma, 2005; Tutuş and Çiçekler, 2016). PB was also used in a few lignocellulosic biomass types such as Pinus pinaster (Gülsoy et al., 2016), Pinus brutia (Cicekler and Tutuş, 2019), Pinus pinea (Erkan et al., 2020), Robinia pseudoacacia (Çiçekler et al., 2021), and Pteridium aquilinum (Gülsoy and Şimşir, 2018). However, the influences of E48, E67, and Col on pulp and paper properties were investigated only in Pinus nigra and Populus tremula (Kilic Pekgözlü et al., 2017). To the best of our knowledge, there was no published data regarding the influences of boron compounds on pulp and paper properties of Scots pine. To this scope, we evaluated the effects of SB, PB, E-48, E-67, and Col additions at the different ratios (2 % and 4 % ovendried wood) as cooking liquor additives on pulp and paper properties of Scots pine.

2 MATERIALS AND METHODS 2. MATERIJALI I METODE

Scots pine, a commercially important forest tree, was obtained from the Bartin province of Türkiye. The bark of the wood samples was peeled and crumbled in 3.5-1.5-0.5 cm dimensions to be made into pulp. The chips were air-dried to 12 % final humidity and stored in dry conditions. SB and PB were purchased from Merck. E48 (Na₂B₄O₇. 5H₂O), E67 (Na₂B₈O₁₃. 4H₂O), and Col (CaB₃O₄(OH)₃-H₂O) were obtained from Eti Mine (Türkiye). The boron oxide (B₂O₃) contents of E48, E67, and Col were 48 %, 67 %, and 40 %, respectively.

Chemical analysis samples were prepared according to the TAPPI T 257 standard. The holocellulose (Wise and Karl, 1962), lignin (TAPPI T 222), α -cellulose (TAPPI T 203), cold-hot water solubility (TAPPI T 207), ethanol solubility (TAPPI T 204), and 1 % NaOH solubility (TAPPI T 212) of samples were determined according to the relevant methods. On the other hand, Scots pine samples were macerated by the chlorite method (Spearin and Isenberg, 1947). Fiber length (L), fiber width (D), lumen width (d), and cell wall thickness (w) of 100 randomly selected fibers were measured. The flexibility ratio [(d/D) × 100], slenderness ratio (L/D), and Runkel ratio (2w/d) were calculated using the measured fiber sizes.

Kraft pulping conditions were as follows: 25 % sulfidity, 18 % active alkali as Na_2O , 4:1 liquor/wood ratio, 90 min. to cooking temperature, 75 min. at cook-

ing temperature, and 170 °C cooking temperature. The 2 % and 4 % (oven-dried wood) of each boron compound were added to kraft cooking liquor. Abbreviations of 2 % and 4 % KBH₄-added cookings were PB-2 and 4% PB-4. The cookings of other boron compounds (SB, E48, E67, and Col) were carried out with similar abbreviations. The boron compound-free (C) was also done as a control. In each cooking, 700 g of wood chips (oven-dried) were cooked in a laboratory-type rotary digester. After digestion, pulps were washed with tap water and were disintegrated in a pulp mixer. Somerville-type pulp screen (TAPPI T 275) was used in the screening of pulp samples. Pulp samples were then beaten to 25 °SR in a Valley Beater according to TAPPI T 200. Freeness, viscosity, screened yield, and kappa number of pulps were determined according to ISO 5267-1, SCAN CM 15-62, TAPPI T 210, and TAPPI T 236, respectively. 75 g/m² handsheets made by a Rapid-Kothen Sheet Former (ISO 5269-2) were conditioned according to TAPPI T 402. Tensile properties (tensile index, stretch, and TEA) (ISO 1924-3), tear index (TAPPI T 414), burst index (TAPPI T 403), and brightness (TAPPI T 525) of the handsheets were determined according to standard methods. The data of the handsheet properties for all pulp samples were statistically analyzed using analyses of variance (ANO-VAs) and the Duncan test at a 95 % confidence level. In Figure 1-6, the different letter lowercase indicates that the difference in the mean values of properties among the compared groups was statistically significant (P<0.05).

3 RESULTS AND DISCUSSION 3. REZULTATI I RASPRAVA

The chemical composition and fiber morphology of Scots pine are given in Table 1 and Table 2, respectively. These results showed that both the chemical composition and fiber morphology of Scots pine used in this study were similar to previous studies.

The pulp yield of kraft pulping is relatively low. Therefore, some researchers have aimed to increase pulp yield using digester additives such as anthraquinone, polysulfur, NaBH₄. The screened yields of pulps (except for E67-4 pulp) were increased with the addition of boron compounds. The highest screened pulp yield, increasing by 13.97 % (6.38 points from 45.67 % to 52.02 %), was obtained from PB-4 pulp. E67-4 pulp (45.51 %) had a lower screened yield than the control pulp (45.67 %) (Table 3). The screened pulp yields of all boron compounds-added pulps were increased with increasing boron addition ratios (except for E67-4 pulp). However, the holocellulose contents of E67-4, Col-2, and Col-4 pulps were lower than the control pulp (Table 3). These results showed that hemicellulose retention during pulping was significantly increased with PB, SB, and E48 additions (both 2 % and 4 %) to cooking liquor. However, hemicellulose retention was slightly increased in the E67-2 pulping. The total pulp yields of all pulps were also increased with boron additions (Table 3). The highest total pulp yield, increasing by 15.43 % (8.50 points from 46.59 % to 55.09 %), was obtained from PB-4 pulp. These results can be ascribed to the preven-

Chemical compositions, %	Present study	Alkan, 2004	Dönmez, 2010	Tutuș <i>et al.</i> , 2010
Kemijski sastav, %	Ovo istraživanje			
Holocellulose / holoceluloza	66.92±1.15	70.97	65.75	73.67
α -cellulose / α -celuloza	43.53±0.36	-	46.27	-
Klason lignin / Klasonov lignin	25.02±0.16	23.57	27.23	28.57
Ethanol solubility / ekstrakt u etanolu	8.47±0.22	-	4.52	-
Hot water solubility / ekstrakt u vrućoj vodi	2.58±0.04	6.64	8.45	3.82
Cold water solubility / ekstrakt u hladnoj vodi	1.58±0.02	5.74	-	3.42
1 % NaOH solubility / ekstrakt u 1 % NaOH	10.61±0.47	13.83	10.62	16.28

Table 1 Chemical composition of Scots pine Tablica 1. Kemijski sastav borovine

 Table 2 Fiber morphology of Scots pine
 Tablica 2. Morfologija vlakana borovine

Fiber properties	Present study	Alkan, 2004	Dönmez, 2010	Gulsoy et al.,
Svojstva vlakana	Ovo istraživanje			2013
Fiber length mm / duljina vlakana, mm	2.89±0.70	4.01	3.47	3.00
Fiber width, µm / širina vlakana, µm	38.05±7.08	49.00	44.10	46.30
Lumen width, µm / širina lumena, µm	19.86±6.65	34.80	26.61	23.40
Cell wall thickness, µm / debljina stanične stijenke, µm	9.14±2.84	7.10	8.74	11.45
Slenderness ratio / omjer vitkosti	75.95	81.91	78.70	64.79
Flexibility ratio / omjer fleksibilnosti	52.19	71.02	60.34	50.54
Runkel ratio / Runkelov omjer	0.92	0.40	0.66	0.98

tion of degradation reactions by boron compounds during pulping. Kilic Pekgözlü et al. (2017) noted that screened pulp yield of *Populus tremula* kraft pulp was decreased with the addition of Col. The authors also reported that E48 and E67 caused screened yield increases. The positive effect of SB on pulp yield was reported in previous studies (Ayata, 2008; İstek and Gönteki, 2009; Gümüşkaya et al., 2011; Gulsoy and Eroglu, 2011; Tutuș et al., 2012; Tutuș et al., 2014; Erișir et al., 2015; Tutuş and Çiçekler, 2016; Akgül et al., 2018; Birinci et al., 2020; Letho et al., 2021) and PB (Gülsoy et al., 2016; Gülsoy and Şimşir, 2018; Çiçekler and Tutuş, 2019; Erkan et al., 2020; Çiçekler et al., 2021). Increases in pulp yield provide a significant economic contribution to the pulp mill. The addition of boron compounds also enables more effective use of forests (Gulsoy and Eroglu, 2011).

The reject ratios of pulps provide information about cooking liquor penetration into chips and pulping efficiency. The boron compounds added to pulps had a higher reject ratio (except for PB2 pulp). The reject ratios of pulps increased with the increasing addition ratio of boron compounds. The lowest and highest reject ratios were observed for SB-2 pulp (0.73 %) and PB-4 pulp (3.04 %), respectively (Table 3). E48, E67, and Col had a negative effect on the kraft pulps of Populus tremula and Pinus nigra (Kilic Pekgözlü et al., 2017). The reject ratio increases in the SB-added pulps were observed in the Pinus nigra (Gulsoy and Eroglu, 2011) and Pteridium aquilinum (Gülsoy and Şimşir, 2018) kraft pulping. On the contrary, some authors noted that SB addition caused reject ratio decreases (Ayata, 2008; Akgül and Temiz, 2006; Copur and Tozluoglu, 2008; Tutus et al., 2010a; Tutuş and Çiçekler, 2016). On the other hand, PB addition results in reject ratio decreases (Çiçekler and Tutuş, 2019; Erkan et al,

2020). On the contrary, PB caused the increase in the reject ratio of pulps (Gülsoy *et al.*, 2016; Gülsoy and Şimşir, 2018; Çiçekler *et al.*, 2021).

The kappa number of pulp is related to the effectiveness of pulping and the delignification degree of pulp. The boron compounds-added pulps had a higher kappa number (except for PB-2 and E48-2 pulps) than that of the control pulp. There was a linear relationship between the kappa number of pulps and the addition ratio of boron compounds. The lowest and highest kappa numbers were found in PB-2 (34.60) and Col-4 (46.60), respectively (Table 3). Kilic Pekgözlü et al. (2017) noted that the kappa number of Populus tremula and Pinus nigra kraft pulp was decreased with the addition of E48 and increased with the addition of E67. The authors also noted that the effect of Col addition on the kappa number of Populus tremula and Pinus nigra kraft pulps changed depending on tree species. SB addition result in increasing delignification (Copur and Tozluoglu, 2008; Ayata, 2008; Gulsoy and Eroglu, 2011; Aytekin, 2011; Birinci et al., 2020) and PB addition (Gülsoy and Simsir, 2018; Cicekler and Tutus, 2019; Erkan et al., 2020). On the contrary, PB had an unfavorable effect on the kappa number of Pinus pinaster kraft pulp (Gülsoy et al., 2016). In addition, the kappa number of alkaline sulfite anthraquinone pulps of Pinus pinea (Gümüşkaya et al., 2011) and Picea orientalis (Erişir et al., 2015) decreased with SB additions.

The pulp viscosity indicates the degree of polymerization of polysaccharides. The addition of boron compounds resulted in pulp viscosity decreases. The increase in the addition ratios of boron compounds caused pulp viscosity increase. The lowest pulp viscosity was found in SB-2 pulp with 818.36 cm³/g (Table 3). The cause of pulp viscosity decreases can be as-

 Table 3 Properties of control and boron compounds-added pulps

 Tablica 3. Svojstva kontrolnih uzoraka i celuloze s dodatkom spojeva bora

Tablea 5. Svojstva kolutolnih uzoraka i četulože s dodatkom spojeva bora							
Cookings Uzorci nakon kuhanja	Screened yield (A), % Prinos nakon prosijavanja (A), %	Reject, % Odbačeno, %	Total yield, % Ukupan prinos, %	Kappa number (B) Kappa broj (B)	Lignin in pulp (C: B*0.15), % Lignin u pulpi (C: B*0,15), %	Holocellulose in pulp (A-C), % Hemiceluloza u pulpi (A-C), %	Viscosity, cm ³ /g Viskoznost, cm ³ /g
Control Kontrolni uzorci	45.67	0.92	46.59	35.07	5.26	40.41	980.64
PB-2	48.91	0.61	49.52	34.60	5.19	43.72	818.36
PB-4	52.05	3.04	55.09	46.07	6.91	45.14	931.00
SB-2	48.94	0.73	49.67	35.93	5.39	43.55	865.82
SB-4	48.93	1.37	50.30	37.50	5.63	43.31	957.39
E48-2	46.21	1.52	47.73	34.50	5.18	41.04	875.51
E48-4	46.63	2.09	48.72	35.47	5.32	41.31	904.79
E67-2	46.22	1.96	48.18	37.90	5.69	40.54	929.68
E67-4	45.51	2.58	48.09	41.60	6.24	39.27	948.71
Col-2	45.68	1.55	47.23	38.27	5.74	39.94	969.85
Col-4	46.31	2.81	49.12	46.60	6.99	39.32	975.26

cribed to higher hemicellulose retention in boron compounds-added pulps. Kilic Pekgözlü *et al.* (2017) noted that viscosity of *Populus tremula* kraft pulp was increased with the addition of E48, E67, and Col. The effect of PB on kappa number of *Pinus brutia* kraft pulp was statistically insignificant (Çiçekler and Tutuş, 2019). The authors noted that SB caused pulp viscosity decreases (Ayata, 2008; Copur and Tozluoglu, 2008; Tutus *et al.*, 2010a). On the contrary, the others reported that SB additions (Akgül and Temiz, 2006; Tutus *et al.*, 2010b; Birinci *et al.*, 2020) and PB additions (Gülsoy and Şimşir, 2018; Erkan *et al.*, 2020; Çiçekler *et al.*, 2021) resulted in pulp viscosity increases.

The addition of all boron compounds caused tensile index increases (Figure 1). The highest tensile index increase of 26.27 % (from 68.02 Nm/g to 85.88 Nm/g) was determined in the E67-2 pulp. Otherwise, tensile index losses were observed in the PB-2 pulp. However, this strength loss was statistically insignificant (P>0.05). Furthermore, increasing the addition ratios of boron compounds (except for PB-4 pulp) caused tensile index increases. This result can be ascribed to an increase in the hemicellulose content of the boron compounds-added pulps. The hemicellulose with high hydrophilic properties causes the increase of strength properties of pulp (Kilic Pekgözlü et al., 2017). The fibers with higher hemicellulose content swell more easily and have high flexibility (Shin and Stromberg, 2007). Flexible fibers provide more contact areas with adjacent fibers, which leads to strong interfiber bonds (Forsström et al., 2005) and a high tensile index (Santos et al., 2008). Kilic Pekgözlü et al. (2017) reported that E48, E67, and Col caused a decrease in the tensile index of Pinus nigra kraft pulp. The authors also noted that the tensile index of E48, E67, and Col added pulps of Populus tremula was found to be higher than that of the control pulp. The losses in the tensile properties with SB addition (Akgül and Temiz, 2006; Copur and Tozluoglu, 2008; Gulsoy and Eroglu, 2011) and PB addition (Gülsoy *et al.*, 2016) were reported in the literature. Conversely, some authors noted that SB (Ayata, 2008; Gülsoy and Şimşir, 2018) and PB (Gülsoy and Şimşir, 2018; Çiçekler and Tutuş, 2019) caused increases in the tensile properties of pulps.

The boron compounds-added pulps had a higher stretch ratio than that of the control pulp (Figure 2) (*P*<0.05). The highest stretch increase was obtained from E67-4 pulp with 21.78 % (from 2.02 % to 2.46 %). Kilic Pekgözlü *et al.* (2017) noted that E48 and E67 had a positive effect on the stretch ratio of *Pinus nigra* and *Populus tremula* kraft pulps. They also reported that Col addition caused the increase of the stretch ratio of *Populus tremula*, while it led to the decrease of the stretch ratio of *Pinus nigra*. Gülsoy and Şimşir (2018) noted that PB and SB had a positive effect on the stretch ratio of *Pteridium aquilinum* kraft pulps. Istek and Özkan (2008) stated that the stretch ratio of *Populus tremula* kraft pulp decreased with SB addition.

The addition of all boron compounds caused TEA increases. E67-4 pulp had the highest TEA increase with 36.69 % (from 77.24 J/m² to 105.58 J/m²). Otherwise, TEA loss observed in the PB-2 pulp was statistically significant (P<0.05). Besides, increasing the addition ratios of E48, E67, and Col led to TEA increases (Figure 3). Kilic Pekgözlü *et al.* (2017) noted that TEA of *Populus tremula* handsheets increased with the addition of E48, E67, and Col (except for 8 % Col), while TEA of *Pinus nigra* handsheets decreased. Gülsoy and Şimşir (2018) noted that SB had a positive effect on the TEA of *Pierid-ium aquilinum* kraft pulp. The effect of SB on the TEA of *Populus tremula* kraft pulp was statistically insignificant (İstek and Özkan, 2008).

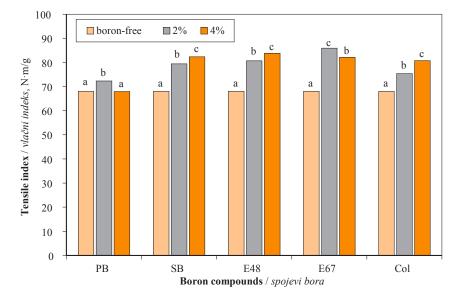


Figure 1 Effects of boron compounds on tensile index of pulps Slika 1. Utjecaj spojeva bora na vlačni indeks celuloze

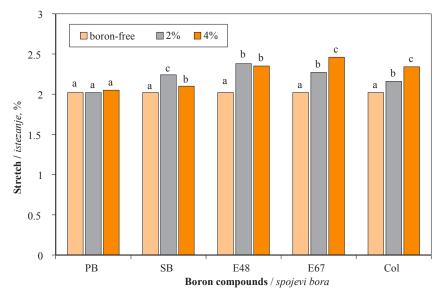


Figure 2 Effects of boron compounds on stretch of pulps **Slika 2.** Utjecaj spojeva bora na istezanje celuloze

The addition of all boron compounds resulted in the burst index increases (P < 0.05). The highest burst index increase was obtained from E48-4 pulp with 33.02 % (from 3.18 kPa·m²/g to 4.23 kPa·m²/g). Besides, the increasing addition ratios of SB and PB caused the burst index losses, while the increasing addition ratios of E48 led to the burst index increase. The effect of increasing addition ratios of E67 and Col on the burst index was statistically insignificant (P>0.05). The burst index increases can be ascribed to more hemicellulose retention in the course of pulping (Rydholm, 1965). Kilic Pekgözlü et al. (2017) stated that E48, E67, and Col had an unfavorable effect on the burst index of Pinus nigra, while they had a positive effect on the burst index of Populus tremula. The burst index losses with SB addition were determined in kraft pulp of Abies bornmulleriana (Akgül and Temiz, 2006), Pinus brutia (Copur and Tozluoglu, 2008), Pinus nigra (Gulsoy and Eroglu, 2011), poppy stems (Tutuş et al., 2011), and Brassica napus (Akgül et al., 2018). Conversely, some authors noted that SB addition caused the burst index increases in Eucalyptus grandis and Eucalyptus camaldulensis (Ayata, 2008), Picea orientalis (Tutus et al., 2010a), Castanea sativa (Aytekin, 2011), Diospyros kaki (Tutuş et al., 2014), Prunus armeniaca (Tutus et al., 2016), and Rhododendron luteum and Rhododendron ponticum (Birinci et al., 2020) kraft pulps. PB had a positive effect on the burst index of Pinus brutia (Çiçekler and Tutuş, 2019) and Pinus pinea (Erkan et al., 2020). Conversely, PB addition led to burst index loss in the kraft pulp of Pinus pinaster (Gülsoy et al., 2016).

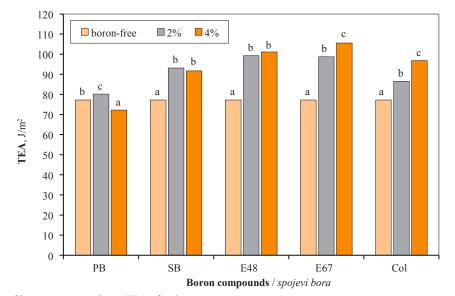


Figure 3 Effects of boron compounds on TEA of pulps **Slika 3.** Utjecaj spojeva bora na TEA celuloze

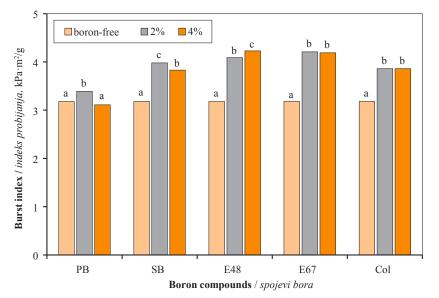


Figure 4 Effects of boron compounds on burst index of pulps **Slika 4.** Utjecaj spojeva bora na indeks probijanja celuloze

All boron compounds caused the tear index increase (P < 0.05). The highest tear index increase was obtained from E48-4 pulp with 37.55 % (from 5.30 $mN \cdot m^2/g$ to 7.29 $mN \cdot m^2/g$). The increasing addition ratios of PB and E67 caused the tear index losses, while the effect of increasing addition ratios of SB, E48, and Col on the tear index was statistically insignificant (P>0.05). E48, E67, and Col had a positive effect on the tear index of Pinus nigra and Populus tremula pulps (Kilic Pekgözlü et al., 2017). The negative effect of PB on the tear index was reported by several authors (Gülsoy and Şimşir, 2018) and SB (Copur and Tozluoglu, 2008; Tutuş et al., 2011; Gulsoy and Eroglu, 2011; Akgül et al., 2018; Gülsoy and Şimşir, 2018). Tutuş and Cicekler (2016) noted that, when 0.5 % NaBH, was added to the soda-oxygen pulping of wheat straw,

the tear index values of the handsheets increased from $3.08 \text{ mN} \cdot \text{m}^2/\text{g}$ to $4.55 \text{ mN} \cdot \text{m}^2/\text{g}$.

The addition of all boron compounds (except for E67 pulp) caused the pulp brightness increases (P<0.05). The lowest and highest brightness values were determined in E67-4 pulp with 18.00 % and SB-2 pulp with 21.35 %, respectively. The brightness of E67 pulp decreased in direct proportion to the increase of the E67 addition ratio. 2 % boron compounds addition caused the increase in handsheet brightness, while 4 % boron compounds addition led to the decrease in handsheet brightness. Kilic Pekgözlü *et al.* (2017) noted that E48 and Col had a positive effect on the handsheet brightness of *Pinus nigra*. On the contrary, E67 had a negative effect on the handsheet brightness of *Pinus nigra*. The authors also reported that the handsheet

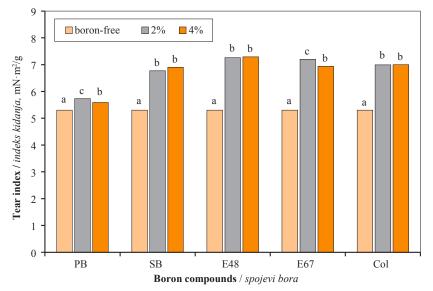


Figure 5 Effects of boron compounds on tear index of pulps **Slika 5.** Utjecaj spojeva bora na indeks kidanja celuloze

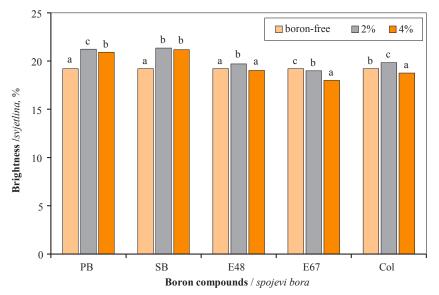


Figure 6 Effects of boron compounds on brightness of pulps **Slika 6.** Utjecaj spojeva bora na svjetlinu celuloze

brightness of *Populus tremula* decreased with the addition of E48, E67, and Col. The brightness increases were reported by several authors in SB-added pulps (Akgül *et al.*, 2007; Gulsoy and Eroglu, 2011; Tutuş *et al.*, 2011, Tutuş *et al.*, 2012; Tutuş *et al.*, 2014, Tutuş and Çiçekler, 2016) and in PB-added pulps (Gülsoy *et al.*, 2016; Gülsoy and Şimşir, 2018; Çiçekler and Tutuş, 2019). Conversely, Gümüşkaya *et al.* (2011) stated that SB had a negative effect on the brightness of alkali sulfite anthraquinone pulp of *Pinus pinea*.

4 CONCLUSIONS

4. ZAKLJUČAK

The results showed that boron compounds-added kraft pulping of Scots pine confers various advantages. The addition of boron compounds to kraft cooking liquor caused increases in screened and total yield of pulps. PB-4 pulp had the highest screened yield and total yield. The lowest reject ratio and kappa number were determined from PB-2 pulp. Besides, E67-4 pulp had the highest tensile properties of handsheets. In addition, the highest burst index and tear index values were obtained from E48-4 pulp. E48, E67, and Col are cheaper than PB and SB. The addition of these boron compounds resulted in relatively lower pulp yield than those of PB and SB pulps. However, they caused stronger pulps. E48, E67, and Col are cheaper than SB and PB. The effects of these boron compounds on the black liquor recovery process should be determined. The effects of E48, E67, and Col on other tree species, that are widely spread and important for the paper industry, should also be investigated.

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Utilization of Lignin Monomer Isolation from Black Liquor of Empty Palm Oil Bunches as a Prebiotic

Upotreba izoliranog monomera lignina iz crnog luga dobivenoga od praznih grozdova palmina ploda kao prebiotika

ORIGINAL SCIENTIFIC PAPER

Izvorni znanstveni rad Received – prispjelo: 17. 2. 2022. Accepted – prihvaćeno: 20. 4. 2022. UDK: 630*86; 665.947.4 https://doi.org/10.5552/drvind.2023.0015 © 2023 by the author(s). Licensee Faculty of Forestry and Wood Technology, University of Zagreb. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license.

ABSTRACT • Waste is generated in the form of black liquor during pulp processing. Furthermore, lignin, the main ingredient of black liquor, contains a phenylpropanoid compound with prebiotic and antimicrobial activity. Also, it is a component of lignocellulose that has prebiotic and antimicrobial activity effects due to its indigestible nature and it comprises phenylpropanoid components. Therefore, this study examines and identifies the lignin purification and test results of its prebiotic activity. The technique used to identify lignin fraction is called Thin Layer Chromatography (TLC) and Gas Chromatography-Mass Spectrometry (GC-MS) plates. Prebiotic activity tests were performed using the calculation of total bacteria on the growth of <u>Lactobacillus casei</u>. The results showed that the purification process using CHCl3, 3 % MeOH:CHCl3, 20 % MeOH:CHCl3, and MeOH yielded 10.68 %, 6.34 %, 11.38 % 44.85 %, respectively. The 3 % MeOH:CHCl3 fraction contained benzaldehyde, 4- hydroxy-3,5-dimethoxy, 1-methylbutyl hexadecanoate, oleic acid, and di-2-ethylhexyl phthalate. The 3 % MeOH:CHCl3 fraction to f 15 % also showed a prebiotic activity for <u>L. casei</u> at 6.1 x 102 colonies/mL.

KEYWORDS: black liquor; lignin monomer; empty palm oil bunch; prebiotic

SAŽETAK • Tijekom prerade pulpe nastaje otpad u obliku crnog luga. Lignin kao glavni sastojak crnog luga sadržava spoj fenilpropanoid prebiotičkoga i antimikrobnog djelovanja. Također, on je i dio lignoceluloze koja zbog svoje neprobavljivosti ima prebiotičko i antimikrobno djelovanje, a sastoji se od fenilpropanoidnih komponenata. Stoga se u ovom istraživanju ispituju i razmatraju pročišćivanje lignina i rezultati prebiotičke aktivnosti. Za identifikaciju frakcija lignina primijenjene su tankoslojna kromatografija (TLC) i plinska kromatografija s masenom spektrometrijom (GC-MS). Ispitivanje prebiotičke aktivnosti provedeno je korištenjem izračuna ukupnog broja bakterija na rast <u>Lactobacillus casei</u>. Rezultati su pokazali da je proces pročišćivanja uz pomoć CHCl_s, 3 % MeOH:CHCl_s, 20 % MeOH:CHCl_s, i MeOH dao prinos od 10,68 %, 6,34 %, 11,38 % i 44,85 %. Frakcija 3 %

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 $MeOH:CHCl_3$ sadržavala je benzaldehid, 4- hidroksi-3,5-dimetoksi, 1-metilbutil heksadekanoat, oleinsku kiselinu i di-2-etilheksil ftalat. Frakcija 3 % $MeOH:CHCl_3$ koncentracije 15 % također je pokazala prebiotičku aktivnost za <u>L. casei</u> pri 6,1 × 10² kolonija/mL.

KLJUČNE RIJEČI: crni lug; monomer lignina; prazni grozdovi palmina ploda; prebiotik

1 INTRODUCTION

1. UVOD

During pulp processing, liquid waste is generated in black liquor, which has lignin content with a complex structure that is difficult to decompose; thus, it can pollute the environment (Lara et al., 2003). However, the lignin content is approximately 25-35 % of the total black liquor (Lara et al., 2003; Goujon et al., 2003). This implies that it can be isolated for further use. Currently, people want to use black liquor as a source of raw material for lignin (Min et al., 2013), which is further used as biomass (Schorr et al., 2014), adhesives (Ramires et al., 2010; Uraki and Koda, 2015), dispersants, surfactants, antioxidants in plastics and rubbers, dyes, synthetic floors, thermosets, paints, and fuel for road maintenance (Laurichesse and Averous, 2013; Mankar et al., 2012; Magnus and Hakan, 2014; Padkościelna et al., 2017; Baurhoo et al., 2008).

Hidayati et al. (2018) isolate lignin from black liquor of empty palm oil bunches by using NaOH at a concentration of 30 % yielded of 5.67 %, with a pH of 5.42, total solid content of black liquor 65.11 %, methyl lignin level of 14.61 %, and equivalent weight 1787.23. Prayuwidayati et al. (2016) showed that lignin is used as a prebiotic, which is considered to have an antimicrobial effect by reducing the growth rate of pathogenic bacteria in the cattle rumen. According to Baurhoo et al. (2008), the tested, purified lignin product has a prebiotic effect. Furthermore, prebiotic is a beneficial component of human food because it enhances the growth and activity of several bacteria in the colon, thus improving the health of the human digestive tract (Toma and Pokrotnieks, 2006). This study aims to identify the components contained in lignin purification products by TLC, Chromatography Column, and GC-MS as testing the prebiotic activity.

2 MATERIALS AND METHODS 2. MATERIJALI I METODE

2.1 Tools and materials

2.1. Oprema i materijali

The materials used were the Empty Palm Oil Bunches (EPOB) black liquor resulting from formacell pulping, acetic acid, formic acid, HCl, $CuSO_4$, H_2O_2 , NaOH, pyridine, MeOH, $CHCl_3$, and equates, as well as NA (Nutrient Agar), NB (Nutrient Broth), media de Man, Rogosa and Sharpe Agar (MRSA), and media de Man, Rogosa and Sharpe Broth (MRSB). Black liquor was obtained from the formacell pulping process using the method used by Hidayati et al. (2017). The advantages of formacell pulping compared to other pulping methods are that it is more environmentally friendly, the pulp yield is high, the pulp produced is sulfur free, and it has high brightness and good strength. The pulping process was carried out in a 1.3 liter autoclave with 20 g of EPOB fiber and liquid for a sample ratio of 15:1 flat bottom, and a wide-mouth boiling flask equipped with a condenser. The solvent ratio of acetic acid: formic acid (85:15) and HCl catalyst was given 0.5 % for 1 hour of cooking time at 130 °C. After cooking, the remaining boiled liquid was separated and collected by filtering. The microbial cultures used were L. casei. Furthermore, the tools used were oven, Buchner funnel, silica gel 60 F₂₅₄ TLC plate, column chromatography, chamber, autoclave, micropipette, incubator, calipers, capillary pipette, GCMS (Varian/CP- 3800 GC and Saturn 2200 MS), and supporting glasses.

2.2 Study method

2.2. Ispitne metode

The study was conducted with a Completely Randomized Design (CRD) in which black liquor was extracted until lignin was obtained twice. Subsequently, lignin was fractionated until a monomer fraction was obtained. TLC analysis identified each lignin monomer fraction, and the prebiotic activity was screened for *L. casei*. The fraction with the highest prebiotic activity was analyzed using GC-MS to determine the chemical composition. Furthermore, they were generated at several concentrations of 0 %, 2.5 %, 5 %, 7.5 %, 10 %, 12.5 %, and 15 %, and they were to be tested for antimicrobial and prebiotic activity for three replications. The data obtained were analyzed descriptively and presented in figures and tables.

2.3 Study implementation 2.3. Provedba ispitivanja 2.3.1 Lignin degradation 2.3.1. Degradacija lignina

The lignin obtained from black liquor due to the pulp cooking with the raw material of EPOB was precipitated and degraded using $CuSO_4$, pyridine, and H_2O_2 . A total of 2 g of lignin precipitate was dissolved with 20 mL NaOH solution to a pH of 12. Afterward, a total of 25 mL of 10^{-2} M $CuSO_4$ and 5 mL pyridine were added to the lignin solution, and then it was stirred with a magnetic stirrer for 30 minutes. 10 ml of

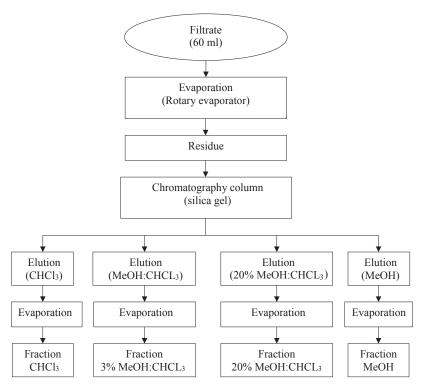


Figure 1 Lignin monomer fractionation flow diagram **Slika 1.** Dijagram toka frakcioniranja monomera lignina

 H_2O_2 1 M was also added five times for 30 minutes, stirred, and stored in an unlighted room for 72 hours.

2.3.2 Lignin purification faction 2.3.2. Pročišćivanje frakcija lignina

The fractionated filtrate was evaporated until a residue was obtained and dissolved in 500 mL H_2O . After that, extraction was performed with EtOAc of 500 mL for three times to obtain H_2O and EtOAc layers that were evaporated until a residue was obtained. The residue was then put into the chromatographic column silica gel and eluted with CHCl₃ (1L) solution. Furthermore, the CHCl₃ fraction was then evaporated, and the obtained residue was purified by preparative thin-layer chromatography (PTLC). The lignin monomer isolation process can be seen in Figure 1.

2.3.3 Lignin fraction identification2.3.3. Identifikacija frakcija lignina

Identification of lignin monomers obtained was made using TLC and GC-MS plates. Subsequently, the isolates were dissolved in hexane and injected into GC-MS devices. The analysis was performed using GC-MS CP-3800 variant with MS Saturn 2200 detector, and VF-5 ms column 30 mm \times 0.25 mm by manual injection method at 240 °C for 40 minutes (Suroso *et al.*, 2018).

2.4 Prebiotic activity test for lignin fraction

2.4. Ispitivanje prebiotičke aktivnosti frakcija lignina

Prebiotic tests were performed microbiologically using *L. casei* grown on MRSA media, and the meas-

ured variable was the number of bacteria counted using the living bacterial colonies method. The lignin monomer fraction was composed of media such that each cup was 0 %, 2.5 %, 5 %, 7.5 %, 10 %, 12.5 %, and 15 %. *L. casei* that had been refreshed on MRSB media was diluted as much as 0.05 mL into 4.95 mL of physiological solution. Then, 1 mL of physiological solution was inoculated into MRSA media, and it was poured into 20 petri cups of 19.5, 19, 18.5, 18, 17.5, and 17 mL and 3 % MeOH:CHCl₃ fraction of 0, 0.5, 1, 1,5, 2, 2.5, and 3 mL were added. Furthermore, the bacteria were incubated for 24 hours in an incubator. The number of bacteria was calculated by counting the living bacterial colonies using the following method of Ogimoto and Imai (1981).

$$Microbe \ population = \frac{Colony \ account \ (kol)}{0.05 \ x \ 10^{x} \ x \ 0.1 \ (ml)}$$
(1)

Note: x: tube xth retail series

3 RESULTS AND DISCUSSION

3. REZULTATI I RASPRAVA

3.1 Lignin isolation

3.1. Izoliranje lignina

The black liquor produced by formacell pulping was then isolated to obtain lignin isolates. Furthermore, the extraction process of lignin from black liquor was performed by adding water to 500 mL of the sample in a 2 L glass beaker and allowed to form a precipitate which was then separated by filtering and dried for three days at room temperature; afterwards, it was observed for its characteristics (Table 1).

Parameter / Parametar	Characteristic / Svojstvo
Color / boja	Black / crna
Form / konzistencija	Solid / čvrsta
рН (25 °C)	4.5
Yield / prinos	1.74%
Water content / sadržaj vode	0.24%

Table 1 Characteristics of isolated lignin**Tablica 1.** Svojstva izoliranog lignina

Lignin isolates obtained have a black appearance, and they are solids in the form of fiber crumbs. They have a moisture content of 0.24 %. The yield of lignin obtained is 1.74 %, and this is presumed because there was no addition of acid to the precipitation process.

3.2 Lignin monomer fraction screening for *L*. casei

3.2. Probir frakcije monomera lignina za *L. casei*

The process of lignin degradation was performed by dissolving it into a solution of $CuSO_4$, pyridine, and NaOH, after which it was stirred and H_2O_2 was added. The obtained fraction was then added to the chromatographic column silica and eluted with CHCl₃, 3 % MeOH: CHCl₃, 20 % MeOH: CHCl₃, and MeOH, respectively. Then, each fraction was dried. The fraction of CHCl₃, 3 % MeOH:CHCl₃, 20% MeOH:CHCl₃, and MeOH yielded 10.68 %, 6.34 %, 11.38 %, and 44.85 %, respectively. The results of each fraction were identified on a TLC plate using UV light to examine the content of chemical compounds qualitatively.

Black spots on the TLC showed the presence of chemical compounds. Furthermore, the spot on TLC of each fraction consists of different compounds based on their retention time. For example, the spots generated at fractions of CHCl₃, 3 % MeOH: CHCl₃, 20 % MeOH: CHCl₃, and MeOH sequentially produced 4, 4, 2, and 1 spot, which was considered to be the dominant compound in each lignin fraction. Figure 2 and Table 2 show the black spot on TLC and the screening results for *L. casei* growth, respectively (Table 2).

Table 2 Screening lignin fraction as a prebiotic for L case	ei
Tablica 2. Probir frakcije monomera lignina za L. casei	

Fraction	Number of microbes, 10 ² colony/mL
Frakcija	<i>Broj mikroba</i> , 10 ² <i>kolonija</i> /mL
CHCl ₃	1.48 ± 0.29
3 % MeOH:CHCl ₃	4.52 ± 0.10
20 % MeOH:CHCl,	2.58 ± 0.23
МеОН	2.41 ± 0.34

The 3 % MeOH:CHCl₃ fraction showed higher prebiotic activity than other fractions. It showed prebiotic activity against *L casei* at 4.52 x 10² colonies/mL, while the CHCl₃ fraction, 20% MeOH:CHCl₃, and MeOH showed prebiotic activity against *L. casei* at 1.48 x 10² colonies/mL, 2.58 × 10² colonies/mL, and 2.41 × 10² colonies/mL, respectively.

3.3 Identification of 3 % MeOH:CHCl₃ fraction compound content

3.3. Identifikacija spojeva u frakciji 3 % MeOH:CHCl₃

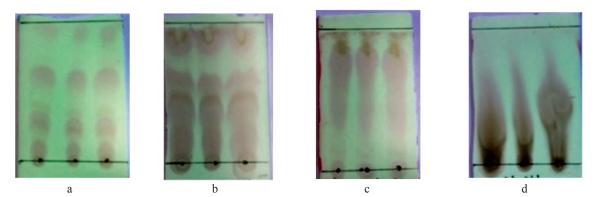
The identification process was performed by injecting a 3 % MeOH:CHCl₃ fraction into the GC-MS (Figure 3).

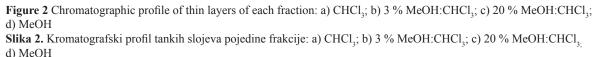
The 3 % MeOH:CHCl₃ fraction contains (a) benzaldehyde, 4-hydroxy-3,5-dimethoxy- 2.76 %, (b) 1-methylbutyl hexadecanoate as much as 41.03 %, (c) oleic acid as much as 3.61 %, and (d) di-2-ethylhexyl phthalate as much as 31.25 %. Furthermore, the main product of the hydrolysis of lignin using NaOH catalysts is monomeric phenols and oligomers (Roberts *et al.*, 2011). Figure 4 and Table 3 show the compound structure of the 3 % MeOH:CHCl₃ fraction and the identification results, respectively.

3.4 Prebiotic activity of 3 % MeOH:CHCl₃ fraction against *L. casei* 3.4 Prebiotičko dielovanje frakcije 3 %

3.4. Prebiotičko djelovanje frakcije 3 % MeOH:CHCl₃ na *L. casei*

Prebiotic activity test uses a concentration between growth media with a fraction of 3% methanolchloroform of 0, 2.5, 5, 7.5, 10, 12.5, and 15 (%). The





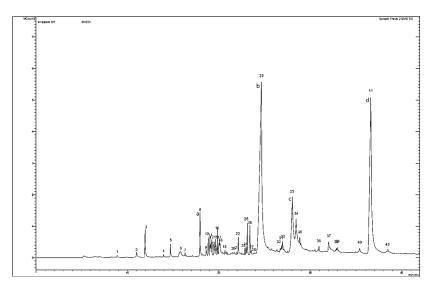


Figure 3 Fraction chromatogram of 3 % MeOH:CHCl₃: a) benzaldehyde, 4-hydroxy-3,5-dimethoxy; b) 1-methylbutyl hexadecanoate; c) oleic acid; d) di-2-ethylhexyl phthalate

Slika 3. Kromatogram frakcije 3 % MeOH:CHCl₃: a) benzaldehid, 4-hidroksi-3,5-dimetoksi; b) 1-metilbutil heksadekanoat; c) oleinska kiselina; d) di-2-etilheksil ftalat

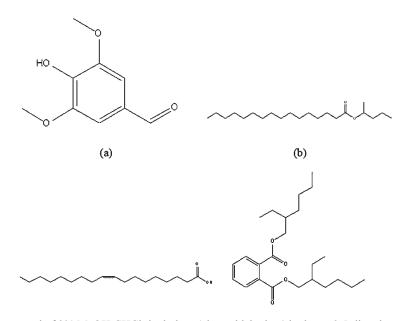


Figure 4 Fraction compound of 3% MeOH:CHCl₃ includes: a) benzaldehyde, 4-hydroxy-3,5-dimethoxy; b) 1-methylbutyl hexadecanoate; c) oleic acid and d) di-2-ethylhexyl phthalate

Slika 4. Spojevi u frakciji 3 % MeOH:CHCl₃ uključuju: a) benzaldehid, 4- hidroksi-3,5-dimetoksi; b) 1-metilbutil heksadekanoat; c) oleinsku kiselinu i d) di-2-etilheksil ftalat

number of colonies from the growth of *L. casei* for each concentration of 3 % MeOH:CHCl₃ fraction is shown in Figure 5.

The 3 % MeOH:CHCl₃ fraction at a concentration of 15 % showed the highest prebiotic activity of 6.10 x 102 colonies/mL against *L. casei*. In comparison, the 3 % MeOH:CHCl₃ fraction at a concentration of 2.5 % showed the lowest prebiotic activity of 2, 69 × 10² colonies/mL against *L. casei*. The prebiotic activity of the 3 % MeOH:CHCl₃ fraction on the growth of *L. casei* is shown in Figure 6.

The results of the analysis of the prebiotic activity of the 3 % MeOH:CHCl₃ fraction showed that the higher the concentration of the growth medium, the higher the growth of *L. casei*. Baurhoo *et al.* (2008) stated that the lignin monomer in the alcell process acts as a prebiotic. Prayuwidayati *et al.* (2016) reported that formacell purification products are generated from EPOB and oligomers, which are phenolic compounds that stimulate the growth of lactic acid bacteria. This shows that the isolated fraction of lignin and its derivatives can replace glucose in MRSA medium, and it is used as an energy source for the growth of *L. casei*. These also showed that the isolated lignin fraction could be used as a feed supplement and support the growth of *L. casei*. Lignin in the purified alcell

Table 3 Identification of lignin fraction monomer compounds of 3 % MeOH:CHCl,
Tablica 3. Identifikacija spojeva u frakciji 3 % MeOH:CHCl, monomera lignina

Retention time Vrijeme retencije	Molecule weight Molekulska masa					
8.886	207	Phenol.2-(1-methylpropyl)methylcarbamate	0.12			
11.008	212	Propanoic acid.3-chloro4-formylphenyl ester	0.38			
11.942	152	anillin				
13.958	166	hanone.1-(4-hydroxy-3-methoxyphenyl)-				
14.709	166	ndecanoic acid.10-methylmethyl ester				
15.839	214	Benzoic acid.4-hydroxy-3-methoxy-	1.04			
16.313	168	Diethyl phthalate	0.13			
17.952	222	Benzaldehyde.4-hydroxy-3.5-dimethoxy-	2.76			
18.621	182	p-Anisic acid.4-nitrophenyl ester	0.32			
18.838	273	m-Anisic acid.3.4-dichlorophenyl ester	0.90			
19.022	413	Carbamic acid.N-[1.1- bis(trifluoromethyl)ethyl]-4.(1.1.3.3-tetra- methylbutyl)phenyl ester	0.95			
19.114	220	4-Methyl-2-tert-octylphenol	0.54			
19.235	296	m-Anisic acid.3.4-dichlorophenyl ester	1.10			
19.447	270	Hexestrol	0.58			
19.643	220	Phenol.2-methyl-4-(1.1.3.3- tetramethylbutyl)-	0.33			
19.708	192	1.3-Dimethyl-5-ethyladamantane	0.74			
19.870	413	Carbamic acid.N-[1.1- Bis (trifluoromethyl)ethyl]-4.(1.1.3.3- tetra- methylbutyl)phenyl ester	1.06			
20.034	220	Phenol.2-methyl-4-(1.1.3.3- tetramethylbutyl)-	0.71			
20.126	228	etradecanoic acid				
21.690	268	2-Pentadecanone.6.10.14-trimethyl-	1.17			
21.980	194	Caffeine	0.12			
22.135	278	1.2-Benzenedicarboxylic acid.bis(2- methylpropyl) ester	0.70			
22.520	338	Erucic acid	0.09			
22.848	604	Tritetracontane	0.28			
22.968	268	9-Hexadecenoic acid. methyl ester.(Z)-	0.41			
23.116	276	7.9-Di-tert-butyl-1-oxaspiro(4.5)deca-6.9-diene-2.8 dione	1.60			
23.395	270	Pentadecanoic acid.14-methylmethyl ester	1.37			
23.358	292	Benzenepropanoic acid.3.5-bis(1.1-dimethylethyl)-4 hydoxy . methyl ester	0.20			
24.659	326	1-Methylbutyl hexadecanoate	41.03			
26.713	298	1-Eicosanol	0.23			
26.837	294	9.12-Octadecadienoic acid (Z.Z)methyl ester	0.32			
26.971	352	9.12.15-Octadecatrienoic acid.2.3 dihydroxypropylester.(Z.Z.Z)-	0.58			
28.036	282	Oleic acid	0.89			
28.453	282	Oleic acid	3.61			
28.853	282	Oleic acid	0.33			
30.953	604	Tritetracontane	0.21			
31.997	324	4.8.12.16-tetramethylheptadecan-4-olide	0.57			
32.860	298	1-Eicosanol	0.11			
32.963	604	Tritetracontane	0.16			
35.399	242	1-decanol.2-hexyl-	0.25			
36.624	390	Di-2-ethylhexyl phthalate	31.25			
38.491	592	1-Hentetracontanol	0.21			

process showed a prebiotic effect on chickens, promoting the growth of beneficial bacteria and improving intestinal morphological structure, as measured by an increase in the number of villi and goblet cells (Baurhoo *et al.*, 2008). Cotta and Whitefield (1998) stated that all hemicellulolytic rumen bacteria could utilize xylooligosaccharides as growth substrates. Therefore, increased digestibility of crude protein and fiber supports ruminant protein and energy metabolism. The non-digestible carbohydrate prebiotics includes lactulose, inulin, resistant starch, and a number of oligosaccharides, a source of carbohydrates for beneficial bacteria in the digestive tract (Crittenden, 1999). Substrates such as inulin, fructooligosaccharides (FOS), and mannan-oligosaccharides (MOS), derived from yeast cells, absorbed the host and were hydrolyzed by endogenous digestive enzymes. The possible mechanism is a decrease in pH due to the

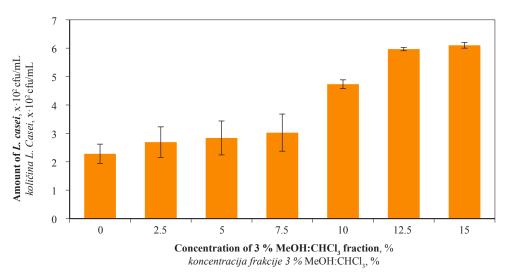


Figure 5 Effect of several concentrations of 3 % MeOH:CHCl₃ fraction as a prebiotic on the number of *L. casei* microbes **Slika 5.** Utjecaj nekoliko koncentracija frakcije 3 % MeOH:CHCl₃ na broj mikroba *L. casei*

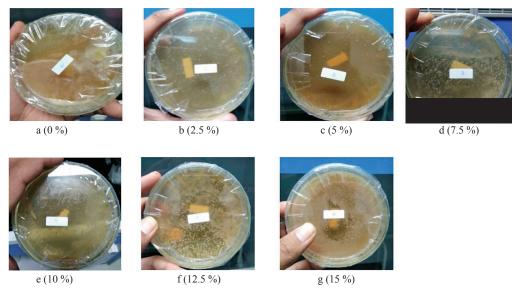


Figure 6 Prebiotic activity of 3 % MeOH:CHCl₃ fraction on the growth of *L. casei* Slika 6. Utjecaj prebiotičke aktivnosti frakcije 3 % MeOH:CHCl₃ na rast *L. casei*

production of short-chain fatty acids, bacteriocin secretion, and immune stimulation.

Meanwhile, MOS as a prebiotic has a different mechanism that does not cause an increase in the population of beneficial bacteria, due to its ability to attach to mannose-specific lectins of G-negative type 1 fimbriae pathogens such as Salmonella and *E. coli*, which is excreted from the digestive tract (Toma and Prokotnieks, 2006; Crittenden, 1999). Similar to the effect of MOS and antibiotic-free diet supplemented with 1.25 % alcell lignin (LL) on Lactobacilli and Bifidobacteria, lignin at low levels is potentially classified as a prebiotic (Baurhoo *et al.*, 2008). The results showed that using lignin in the alcell process (12.5 g) increases the concentration of intestinal Lactobacilli and Bifidobacteria in broilers (Baurhoo *et al.*, 2008). In addition, the proliferation of gastrointestinal microbiota populations such as Lactobacilli and Bifidobacteria was increased through the consumption of prebiotics. The increase in the number of microbiotic in the feces ranges from 10-100 times (Thomas *et al.*, 2004).

4 CONCLUSIONS

4. ZAKLJUČAK

Lignin, the main ingredient of black liquor generated from bunches of empty oil palm fruit, contains benzaldehyde, 4-hydroxy-3,5-dimethoxy, 1-methtylbutyl hexadecanoate, oleic acid, and di-2-ethylhexyl phthalate. Furthermore, its fraction of 3 % MeOH:CHCl₃ with a concentration of 15 % showed prebiotic activity against *L. casei* of 6.1×10^2 colonies/m. These results indicated that the lignin monomer is potentially prebiotic.

Acknowledgements - Zahvala

Thank you to the Ministry of Education and Culture of the Republic of Indonesia for the financial assistance, the laboratory for testing the quality of agricultural products, the Faculty of Agriculture for the support of laboratory facilities.

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Effects of Medium Density Fiberboards (MDF) Recycling Methods on Fiber Dimensions and Some Reconstructed Board Properties

Učinci recikliranja srednje guste ploče vlaknatice (MDF) na dimenzije vlakana i na neka svojstva ploča od recikliranih vlakana

ORIGINAL SCIENTIFIC PAPER

Izvorni znanstveni rad Received – prispjelo: 20. 5. 2022. Accepted – prihvaćeno: 3. 8. 2022. UDK: 674.817 https://doi.org/10.5552/drvind.2023.0037 © 2023 by the author(s). Licensee Faculty of Forestry and Wood Technology, University of Zagreb. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license.

ABSTRACT • In this study, different methods, including acid hydrolysis (phosphoric and formic acid), thermohydrolysis, and microwave methods were tested for recycling of fiberboards made of 100 % beech and 70 % beech + 30 % pine wood fibers, which are widely sold in the market. The impacts of the using methods on the fibers were investigated with the help of a device that determines the fiber dimensions according to optical principles. In a laboratory setting, test boards were constructed using recycled fibers, and the changes in certain physical and mechanical qualities were studied. As a result, it was determined that recycled fibers obtained from microwave and thermo-hydrolysis fibers could be effectively used in fiberboard production. However, the proportion of fibers, longer than 1.24 mm, was decreased by about 30 % with the phosphoric acid method for MDF samples composed of 100 % beech fiber. The fiberboards could not be obtained from fibers recycled through acid hydrolysis except for phosphoric acid method with 100 % beech fibers.

KEYWORDS: waste MDF; recycling; recycled fibers; fiber properties; board strenght

SAŽETAK • U radu se istražuju različite metode recikliranja ploča vlaknatica kao što su kiselinska hidroliza (uz pomoć fosforne i mravlje kiseline), toplinska hidroliza i mikrovalne metode. Reciklirani su uzorci ploča vlaknatica izrađenih od 100 % vlakana drva bukve te ploča vlaknatica izrađenih od 70 % vlakana drva bukve i 30 % vlakana drva crnog bora. Ploče vlaknatice takvog sastava uvelike su zastupljene na tržištu. Utjecaj metoda recikliranja na vlakna istraživan je uz pomoć uređaja kojim se određuju dimenzije vlakana na optičkim načelima. Ploče od recikliranih vlakana izrađene su u laboratorijskim uvjetima i na njima su promatrane promjene određenih fizičkih i mehaničkih svojstava. Utvrđeno je da se reciklirana vlakna dobivena mikrovalnim metodama i toplinskom hidrolizom mogu učinkovito primjenjivati u proizvodnji ploča vlaknatica. Međutim, kiselinskom hidrolizom uz dodatak fosforne kiseline u uzorcima izrađenim od 100 % bukovih vlakana udio vlakana duljih od 1,24 mm smanjuje se za

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oko 30 %. Ploče vlaknatice nisu se mogle proizvesti od recikliranih vlakana dobivenih kiselinskom hidrolizom. Za njihovu proizvodnju pogodna su se pokazala samo reciklirana vlakna od 100 % bukovih ploča vlaknatica srednje gustoće dobivena kiselinskom hidrolizom uz dodatak fosforne kiseline.

KLJUČNE RIJEČI: otpadni MDF; recikliranje; reciklirana vlakna; svojstva vlakana; svojstva ploče

1 INTRODUCTION

1. UVOD

Fiberboards, particularly medium-density fiberboards (MDF), have surpassed other wood-based boards as one of the most important raw materials in the furniture industry, owing to their more homogenous and superior surface structure (Moezzipour *et al.*, 2018). The total global production of MDF and highdensity fiberboard (HDF) was 71 164 023 m³ in 2010, and 104 095 018 m³ in 2020, according to the 2021 statistical data released by the Food and Agriculture Organization of the United Nations (FAO). Turkey's overall production of MDF and HDF increased from 3 265 000 m³ to 4 775 000 m³ bet from 2010 to 2020 (FAOSTAT, 2021).

Rapid expansion in the fiberboard industry is putting strain on available forest resources. With the developments in other forest industry sectors, it is expected that the supply of raw wood materials will become constrained in the near future (Demirkir and Colak, 2006). Additionally, like with other wood-based composites, leaving MDFs uncontrolled in landfills or burning them as fuel at the end of their lifecycle is expected to cause serious environmental problems (Demirkır and Çolak, 2006; Lykidis and Grigoriu, 2008; Nuryawan et al., 2020). Burning of this kind of products releases toxic gases such as formaldehyde to the atmosphere as a result of the use of formaldehydebased thermoset glue matrices such as urea-formaldehyde (UF), phenol-formaldehide and melamine-formaldehyde in their manufacture, as well as greenhouse gases such as methane and carbon dioxide (Lubis et al., 2018a; Moezzipour et al., 2017; Zeng et al., 2018; Hagel et al., 2021).

Recycling these waste boards into products and raw materials provides a significant solution to both solid waste management and raw material shortages in the relevant sectors (Demirkır and Çolak, 2006). In this context, mechanical, thermo-hydraulic, chemical, and other techniques for combining them have all been developed in recent years (Antov and Viktor, 2019). Thermo-hydrolysis recycling methods, which produce lignocellulosic fibers from fiberboards, are carried out by removing the polymer matrix established by the glues in the panel samples and generally by breaking the bonds between the polymer matrix and fiber using heat and water vapor (Lubis *et al.*, 2018a; Moezzipour *et al.*, 2018; Hagel *et al.*, 2021). Many different techniques for recycling wood fibers from composite panels have been developed based on this principle (Roffael and Hüster, 2012). Fiber/chip boards, to be smoothly recycled using the thermo-hydrolysis process with a chemical pre-treatment (WKI method), are first immersed in a solution containing a solution of urea, ammonia, or another chemical that dissolves the UF adhesive and then recycled in an autoclave at a suitable temperature (Michanickl and Boehme, 2002). Wan et al. (2004) reported that 0.5 % butane tetracarboxylic acid, together with heat treatment, successfully recycles particleboard and MDF samples made with UF. Lubis et al. (2018b) stated that alkaline hydrolysis removed 25 % of the UF polymer from waste MDF, while neutral hydrolysis removed 50 %, and acid hydrolysis removed 75 %. Additionally, a micro-release method has been developed with the purpose of generating atomic heat by activating the water and hydroxyl ions on the panel with microwave energy, specifically targeting to solve the bonds between fibers and also fiber adhesive (Demirkır and Çolak, 2006, Mitchell and Stevens, 2009).

Although numerous methods for recycling waste MDF panels have been developed, research on MDF recycling, the effect of recycling methods on fiber characteristics, and the qualities of boards made from recycled fibers are quite restricted. This study aims to:

- Compare some recycling methods for MDF samples produced from different fiber composition,
- Determine and compare the fiber and fiberboard properties for different MDF recycling methods,
- Analyze the effect of some studied recycling methods on fiber quality.

2 MATERIALS AND METHODS

2. MATERIJALI I METODE

2.1 Material

2.1. Materijal

In this study, thermo-mechanical pulping (TMP) fibers, which are produced using 100 % *Fagus orianta-lis* (beech) and a *Fagus orientalis* (beech) - *Pinus nigra* (pine) mixture (70 % : 30 % w/w), and MDFs manufactured in the fiberboard production line from these fibers, were obtained from Kastamonu Entegre A.Ş. Kastamonu O.S.B. factory (Kastamonu, Turkey) according to the company production specifications. The MDF samples were cut into strips of 1 cm width and of appropriate length, using various cutting tools to pre-

pare them for recycling, and then stored under room conditions.

Ammonium chloride, ammonium sulfate, formic acid, and phosphoric acid were provided in analytical purity for the recycling procedures used in the study. To manufacture boards from recycled fibers, urea-formaldehyde adhesive with 55 % solids content (urea/ formaldehyde ratio of 1:1.17; pH 8.4; density 1.24 g/ cm³; viscosity 65 cps at 25 °C) was used, along with 10 % ammonium chloride solution as a hardener.

2.2 Recycling of fiberboards

2.2. Recikliranje ploča vlaknatica

The boards were recycled in accordance with the experimental design shown in Table 1. Samples separated for recycling by the micro release method were kept in boiling distilled water for 5 minutes before being processed for 5 minutes at medium wave frequency in an industrial microwave device. The MDF samples then swelled and lost their integrity, and their fibers were separated from each other. The fibers were dispersed manually and dried under room conditions (Mitchell and Stevens, 2009). The WKI method, which is a thermo hydraulic method, was modified and applied to the samples. Instead of urea or ammonia solutions, 25 % solutions of ammonium chloride (WKI 1) and ammonium sulfate (WKI 2) were used in the method. The presence of acid ions in the aqueous solutions of both solutions was a significant factor in the selection of ammonium chloride and ammonium sulfate. In addition, these chemicals are widely employed by wood-based board factories as a hardener for UF resin (Pizzi, 2003). The MDF samples immersed in 25 % ammonium salt solutions were then treated in an autoclave at 121 °C for 30 minutes. After the autoclave, the samples were washed with distilled water to separate and fibers were dried under room conditions before the MDF production. Formic acid (AH 1) and phosphoric acid (AH 2) were chosen for MDF recycling via acid hydrolysis. The properties that contributed to the selection of these two acids, found in the literature, were the ability of formic acid to dissolve cellulose even at high concentrations without abrasion (Hafizoğlu and Deniz, 2010) and the ability of phosphoric acid to catalyze the hydrolysis of UF adhesive (Lall *et al.*, 1982). The MDF samples to be recycled were heated and mixed in a 25 % acid solution until the fibers separated. After washing the acquired fibers with distilled water with the help of filter paper, they were left to dry under room conditions.

2.3 Investigation of changes in fiber morphologies

2.3. Istraživanje promjena u morfologiji vlakana

Dimensional analyses (length and width) of fiber samples taken prior to the production and recycling of the boards were performed using the Imal brand optical laboratory fiber scanner (Fibercam 100®, Imal Pal, Italy). The gadget, in which certain masses of the obtained fibers were placed, automatically determined the percent fiber amount in certain frequency ranges according to the total fiber measured optically.

2.4 Production of fiberboard from recycled fibers and determination of some properties

2.4. Proizvodnja ploča vlaknatica od recikliranih vlakana i određivanje nekih njihovih svojstava

The panels were manufactured from dried recycled wood fibers at a target density of 700 kg/m³ and had dimensions of 0.360 m \times 0.310 m \times 0.011 m. A drum gluing mixer, with two-point injection, 100 lt sample volume, and 5 rpm rotating speed, was used in the gluing process of the recycled fibers in fiberboard production for 10 minutes. Samples were pressed with a laboratory press (SSP 125, Cemil Usta, Turkey) for 2 MPa at 190 °C for 8 min, including the warm-up time of the additional press plates used. However, some acid hydrolyzed fiber groups were deformed during hot pressing. After that, the undeformed fiberboards at the end of the hot press were conditioned in the air-condi-

Board type	Recycle method	Applied chemical				
Vrsta ploče	Metoda recikliranja	Upotrijebljene kemikalije				
	Micro release / mikrootpuštanje	Distilled water / destilirana voda				
100.0/harsh	AH 1	25 % Formic acid / 25 % mravlja kiselina				
100 % beech 100 % bukovina	AH 2	25 % Phosphoric acid / 25 % fosforna kiselina				
100 % bukovina	WKI 1	25 % Ammonium chloride / 25 % amonijev klorid				
	WKI 2	25 % Ammonium sulfate / 25 % amonjjev sulfat				
- - - - - - - - - -	Micro release / mikrootpuštanje	Distilled water / destilirana voda				
Beech - pine mixture $(70 + 20.9)$	AH 1	25 % Formic acid / 25 % mravlja kiselina				
(70 : 30 % w/w) smjesa bukovine i borovine (70 : 30 % težine)	AH 2	25 % Phosphoric acid / 25 % fosforna kiselina				
	WKI 1	25 % Ammonium chloride / 25 % amonijev klorid				
(10.50/01021110)	WKI 2	25 % Ammonium sulfate / 25 % amonjjev sulfat				

 Table 1 Experiment design of MDF recycling

 Tablica 1. Eksperimentalni postupci recikliranja MDF-a

tioned room. The following properties of the boards were determined according to related standards:

- Density (d: kg/m³) (EN 323, 1993)
- Thickness swelling percent in 24 hours (TS: %) (EN 317, 1993)
- Water absorption percent in 24 hours (WA: %) (EN 317, 1993)
- Bending Strength (MOR: N/mm²) (EN 310, 1993)
- Modulus of elasticity (MOE: N/mm²) (EN 310, 1993).

3 RESULTS AND DISCUSSION3. REZULTATI I RASPRAVA

3.1 Effect of recycling methods on fiber morphology

3.1. Utjecaj metoda recikliranja na morfologiju vlakana

The fibers obtained from the boards via the recycling methods were examined using an optical laboratory fiber scanner, and the fiber length analysis results obtained from the device are given in Table 2.

Table 2 shows that recycling procedures have a strong influence on the fiber lengths of 100 % beech fibers. Contrary to the raw fiber samples, the micro release technique had no effect on the rate of fine fibers. However, a considerable increase was observed in recycled fine fibers with modified WKI procedures. In addition, a certain amount of decrease is observed in fiber fractions with lengths greater than 1.24 mm, which can be classified as coarse fibers. This indicates that recycling processes have a negative effect on fiber lengths. Şahin (2014) stated that, in the recycling of

paper fibers, there is generally a serious decrease in fiber length and strength until the 4th recycling of the fibers and that the recycled fibers are shorter than the fibers originally obtained from wood. In addition, in another study, fibers shorter than 0.09 mm were defined as fine fibers, and it was stated that the increase in the percentage of fine fibers caused a significant decrease in the elasticity modulus determination values of the sheets, especially in bending (Moezzipour et al., 2018). Another study claimed that, with the addition of recycled fibers at different rates, the fiber size decreased in the long fraction (1.711 mm and above), and the percentage of the short fiber class (between 0.2-0.956 mm) increased (Klimczewski and Nicewicz, 2013). Hagel et al. (2021) concluded that the fibers recycled by the steam reactor are short and that these fibers are shortened with the application of the refinery process. The results obtained in this study are consistent with previous studies on fiber recycling in the literature (Hagel et al., 2021).

Similar to results obtained using 100 % beech fibers, recycling methods have a negative effect on the fiber lengths of fibers produced from a mixture of 70 % beech and 30 % pine as shown in Table 2. In particular, a clear increase in the percentage of coarse and fine fibers was observed, whereas the coarse fiber fractions decreased (length 1.24 mm).

The results of the fiber width analysis of the recycled fibers using the optical fiber scanner device are shown in Table 3.

The negative effects of certain recycling methods on fiber width distributions are evident in Table 3. It

Board type	Recycle method												Recycle Raspon mierenia veličine vlakana mm						Measured fiber count
Vrsta ploče	Metoda recikliranja	0.00 / 0.09	0.09 / 0.13	0.13 / 0.22	0.22 / 0.51	0.51 / 1.24	1.24 / 1.98	1.98 / 3.0	3.0 / 4.0	4.0 / 5.0	5.0 / 30.0	Broj izmjerenih vlakana							
	Raw fiber sirova vlakna	0.1*	1.6	4.1	18.7	36.1	18.2	11.6	4.6	2.3	2.6	139058							
100 % beech	Micro release mikrootpuštanje	0.1	1.7	3.9	18.5	39.1	19.1	9.8	4.1	2.2	1.6	335056							
100 % bukovina	AH 1	0.1	1.5	3.9	19.0	39.1	17.9	10.1	4.1	2.8	1.5	336228							
συκονιπά	AH 2	0.3	2.8	5.9	24.6	39.3	15.1	7.5	2.4	1.4	0.8	305972							
	WKI 1	0.3	2.6	5.1	20.7	38.8	17.6	9.1	3.3	1.6	1.0	396388							
	WKI 2	0.2	2.3	4.8	20.3	37.7	16.7	9.9	4.1	2.1	2.0	367983							
Beech - pine mixture	Raw fiber sirova vlakna	0.1	1.7	4.0	18.4	33.4	16.9	12.1	5.2	3.6	4.7	137472							
(70:30 % w/w)	Micro release <i>mikrootpuštanje</i>	0.1	1.5	3.7	17.1	35.3	18.2	11.9	5.4	3.4	3.4	300061							
smjesa	AH 1	0.1	1.4	3.7	17.6	36.0	18.2	11.6	5.0	3.0	3.6	372036							
bukovina i	AH 2	0.1	1.1	3.1	16.4	35.2	20.0	12.2	5.2	5.0	1.8	53639							
borovine (70 : 30 %	WKI 1	0.2	2.1	4.7	19.4	35.9	16.9	10.9	4.8	3.0	2.8	254811							
(70 : 30 % težine)	WKI 2	0.2	2.4	5.1	20.8	35.2	16.0	9.9	4.0	2.3	3.3	332847							

Table 2 Fiber length analysis results**Tablica 2.** Rezultati analize duljine vlakana

* The % value of the measured fiber amount and the number of fibers at the relevant frequency / postotna vrijednost izmjerenih vlakana i broj vlakana pri relevantnoj frekvenciji

Board type	Recycle method		Measured fiber count						
Vrsta ploče	Metoda recikliranja	0 / 0.09	0.09 / 0.13	0.13 / 0.22	0.22 / 0.51	0.51 / 1.24	1.24 / 1.98	1.98 / 3.0	Broj izmjerenih vlakana
	Raw fiber sirova vlakna	7.4*	20.2	37.0	26.5	9.0	-	-	139058
100 % beech	Micro release mikrootpuštanje	8.4	22.0	39.0	25.7	5.0	-	-	335056
100 % bukovina	AH 1	9.9	23.0	38.6	23.2	4.7	0.6	-	336228
	AH 2	10.8	26.3	39.5	19.8	3.6	-	-	305972
	WKI 1	8.0	21.2	38.3	27.9	4.6	-	-	396388
	WKI 2	9.7	23.0	37.5	24.1	5.4	0.3	-	367983
Beech - pine	Raw fiber sirova vlakna	9.6	22.3	37.9	21.1	9.1	-	-	137472
mixture (70 : 30 % w/w) smjesa bukovine I borovine (70 : 30 % težine)	Micro release mikrootpuštanje	7.9	21.5	39.4	24.3	6.9	-	-	300061
	AH 1	8.3	22.7	40.5	22.6	6.1	-	-	372036
	AH 2	6.3	20.7	40.6	22.5	7.7	2.1	-	53639
	WKI 1	10.0	24.0	38.5	21.2	6.3	-	-	254811
	WKI 2	8.6	22.7	39.3	22.9	6.5	-	-	332847

Table 3 Fiber width analysis results**Tablica 3.** Rezultati analize širine vlakana

* The % value of the measured fiber amount and the number of fibers at the relevant frequency / postotna vrijednost izmjerenih vlakana i broj vlakana pri relevantnoj frekvenciji

was observed that some fibers were extremely thick, and there was a particularly significant increase in the ratio of fibers with a very small width (<0.22 mm). The primary reason for this is the glue and paraffin residues on the recycled fibers. These microscopic residues can remain on the surfaces of the recycled fibers and even reduce the bonding ability of the fibers as they bind to the -OH groups in the fibers (Zeng et al., 2018). The increase in fine fiber fractions may be due to the fact that they damage the fibers while breaking off during recycling, and the optical fiber scanner (Fibercam 100 (R) probably classifies them as fines since they are separated from the fibers by breaking off a certain piece. Lubis et al. (2020) recycled fibers via hydrolysis with water and oxalic acid and observed that the amount of total nitrogen in the plate increased, although the amount of mass lost increased with the increase in the amount of recycled fiber in the final board production.

Similar to the results obtained using 100 % beech fibers, Table 3 shows that recycling methods have a negative effect on the fiber width of fibers produced from a mixture of 70 % beech and 30 % pine. In particular, an increase in the amount of fine and thin fibers was clearly observed, while there was a decrease in coarse fiber fractions (width \geq 1.24 mm).

3.2 Characteristics of boards produced from recycled fibers

3.2. Svojstva ploča proizvedenih od recikliranih vlakana

Table 4 shows the performance of MDF panels made from recovered fibers via various recycling procedures from 100 % beech and 70 % beech + 30 % pine

boards. In both types of boards, fibers recycled with formic acid and fibers recycled with phosphoric acid from 70 % beech + 30 % pine boards, deformation occurred in the fibers during hot pressing, compromising the board integrity. As a result, board manufacturing in these groups was less effective than in others. The acid hydrolysis process causes damage to the fibers, and the fact that the condensation processes of the UF resin are complete before the hot pressing process and are hydrolyzed during pressing with the impact of the remaining acid in the fibers, are the primary reasons for this (Lubis *et al.*, 2018b). By treating these fibers with high-concentrated acid pre-treatment, they can be assessed as raw materials for ethanol production (Zhao *et al.*, 2019).

The analysis of the densities of the recycled MDF panel samples, presented in Table 4, clearly shows that they met the goal density and that the obtained results were equivalent to those of the original fiberboard samples. Figure 1 was created according to the data in Table 4 to enable the comparison of the 24-hour water absorption. The water absorption performance of the recycled fibers was found to be quite poor. This is because the original MDF samples were sourced from a mill, and the majority of companies used additives to improve water absorption values. Additionally, the shrinkage of the fibers and the residual UF resin on the fibers hindered the fiber-resinfiber bonds during gluing (Zeng et al., 2018). Apart from the hydrolytic degradation of UF, the boards became more dimensionally unstable, a reaction that is likely to occur during the hot press process in both residual glue and newly added glue in the recycled

		1	1			
Board type Vrsta ploče	Recycle method Metoda recikliranja	Density, kg/m ³ Gustoća, kg/m ³	24 h WA, %	24 h TS, %	<i>MOR</i> , N/mm ²	<i>MOE</i> , N/mm²
	Original fiberboard originalna ploča vlaknatica	720.17 (9.22)	29.31 (1.31)	5.05 (0.26)	31.10 (1.25)	3315.59 (52.94)
-	Micro release mikrootpuštanje	733.69 (13.57)	107.05 (13.02)	36.88 (5.14)	6,79 (1.79)	1998.95 (407.60)
	AH 1	_*	-	-	-	-
100% beech 100% bukovina	AH 2	706.02 (21.58)	75.97 (1.50)	8.72 (0.74)	3.19 (0.89)	2116.44 (802.34)
-	WKI 1	711.97 (16.16)	59.65 (3.64)	21.54 (2.54)	5.18 (1.04)	3274.54 (727.87)
_	WKI 2	713.15 (21.71)	96.68 (7.36)	16.29 (2.56)	8.94 (2.21)	5087.742 (1425.99)
	Original fiberboard originalna ploča vlaknatica	723.00 (8.27)	33.36 (1.48)	8.15 (1.71)	32,39 (1.76)	3260.64 (87.31)
Beech - pine mixture (70 : 30	Micro release mikrootpuštanje	703.23 (24.56)	104.94 (8.41)	29.12 (6.79)	6.36 (1.59)	2536.91 (672.25)
% w/w)	AH 1	-	-	-	-	-
bukovina	AH 2	-	-	-	-	-
– borovina (70 : 30 % težine)	WKI 1	733.45 (13.19)	85.06 (6.98)	30.45 (4.01)	2.95 (0.64)	828.57 (347.12)
	WKI 2	715.46 (20.26)	97.48 (4.42)	27.30 (6.36)	5.80 (1.12)	1452.48 (344.26)

Table 4 Physical and mechanical properties of boards with standard deviations **Tablica 4.** Fizička i mehanička svojstva ploča sa standardnim devijacijama

* not produced / nije proizvedena

boards (Nuryawan *et al.*, 2020). In general, the water uptake values of the sample groups, hydrolyzed by chemical treatment, were lower than those obtained for MDF samples via micro release method. The reason for this situation can be attributed to the high percentage of coarse fibers in the methods using chemicals compared to the micro-release method.

The swelling ratios, measured from the thickness of the MDF panel samples, after 24 hours, are showed in Figure 2, which was prepared according to the data in Table 4. Although the changes in swelling values are in parallel with the changes in water absorption values, the 100 % beech fiber recycled boards treated with phosphoric acid gave quite good results compared to the original fiberboard samples.

The results of bending strength tests on MDF panel products acquired through various recycling processes are shown in Figure 3. Bending strength data were found to be quite low compared to those of the original fiberboards. The reason for this can lie in the decrease in fiber lengths and also in the fact that the fiber-fiber and fiber-glue-fiber bonds are not formed efficiently in panel production and that solidi-fied UF resin on the recycled fibers prevents fiber-fiber bonding ability (Zeng *et al.*, 2018; Bütün Buschalski and Mai, 2021). Moezzipour (2018) de-

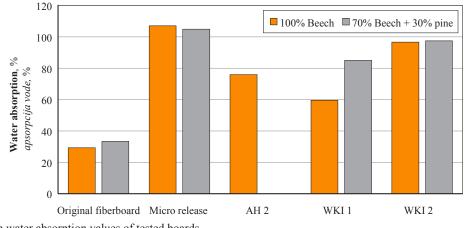


Figure 1 24 h water absorption values of tested boards **Slika 1.** Vrijednosti apsorpcije vode ispitivanih ploča nakon 24 sata

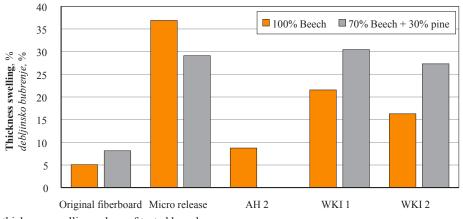


Figure 2 24 h thickness swelling values of tested boards **Slika 2.** Vrijednosti debljinskog bubrenja ispitivanih ploča nakon 24 sata

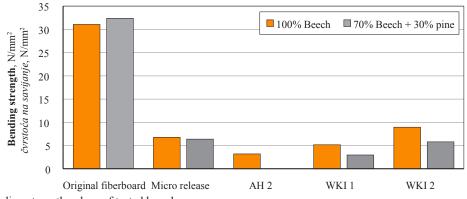


Figure 3 Bending strength values of tested boards **Slika 3.** Vrijednosti čvrstoće ispitivanih ploča na savijanje

fined fibers shorter than 0.09 mm as fines and stated that the increase in the amount of fines, in particular, caused a noticeable decrease in the elasticity modulus values determined for the MDF panel samples.

When evaluating the modulus of elasticity in bending, the obtained MDF sample groups from 100 % beech fibers, treated with ammonium solutions, give more elastic boards than 70 % beech+30 % pine fiber mixture (Figure 4). In their study, Lykidis and Grigoriou (2008) demonstrated that the fiber elasticity increases with hydrothermal treatment. They stated that this situation was almost certainly the primary reason for the increase in the modulus of elasticity. Based on this, it can be said that the low modulus of elasticity of the fibers, obtained through the microwave method, results in more rigid fibers than the others. Microwave, defined as electromagnetic radiation, affects the hydroxyl groups and changes the felting properties of the fibers.

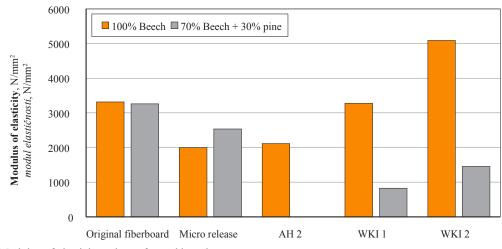


Figure 4 Modulus of elasticity values of tested boards **Slika 4.** Vrijednosti modula elastičnosti ispitivanih ploča

In addition to this, the decrease in the bending strength and modulus of elasticity in the acid-treated group (AH 2) shows that the fibers are damaged during both processing and drying of recycled fibers.

4 CONCLUSIONS 4. ZAKLJUČAK

As a result of the study, it was observed that the fibers obtained by recycling waste boards could be reused in the fiberboard production industry.

It was observed that the recycling process has an absolute effect on the fiber dimensions and that these changes directly affect the physical and mechanical properties of the fiberboards.

The micro-release method provided better quality recycled fibers and boards according to the tested and evaluated characteristics.

Ammonium salts are more readily available in solid form than ammonia solution. The mild acidity of these solutions also prevents undesirable reactions during fiber recycling process. The type of ammonium salts, used percentages, and process factors, including temperature and time, will change this approach.

Acid hydrolysis seriously damaged fibers, should be more suitable for other applications such as bioethanol manufacturing.

Acknowledgements – Zahvala

A part of this study was presented in the International Forestry Symposium IFS2016 held in 2016 by Kastamonu University Faculty of Forestry and placed in the Abstract Proceedings.

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Utilization of Scots Pine (*Pinus sylvestris* L.) Timber with Defects in Production of Engineered Wood Products

Iskorištavanje borovine (*Pinus sylvestris* L.) s greškama za proizvodnju kompozitnog drva

ORIGINAL SCIENTIFIC PAPER

Izvorni znanstveni rad Received – prispjelo: 4. 7. 2022. Accepted – prihvaćeno: 20. 10. 2022. UDK: 630*85; 674.06 https://doi.org/10.5552/drvind.2023.0048 © 2023 by the author(s). Licensee Faculty of Forestry and Wood Technology, University of Zagreb. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license.

ABSTRACT • This study presents opportunities for the utilization of timber by-products with defects for manufacturing engineered wood panels. Three gluing methods were proposed for this waste raw material derived from Scots pine (<u>Pinus sylvestris</u> L.) wood. The methods used for combining and gluing enabled a more complete and complex utilization of wood with defects. The physical properties (density and moisture content) and mechanical properties (bending strength and modulus of elasticity) of the laboratory-fabricated engineered wood panels were evaluated in accordance with the European standards. The highest density of 643 kg/m³ and bending strength values (28.6 N/mm²) were obtained from the panels manufactured using method 3 and veneered with beech veneer sheets. The modulus of elasticity of the laboratory-made engineered wood panels reached values of up to 5580 N/mm². This study demonstrated the feasibility of the utilization of defective wood pieces in the manufacturing of engineered wood panels.

KEYWORDS: <u>Pinus sylvestris</u> L.; engineered wood; knots; cross-laminated timber (CLT); solid wood panels

SAŽETAK • U radu je predstavljena mogućnost iskorištavanja otpadnog drva s greškama za proizvodnju kompozitnog drva u graditeljstvu. Predložene su tri metode lijepljenja otpadnog drva borovine (<u>Pinus sylvestris</u> L.). Metode kombiniranja i lijepljenja omogućile su potpunije iskorištavanje drva s greškama. Fizička svojstva (gustoća i sadržaj vode) i mehanička svojstva (čvrstoća na savijanje i modul elastičnosti) laboratorijski proizvedenih kompozitnih drvnih ploča za graditeljstvo ocijenjena su prema europskim standardima. Najveću gustoću (643 kg/m³) i čvrstoću na savijanje (28,6 N/mm²) imale su ploče proizvedene metodom 3 i furnirane bukovim furnirom. Modul elastičnosti laboratorijski proizvedenih kompozitnih drvnih ploča za graditeljstvo dosegnuo je vrijednost od 5580 N/mm². Ovo je istraživanje uputilo na mogućnost iskorištavanja drva s greškama za proizvodnju kompozitnih drvnih ploča namijenjenih graditeljstvu.

KLJUČNE RIJEČI: Pinus sylvestris L.; građevno drvo; kvrge; križno lamelirano drvo (CLT); masivne drvne ploče

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1 INTRODUCTION

1. UVOD

The demand for various types of wood to meet consumer demands is constantly increasing. Estimates indicate that the supply of large construction timber in Bulgaria will become increasingly scarce in the coming decades (Kostov, 1993; Kostov, 2009). This trend is also observed in other countries around the world (Warde, 2006; Nazir *et al.*, 2018; Alberdi *et al.*, 2020; Odppes *et al.*, 2021). This is due to the increased logging intensity in forests, which has resulted in a significant reduction in forest areas. All of these result in a reduction in the diameter of the round wood, as well as substantial difficulties in manufacturing finished products of the desired size and quality. This, in turn, necessitates the search for methods and technologies for more rational utilization of wood resources.

Scots pine (Pinus sylvestris L.) is one of the most commercially important tree species used in the Bulgarian wood and wood-based industry for a wide variety of value-added applications. The exploitation characteristics of Scots pine wood have been extensively studied and are well known (Trichkov, 2016; Trichkov, 2018). This wood is widely used in construction for construction materials, fasteners, formwork panels; for the interior furnishings of residential and public buildings; for the production of furniture, flooring, paneling and joinery; in the construction of railway lines and in mining; in the chemical and pharmaceutical industry; in shipbuilding and aircraft construction. The wide application of Scots pine wood is also due to its physical, mechanical, technological, operational and aesthetic qualities. It is relatively light and has high strength indicators. It is processed without difficulty and is highly durable (Mederski et al., 2015; Mclean, 2019; Burawska-Kupniewska et al., 2020).

In terms of industrial wood use, the distribution of round wood according to diameter should be emphasized. Recent data from the Bulgarian Executive Forest Agency (2020), given in Table 1, show that the areas afforested with Scots pine in the country have decreased over time.

During the period 2015-2020, the total stock increased by 2.6 million m³, primarily in plantations that have reached logging maturity, which is over 100 years. It should be noted that the majority of these forests are located in protected areas with very limited wood harvesting.

With the intensive reduction of forest stand diameter, more and more rational wood utilization is required. There are low quantitative and qualitative yields in the processing of thin logs in terms of size and quality characteristics (Heräjärvi, 2004; Campbell, 2013). The use of thin logs in the production of engineered wood products results in a significant loss of wood in the form of large and small waste, especially when multiple knots are present. The method of gluing quality wood after removing its flaws provides excellent opportunities for utilizing low-quality and thin round wood (Barbour *et al.*, 2003; Hernandez *et al.*, 2005; Lyhykainen *et al.*, 2009).

The size and number of knots in the wood have a significant impact on its physical and mechanical properties (As *et al.*, 2006; Koman *et al.*, 2013; Montero *et al.*, 2015; Burdarov, 2019; Kaiser, 2019; Wright *et al.*, 2019). The presence of many defects in the wood, particularly wood knots, significantly reduces the final quantitative yields in the production of engineered wood products. The percentage of defects in the wood is most noticeable in small-diameter logs (Koynov, 2016; Trichkov *et al.*, 2018).

To address the global shortage of wood raw materials, efforts are being made in several areas, including the search for new, alternative raw material sources, optimized use of wood and other lignocellulosic raw materials, reduction of wood waste generated during processing, recycling and upcycling of wood and wood-based materials, and efficient utilization of wood waste and by-products in various value-added industrial applications (Antov *et al.*, 2018; Antov and Savov, 2019; Neykov *et al.*, 2020; Lee *et al.*, 2022; Pędzik *et al.*, 2022).

 Table 1 Distribution of afforested area (ha) and stock (m³) of Scots pine in Bulgaria by age classes for 2015/2020 (Executive Forest Agency 2015/2020)

Tablica 1. Raspodjela pošumljene površine (ha) i zalihe bijelog bora (m³) prema dobnim razredima za 2015. i 2020. godinu (Executive Forest Agency 2015./2020.)

Pinus		Distribution by age classes (by years), thousand ha											
sylvestris L.		Raspodjela po dobnim razredima (po godinama), tisuće ha											
	Total	Total 1-20 21-40 41-60 61-80 81-100 101-120 121-140 Over / Više											
	Ukupno								od 140				
2015	553.6	50.1	215.5	132.5	51.9	61.4	33.9	7.2	1.1				
2020	534.2	29.6	148.9	191.6	49.4	57.1	44.9	11.0	1.7				
Pinus		·	Distrib	oution by ag	e classes (b	y years), mi	llion m ³						
sylvestris L.			Raspodjela	po dobnim r	razredima (p	o godinama,), <i>milijuni</i> m	3					
2015	143.9	4.1	50.7	39.3	15.3	20.7	11.4	2.1	0.3				
2020	146.5	1.9	34.4	58.1	14.8	19.0	15.0	2.9	0.4				

According to industrial data, the final quantitative yields in the production of engineered wood range between 25 % and 30 % of the volume of logs. In the processing of solid wood materials for the production of engineered wood, quantitative yields reach 30-35 %. If the solid wood materials are obtained from thin logs, this percentage is significantly decreased. The remaining wood (65-70 %) in the form of large and small waste and by-products with knots, shavings, sawdust, etc., is used as raw material for manufacturing pellets, technological chips or burned for energy. Thus, the valorization of timber waste and by-products in the production of value-added products, including engineered wood, represents an efficient way to achieve sustainable resource management.

Several techniques for bonding small pieces of solid wood and fabricating products with enhanced mechanical properties have been developed (Nairn, 2017; Nazerian at al., 2018). The lack of information on other commercial applications of large waste obtained in the production of engineered wood, aside from those mentioned above, makes it impossible to make an objective assessment of their complex recovery. This justifies the need of conducting extensive studies aimed at establishing more efficient ways of waste wood utilization in the production of finished products to replace conventional materials in the furniture industry. Therefore, the aim of this research work was to investigate the feasibility of using Scots pine timber with defects in manufacturing engineered wood panels. Different gluing methods and their effects on the physical and mechanical properties of the panels were also investigated.

2 MATERIALS AND METHODS

2. MATERIJALI I METODE

Large-sized timber with various defects (knots, resin pocked, bark pocked, slop of grain, etc.) was used

in this study (EN 844, 2019). These wood by-products were referred to as "waste pieces." They were obtained from Scots pine (*Pinus sylvestris* L.) wood during the industrial production of engineered wood products at the "SREDNA GORA AD" company (Stara Zagora, Bulgaria). The by-products were sorted after determining their dimensional characteristics. Three different methods for bonding these waste raw materials and producing solid wood panels were proposed in this study. The obtained physical and mechanical properties of the products were determined.

A graphical representation of the different methods used for gluing waste pieces generated during the manufacturing of engineered wood and their assembly into the final product, i.e. glued solid wood panels, is presented in Figure 1. The length of the waste pieces was primarily determined by the size and number of knots in the wood. Waste pieces had cross-sectional dimensions of 40 mm × 140 mm. Their length ranged from 114 to 125 mm. The moisture content found in the experimental wood after conditioning was 7-8 %. This value also corresponds to the moisture content of the materials for the production of engineered wood after drying and subsequent conditioning.

In the process of sawing, respectively removing the high-quality from the low-quality wood, an insignificant amount of wood dust was found on the waste pieces with defects. However, the test wood was cleaned of wood dust before being glued. In this way, quality bonding between the individual elements was ensured.

Timber by-products with defects were obtained by cutting lamellas of the same dimensions. This ensures that the waste pieces have the same cross-sectional dimensions but different lengths. To manufacture the final product, they were first glued together by smoothing (Figure 1-1), to get a lamella of the desired length. Following the formation of lamellas of the required length, they were glued on the edges to form a

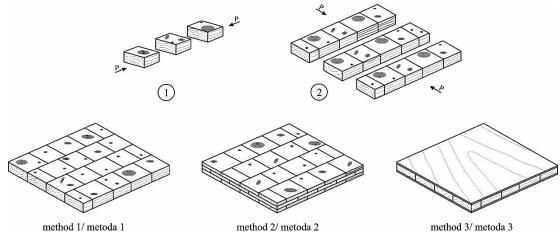


Figure 1 Bonding of Scots pine timber by-products obtained in production of engineered wood Slika 1. Lijepljenje nusproizvoda od borovine dobivenih u proizvodnji kompozitnog drva za graditeljstvo



Figure 2 Laboratory panels made from Scots pine timber by-products Slika 2. Laboratorijske ploče proizvedene od nusproizvoda borovine

solid wood panel, according to the presented methodology (Figure 1-2). Test specimens were cold-pressed at a pressure of 0.9 MPa in laboratory conditions. Fast drying PVAc glue was used in this study. Splicing of the waste pieces lengthwise to form lamellas, as well as the widthwise gluing of the resulting lamellas, was carried out using a laboratory hydraulic press.

Three methods of gluing the waste pieces were proposed in this paper. In method 1, solid wood panels were formed, with the resulting lamellas glued only along the edge to form panels with the desired width. Method 2 produced three-layer solid wood panels with mutually perpendicular wood fiber arrangements. They are similar to CLT (cross laminated timber- EN 16351, 2021). Method 3 yields a bonded panel of waste pieces, which is used as a middle layer in the manufacture of veneered panels. This avoids the negative impact of defects on the aesthetic and mechanical properties of the final product.

Polyvinyl acetate (PVAc) adhesive Jowacoll 103.06 produced by Jowat was applied, while the amount of applied glue was 200-220 g/m². The time required to cure the test specimens was 3 hours. PVA is a fast-setting wood adhesive, suitable for cold and hot bonding of wood products with high adhesive ability of 450 N/mm². The combination and gluing of the manufactured elements were achieved with laboratory clamps and presses.

Depending on the performance, quick-setting and other two-component adhesives can be used (Lubis et al., 2022; Savov et al., 2022). The pressing pressure applied was 0.1 - 0.2 MPa. The finished panel dimensions were 650 mm \times 650 mm \times 38 mm. In method 3, European beech (Fagus sylvatica L.) wood veneers with a thickness of 1.1 mm were used. In Method 4, the specimens were tested only in the direction along the grain of the outer veneer sheets. The aim was to find out what effect the application of a beech wood veneer with the thickness of only 1.1 mm would have on the strength of the final product. In the intermediate/inner layer, the direction of the wood grains is as in Method 1 (\perp). When testing several specimens in a direction perpendicular to the wood grain of the beech face veneer, the values did not differ significantly from those of Method 1 (II). Splitting occurs in the face veneer sheet under perpendicular loading. Figure 2 depicts the panels made from waste pieces in this study.

The produced engineered wood panels were conditioned until a constant mass was reached prior to physical and mechanical properties evaluation. For physical properties, density and water content of the panels were determined in accordance with BDS EN 323: 2001 and BDS EN 326-1: 2001.

The purpose of the density test was to investigate the effect of the knots and amount of glue on the density of the final product.

The mechanical properties were evaluated at the facilities of the University of Forestry, Sofia. A universal testing machine HECKERT FP 10/1 (Germany) with the test range (maximum strength) = 10000 N was used. The modulus of elasticity in static bending, as well as the bending strength parallel and perpendicular to the grain, was evaluated based on BDS EN 408 + A1: 2012. The deflection to complete destruction of the experimental wood was also observed.

The obtained data were processed using a T-test. The results were compared with values from tests of solid natural Scots pine wood, with the presence of knots (Mirski *et al.*, 2020a; Mirski *et al.*, 2020b; Wieruszewski *et al.*, 2022).

3 RESULTS AND DISCUSSION

3. REZULTATI I RASPRAVA

3.1 Physical properties of engineered wood panels

Fizička svojstva kompozitnih drvnih ploča za graditeljstvo

The moisture content of the engineered wood panels, fabricated from Scots pine timber pieces with defects varied from 8 % to 10 %. The values obtained for the density of the panels produced using the different methods are given in Table 2.

The average density of the panels obtained by method 1 was 581 kg/m³. According to published data, the density of Scots pine wood is approximately 520

 Table 2 Density of panel made using different methods

 Tablica 2. Gustoća ploča proizvedenih različitim metodama

Type of panel	Method 1	Method 2	Method 3
Vrsta ploče	Metoda 1	Metoda 2	Metoda 3
Density Gustoća	581 kg/m ³	607 kg/m ³	643 kg/m ³

kg/m³ (Aleinikovas and Grigaliūnas, 2006; Bluskova, 2009; Montero *et al.*, 2011; Konofalska *et al.*, 2021; Roszyk *et al.*, 2020). The 11.7 % increase in density was attributed to the presence of a large number of knots with large diameters and higher density in the experimental wood. In method 2, the average density of the panels was 607 kg/m³. The increment in density was due to the fact that the three-layer glued solid wood panel contained a high concentration of knots. The panels obtained by method 3 exhibited the highest density value of 643 kg/m³, mainly due to the presence of a large number of knots and adhesive used to bond beech veneers on the face and back of the panels.

3.2 Mechanical properties of engineered wood panels

3.2. Mehanička svojstva kompozitnih drvnih ploča za graditeljstvo

The results obtained for the bending strength of the panels fabricated by the different methods are presented in Figure 3. The panels obtained by method 1 were tested in two directions: perpendicular to the grain (\perp) and parallel to the grain (II).

Bending strength of the panels made by method 1, in direction perpendicular to the grain, ranged from 4.1 N/mm² to 5.6 N/mm², with an average value of 4.9 N/mm². It was observed that none of the tested specimens were destroyed in the adhesive joint area, but rather in the wood or at places with numerous defects. On the other hand, bending strength, in direction parallel to the grain of the panels, produced by method 1, ranged between 7.5 N/mm² and 10.6 N/mm², with an average value of 8.8 N/mm². The majority of test specimens were destroyed in the defective areas rather than at the adhesive joint site. The test specimens were destroyed in areas with numerous defects such as rotten knots, fibre twisting, oblique layering, etc.

 Table 3 Statistical data of bending strength values of engineered wood panels produced in this research

 Tablica 3. Statistički podatci o vrijednostima čvrstoće na savijanje proizvedenih kompozitnih drvnih ploča za graditeljstvo

	Ben	ding stre savija	e ngth / (<i>inje</i> , N/1		a na
Type of panel Vrsta ploče	\bar{x}	$S_{\rm x}^2$	$S_{\rm x}$	m _x	$p_{\rm x}$
Method 1 ($^{\perp}$) metoda 1 ($^{\perp}$)	4.9	0.233	0.483	0.171	3.5
Method 1 (II) metoda 1 (II)	8.8	1.161	1.077	0.381	4.3
Method 2 <i>metoda 2</i>	15.2	30.671	5.538	1.958	12.9
Method 3 metoda 3	28.6	1.738	1.318	0.466	1.6

Bending strength of the panels, produced by method 2, varied in the range from 9.1 N/mm² to 22.2 N/mm², with an average value of 15.2 N/mm². The obtained values for bending strength and modulus of elasticity for this type of panels are significantly lower (\approx 30-40 %) compared to the literature data as well as to standard EN 16351:2021 (Buck et al., 2016; Sikora et al., 2016; Mohd Yusof et al., 2019). This lowering of the mechanical indicators is due to the fact that in the examined panels there is a large number of cut pieces, due to their size, the absence of finger joints, and also the presence of numerous defects. The density of the boards obtained by method 2 is very close to that of CLT, despite the presence of a large number of knots in the wood. Panels fabricated by method 3 exhibited the highest bending strength values, ranging from 27.0 N/mm² to 30.1 N/ mm², with an average bending strength of 28.6 N/mm².

Bending strength values were calculated with an accuracy index of p_x (Table 3). For panels made by using method 1 ($^{\perp}$, II) and method 3, p_x was less than 5

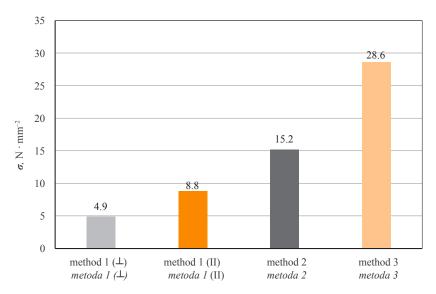


Figure 3 Bending strength of laboratory panels fabricated from Scots pine timber by-products **Slika 3.** Čvrstoća na savijanje laboratorijskih ploča proizvedenih od nusproizvoda borovine

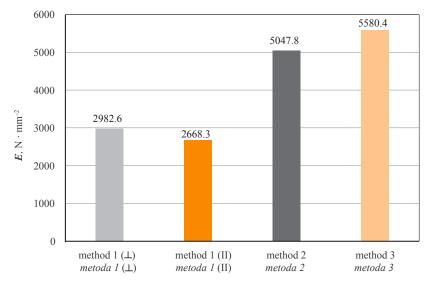


Figure 4 Modulus of elasticity (*E*, N/mm²) of laboratory panels fabricated from Scots pine timber by-products **Slika 4.** Modul elastičnosti (*E*, N/mm²) laboratorijskih ploča proizvedenih od nusproizvoda borovine

%, i.e. the results obtained were statistically significant. With regards to the panels fabricated by method 2, a large scatter relative to the mean was observed (p_x = 12.9 %). This could be attributed to the three layers of thickness, as well as splicing in the width of the waste pieces. This was done with spliced lengths that were randomly overlapped.

A graphical representation of the results obtained for the modulus of elasticity and deflection of the engineered wood panels, fabricated in this research, is presented in Figure 4 and Figure 5.

According to the data presented in Figures 4 and 5, the modulus of elasticity and flexural strength are directly related. According to method 1 (\perp), the exper-

imental wood was destroyed when the deflection reached 4.3 mm at a limit value of 354 N. Method 1 (II) test specimens were destroyed at a limit value of 758 N and deflected of 7.1 mm. The results of methods 2 and 3 for the destruction of test specimens were 1233 and 2659 N, respectively. Methods 2 and 3 calculated deflection values of 7.2 and 8.4 mm, respectively.

After conducting a *T*-test, it was found that for the modulus of elasticity (E) and bending strength (σ), method 1 (\perp) and method 1 (II), there is a statistically significant difference between the values. Accordingly, *p*-value = 0.026. Therefore, the direction of testing has an influence on the modulus of elasticity. This is also true for bending strength, *p*-value = 0.000006. In meth-

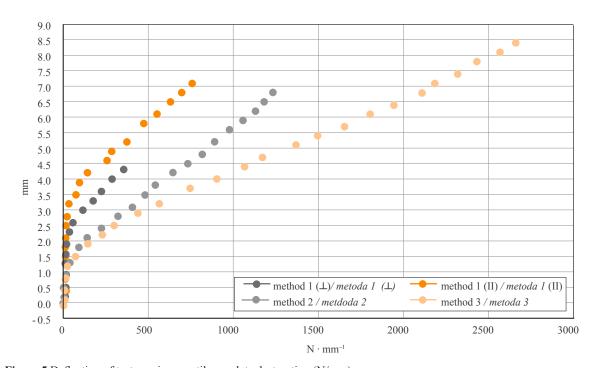


Figure 5 Deflection of test specimen until complete destruction (N/mm) **Slika 5.** Otklon ispitnog uzorka do potpunog pucanja (N/mm)

od 2 and method 3, there is no difference in modulus of elasticity (*E*, *p*-value = 0.035), but there is a difference in bending strength (σ , *p*-value = 0.000172).

4 CONCLUSIONS

4. ZAKLJUCAK

The present study has a significant potential for future research, as industrial practice produces a huge amount of waste wood in the form of large pieces.

The possibilities of using three gluing methods in the production of engineered wood panels from Scots pine timber by-products with defects were demonstrated in this research. The method involving veneering of the panel surfaces demonstrated the best mechanical properties. A promising alternative use for this wood waste is also to assemble the defective wood pieces into a CLT-like structure. There is a significant amount of wood loss, varying from 65 % to 70 % in the production of engineered wood, in the form of sawdust, shavings, small and large residues. The rationale for using the described methods of manufacturing engineered wood panels is due to the following: the solid wood materials intended for the production of engineered wood were dried to a final humidity of 8-9 %, which is also the humidity of the waste pieces used; the lamellas were treated with a four-sided planer prior to format cutting, which is a requirement for the same cross-sections of the waste pieces; a technological opportunity is created by the correct geometric shape of the obtained waste pieces, which makes gluing easier by smoothing the end-toend butt joint. Another technological operation is saved when splicing waste pieces lengthwise. Moreover, the absence of finger joints reduces processing time, wood and adhesive consumption. The factors described above, as they save many technological operations, are a significant prerequisite for the use of this raw material in the production of glued wood products. The determined physical and mechanical properties allow the use of the laboratory-produced panels in the furniture industry and even as a construction material. The bending strength results, ranging from 4.9 to 28.6 N/mm², were comparable to some of the most commonly used wood-based panels. In this case, products that can largely replace conventional materials in industry are obtained. These products can be widely used when high mechanical properties are not required, including table tops, partition walls, cladding, door frames, etc. The industrial reuse of timber by-products for manufacturing engineered wood panels with acceptable exploitation properties could greatly contribute to maximize the resource efficiency and enhance the competitiveness of wood industry companies.

Acknowledgements - Zahvala

The authors wish to thank "SREDNA GORA AD" company for providing the raw materials.

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Alma Žiga¹, Josip Kačmarčik² **Plywood Cantilever Deflection: Experimental, Analytical and FEM Approach**

Progib konzole od furnirske ploče: eksperimentalni, analitički i FEM pristup

ORIGINAL SCIENTIFIC PAPER

Izvorni znanstveni rad Received – prispjelo: 13. 7. 2022. Accepted – prihvaćeno: 4. 11. 2022. UDK: 691.116 https://doi.org/10.5552/drvind.2023.0053 © 2023 by the author(s). Licensee Faculty of Forestry and Wood Technology, University of Zagreb. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license.

ABSTRACT • In this paper, the elastic behaviour in bending of three-layer plywood cantilever beams is analysed. Deflections of straight and half-circle cantilevers, loaded with a force at free end is determined experimentally and calculated using analytical and finite element method approach. The analytical calculation of deflection for the strait cantilever is obtained using a transformed cross section. The deflection of half-circle cantilever is determined by the classical laminated plate theory and Castigliano's theorem. Loads and cantilever dimensions are varied in the study using the design of experiment. The deflection regression models for straight and semicircular plywood cantilevers are obtained from the experimental results. Analytically and numerically determined deflections of strait and half-circle cantilevers show very good agreement. Experimentally recorded deflections are approximately 30 % higher than analytical values. Stiffness properties and deflection values are influenced by direction of fibres in the outer layers of a three-layer plywood beam.

KEYWORDS: *beam deflection; design of experiment; half-circle cantilever; orthotropic composite material; poplar plywood; straight cantilever*

SAŽETAK • U radu je analizirano elastično ponašanje konzolnih greda od troslojne furnirske ploče pri savijanju. Progibi ravnih i polukružnih konzola opterećenih silom na slobodnom kraju određeni su eksperimentalno te izračunani analitičkim postupkom i metodom konačnih elemenata. Analitički proračun progiba ravne konzole dobiven je primjenom transformiranog presjeka. Progib polukružne konzole određen je klasičnom teorijom lamelirane ploče i Castiglianovim teoremom. Opterećenja i dimenzije konzola u istraživanju variraju u skladu s postavkom eksperimenta. Iz eksperimentalnih rezultata dobiveni su regresijski modeli progiba za ravne i polukružne konzole od furnirske ploče. Analitički i računski utvrđeni progibi ravnih i polukružnih konzola vrlo se dobro podudaraju. Eksperimentalno zabilježeni progibi približno su 30 % veći od analitički utvrđenih. Na svojstva krutosti i vrijednosti progiba grede od troslojne furnirske ploče utječe smjer vlakanaca u njezinim vanjskim slojevima.

KLJUČNE RIJEČI: progib grede; postavka eksperimenta; polukružna konzola; ortotropni kompozitni materijal; furnirska ploča od topolovine; ravna konzola

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1 INTRODUCTION

1. UVOD

Wood has anisotropic mechanical properties (Bodig and Jayne, 1993; Stark et al., 2010), but when the principal axes coincide with the orientation of the grain, wood can be considered orthotropic and, in certain cases, transversely isotropic. Plywood is a woodbased laminate where wood material is adhesively bonded together. The macroscopic structure of plywood is created by gluing alternately arranged, thin cross-bands, i.e. veneers of various thickness. At microscopic level, a veneer is a lamina strengthened by cellulose fibres and surrounded by a matric mainly composed of lignin. Analytical calculations of stiffness properties of plywood, as symmetric laminate, can be performed using the classical laminated plate theory (CLPT). A study (Merhar, 2020) had shown that the laminate theory, which was well established and applied in the world of synthetic composites, could also be applied to plywood composites. The influence of fibre reinforcement and wood species on the physical and mechanical properties of veneer plywood as well as on laminated veneer lumber was also investigated (Brezović et al., 2003; Bal, 2014; Sikora et al., 2019). The CLPT and the finite element method (FEM) were used in a study (Makowski, 2019) to determine stress distribution in individual layers of 18 mm thick beech plywood, in the 3-point flexural bending test. Analysis (Bal and Bektab, 2014) showed that the effects of tree species, direction of load, and type of adhesive on flexural properties were significant, and it was determined that the effect of the type of adhesive is based on the density of the plywood. The influence of the veneer composition on the mechanical properties of rectangular and curved form of laminated wood was investigated by FEM and transformed cross section method in (Hajdarevic et al., 2017). A research (Labans et al., 2010) determined elastic properties for individual veneer specimens in order to evaluate the input data for plywood products analyses using FEM. The study (Merhar, 2021) emphasised the correct input for FEM. Veneer is usually produced by peeling, which exerts large bending deformations in the tangential direction, resulting in local cracks. Because of the small cracks, the tensile strength in the tangential direction is much lower (up to three-times lower) than the tensile strength of solid wood. When applying the laminate theory to plywood, the drawback could be the lack of input data as well as the variability of the data for particular tree species (the variability of wood properties in the main directions can be up to 10 %). A study (Wilczyński and Warmbier, 2012) determined the elastic moduli of veneers assembled in pine and beech plywood panels, where the effects of the resin type and the number of

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veneer plies in the plywood were evaluated. It was concluded that glue line (with Young's modulus ranging from 1000 to 10 000 MPa) had negligible influence on Young's modulus of the veneer in the grain direction, while in perpendicular direction to the grain, Young's modulus of veneer had 19.4 % lower value for maximum glue modulus. It is pointed out that the properties of veneers assembled in plywood differed from those of the wood from which the veneers were made. In the research (Tsen, 2013), bending strength (MOR) and modulus of elasticity (MOE) of red seraya structural plywood were obtained by EN 310 thee-point bending and EN 789 four-point bending test. Also MOR and MOE were obtained by FEM simulation. The FEM results were 39 % and 14 % higher than those from EN 310 and EN 789 experiments, respectively.

This study aims to investigate elastic properties and behaviour of plywood cantilever beams - strait and semicircle, and to provide and compare analytical, numerical and experimental calculation methods for the deflection at the free end, in the point where concentric force is applied. The motivation for the research was the problem of how to calculate deflection in the design of parts of toys and souvenirs made using CO_2 laser cutter from 4 mm plywood (Žiga *et al.*, 2018; Žiga and Begić-Hajdarević, 2021), which were subjected to bending. However, the calculation methods presented here could be implemented in the design of various plywood structures.

The analytical calculation of the deflection for strait cantilever was done using transformed cross section, but semicircle cantilever required CLPT approach and the use of Castigliano's theorem. As far as the authors were aware, the analytical calculation for deflection of semicircle, horizontally layered cantilever could not be found anywhere in the literature. The numerical calculations were done in FEM software, where cantilevers and boundary conditions were modelled and deflections at the free end were obtained. The deflections of plywood specimens were experimentally measured on coordinate measuring machine (CMM) and the results were used to obtain the power regression model. The design of the experiment was implemented to obtain the dimensions of cantilever beams and load values for the research. All the results were compared and analysed in the paper.

2 MATERIALS AND METHODS 2. MATERIJALI I METODE

2.1 Plywood material and cantilever beam configurations

2.1. Furnirska ploča i konfiguracija konzolnih greda

The deflection of straight (Figure 1a) and halfcircle cantilevers (Figure 1b) from plywood is investi-

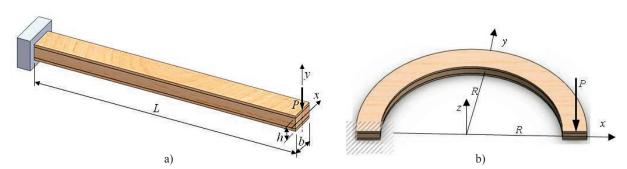


Figure 1 Plywood cantilever beam configurations: a) straight, b) half-circle (P type) **Slika 1.** Konfiguracija konzolne grede od furnirske ploče: a) ravna, b) polukružna (tip P)

gated in the paper. The load at the end, the length of the straight or the radius of the half-circle cantilever and the cantilever width were varied, using the design of experiment.

Two types of poplar plywood sheets consisting of three veneer layers 4 mm thick were used: the first type with the central veneer 2.3 mm thick and the outside veneers 0.8 mm thick; and the second type, with three equal veneer thickness of 1.3 mm. Both sheets were acquired commercially, at a local store, with density of 410 kg/m³ and with formaldehyde emission E1, measured in accordance with DIN EN 16516. This and similar types of plywood are often used for making toys and souvenirs, due to its characteristics such as low price and low density, which makes it easy to cut on small CO₂ laser cutter (80-120 kW power) and provides low weight of parts. Cantilever specimens from the first type of plywood, with central veneer thicker than outer ones, were cut so that the fibres in the outer layer were perpendicular to the cantilever span (label P, figure 1). Figure 2 shows plywood laser cut where a central veneer has fibres parallel to the cutting plane and outer veneer fibres are perpendicular to the cutting plane. Cantilever specimens from the second type of plywood, with the equal veneer thickness, were cut so that the outer layer fibres were longitudinal to the span of cantilever (label L).

Poplar veneers are orthotropic material with nine independent constants of material. In the analytical calculation and FEM analysis, the properties for poplar

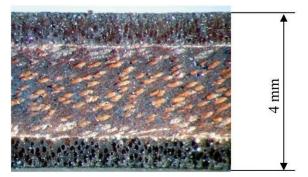


Figure 2 Laser-cut of poplar plywood, 4 mm in thickness **Slika 2.** Laserski izrezana furnirska ploča od topolovine debljine 4 mm

veneer were taken from the paper (Brezović, *et al.*, 2003): $E_1 = 9600$ MPa, $E_2 = 420$ MPa, $E_3 = 880$ MPa, $v_{12} = 0.39$, $v_{23} = 0.33$, $v_{13} = 0.32$, $G_{12} = 660$ MPa, $G_{13} = 720$ MPa, $G_{23} = 110$ MPa. Glue layer thickness can be observed in Fig. 2. It is a white layer, partially absorbed by adjacent wood layers. In numerical and analytical model, the average value of thickness was set to be 0.05 mm. As there were no data about glue line modulus, it was assumed that the value of this modulus for formaldehyde-based adhesives could range from 1000 to 10000 MPa (Wilczyński and Warmbier, 2012). The lowest value of 1000 MPa was chosen. Poison ratio was set to 0.2 (Piao *et al.*, 2008).

2.2 Design of experiment 2.2. Postavke eksperimenta

Full orthogonal, three-factor plan (Figure 3), with 4 measurement repetitions in central plan point is implemented in the paper thus making $N = 2^k + n_0 = 2^3 + 4 = 12$ experimental runs. The 12 experimental runs are required for each of the two types of plywood fibre orientation and for two different geometries of cantilever beam, making it in total 48 experimental runs. The model factors for experiment are: x_1 – load (*P*), x_2 - length for straight cantilever (*L*) or radius for half-circle cantilever (*R*) and x_3 - width of cross-section

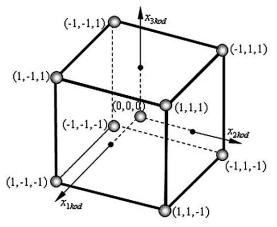


Figure 3 Location of experimental points of full orthogonal plan in hyper-space

Slika 3. Položaj eksperimentalnih točaka cijele ortogonalne projekcije u hiperprostoru

···· ··· ··· ··· ··· ··· ··· ··· ··· ·	· · I ·											
Exp. runs	1	2	3	4	5	6	7	8	9	10	11	12
$x_1(P)$	-1	1	-1	1	-1	1	-1	1	0	0	0	0
$x_2(L \text{ or } R)$	-1	-1	1	1	-1	-1	1	1	0	0	0	0
$x_3(b)$	-1	-1	-1	-1	1	1	1	1	0	0	0	0

Table 1	Experiment design matrix
Tablica	1. Matrica postavki eksperimenta

(*b*), see Figure 3. The experiment design matrix, for both investigated cantilevers, is presented in Table 1.

Levels of loads and dimensions are arbitrarily chosen to produce measurable deflection but also not to exert stresses above material strength. The cantilever dimensions also correspond to the dimensions of possible parts in mechanical toys and souvenirs (Žiga *et al.*, 2018). The level values for straight and half-circle cantilever of all factors are shown in Table 2. The basic level was chosen to be geometrical mean of the upper and lower level.

It can be observed from the experiment design matrix that five different specimen dimension configurations are needed. The five test specimens were lasercut for each of the two types of plywood fibre orientation. Figure 4 shows straight and half circle cantilever specimens with fibres in outer ply perpendicular to the span (P-type). On one side of the specimens, there is a wide segment for clamping, and on the other a segment with the narrow lateral grove for positioning the load. From the experimental results, power regression models that provide a relation between three independent variables (x_1, x_2, x_3) and the deflection as a dependent variable is determined. The power regression model for straight cantilevers is obtained using last squared method. The paper by Žiga *et al.* (2019) presents in detail how to determine coefficients of power model function.

2.3 Analytical method for cantilever deflection

2.3. Analitička metoda za izračun otklona konzole

2.3.1 Deflection of straight cantilever2.3.1. Otklon ravne konzole

The straight cantilever loaded with concentrated force at the end of the span (Figure 1a) is analysed. In the cross-section, the cantilever is a symmetrical composite laminate made of veneer sheets and adhesive layers, so the method of transformed cross section can be applied (Bodig and Jayne, 1993). In this method,

		Straight ca	antilever / Rav	na konzola	Half-circle cantilever / Polukružna konzola					
Factor / Faktor		Low level	Basic level	High level	Low level	Basic level	High level			
		Niža	Srednja	Viša	Niža	Srednja	Viša			
		vrijednost	vrijednost	vrijednost	vrijednost	vrijednost	vrijednost			
$\mathbf{I} = \mathbf{I} / \mathbf{I} = \mathbf{I} $	Ν	0.5	0.736	1.01	0.5	0.736	1.01			
Load / opterećenje (P)	x_1	-1	0	+1	-1	0	+1			
Length (L) or Radius (R)	mm	80	100	120	26	34	42			
duljina (L) ili radijus (R)	<i>x</i> ₂	-1	0	+1	-1	0	+1			
XX7. 1.1 (Y · · (1)	mm	6	8	10	7	8	9			
Width / širina (b)	x_{2}	-1	0	+1	-1	0	+1			

Table 2	Levels of model factors
Tablica	2. Razine faktora modela



Figure 4 P-type specimens: a) straight cantilever, b) half-circle cantilever **Slika 4.** Uzorci tipa P: a) ravna konzola, b) polukružna konzola

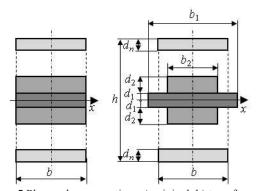


Figure 5 Plywood cross section: a) original, b) transformed **Slika 5.** Presjek furnirske ploče: a) izvorni, b) transformirani

only one reference value of the elasticity modulus e.g. the outer lamina elasticity modulus E_n , in the direction of the cantilever span, is used in the deflection calculation. The transformed cross-section (Figure 5) is made so that lamina other than the reference one has a transformed width defined by

$$b_i = b\left(\frac{E_i}{E_n}\right) \tag{1}$$

Where *b* is the original width of the lamina, E_i is the lamina modulus of elasticity in the direction of span, E_n is the modulus of elasticity of the reference lamina. Thicknesses of all lamina do not change; the original values are used. The moment of inertia I_x of the transformed cross section with *n* lamina in one symmetrical half of the cross section (Figure 5) is calculated with:

$$I_{x} = \frac{2 \cdot b}{E_{n}} \sum_{i=1}^{n} \left[\frac{E_{i} \cdot d_{i}^{3}}{12} + E_{i} \cdot d_{i} \left(\sum_{j=1}^{i-1} d_{j} + \frac{d_{i}}{2} \right)^{2} \right]$$
(2)

Where d_i is the thickness of the *i*-lamina.

After the transformed cross section is defined, the deflection of the cantilever is calculated with:

$$\delta = \frac{P \cdot L^3}{3 \cdot E_n \cdot I_x} \tag{3}$$

Where *P* is the load at the end of cantilever and *L* is the length of cantilever.

2.3.2 Deflection of half-circle cantilever2.3.2. Otklon polukružne konzole

The deflection of the half-circle cantilever (Figure 1b) at the point of application of the force P and in the direction of the force will be determined using procedures from Horibe and Mori, 2015; Dahlberg, 2004 and Žiga *et al.*, 2018.

Figure 6a shows cross-section of the beam situated at angle φ . The force *P* is normal to the *xy* plane. The bending moment M_b and torsional moment M_t are acting on this cross-section. The shear force is omitted in the figure since its influence on beam deflection can be neglected. For positive values of *x* and *y*, angle φ varies between 0 and $\pi/2$, so both cos φ and sin φ have positive values. From the moment equilibrium equations about *x* and *y* axes, the moments M_b and M_t are:

$$M_{b} = -P \Big[y \cos \varphi + (R - x) \sin \varphi \Big] = -P \cdot R \sin \varphi \qquad (4)$$

$$M_{t} = P \lfloor (-R+x) \cos \varphi + y \sin \varphi \rfloor = P \cdot R (1 - \cos \varphi)$$
(5)

The strain-stress relation for unidirectional laminate (Figure 6b) in its plane (Jones, 1999; Cuntze, 2015) is:

$$\begin{cases} \boldsymbol{\varepsilon}_{x} \\ \boldsymbol{\varepsilon}_{y} \\ \boldsymbol{\gamma}_{s} \end{cases} = \begin{bmatrix} \boldsymbol{S}_{xx} & \boldsymbol{S}_{xy} & \boldsymbol{S}_{xs} \\ \boldsymbol{S}_{yx} & \boldsymbol{S}_{yy} & \boldsymbol{S}_{ys} \\ \boldsymbol{S}_{sx} & \boldsymbol{S}_{sy} & \boldsymbol{S}_{ss} \end{bmatrix} \cdot \begin{cases} \boldsymbol{\sigma}_{x} \\ \boldsymbol{\sigma}_{y} \\ \boldsymbol{\tau}_{s} \end{cases}, \tag{6}$$

Where S_{ij} are the transformed compliance coefficients for in-plane behaviour; ε_x , ε_y are the in-plane normal strains; γ_s is the in-plane shear strain; σ_x , σ_y are the in-plane normal stresses; τ_s is the in-plane shear stress. The *xyz* coordinate system in Figure 6b is assumed to have its origin in the middle of the laminate thickness.

Applying classical laminated plates theory, the following relationships can be obtained (Daniel *et al.*, 2006):

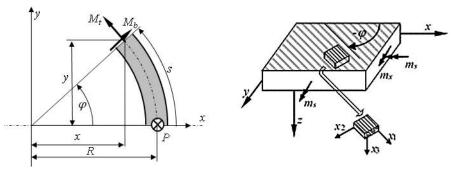


Figure 6 a) Moment equilibrium b) Moments acting on composite cantilever segment **Slika 6.** a) Momenti ravnoteže, b) momenti koji djeluju na segment kompozitne konzole

$$\begin{cases} \sigma_x \\ \sigma_y \\ \tau_s \end{cases} = z \cdot \frac{12}{h^3} \begin{cases} m_x \\ m_y \\ m_s \end{cases}$$
(8)

Where m_x , m_y are the bending moments per unit length; m_s is the twisting moment per unit length; κ_x , κ_y , are the bending curvatures of the middle surface; κ_s is the twisting curvature of the middle surface.

Strain energy per unit volume for linearly elastic behaviour induced by bending and twisting is (Mujika and Mondragon, 2003):

$$u_{M} = \frac{1}{2} \Big(\sigma_{x} \cdot \varepsilon_{x} + \sigma_{y} \cdot \varepsilon_{y} + \tau_{s} \cdot \gamma_{s} \Big)$$
(9)

Substituting stress–strain relations from Equation (6):

$$u_{M} = \frac{1}{2} \left(S_{xx} \sigma_{x}^{2} + S_{yy} \sigma_{y}^{2} + S_{ss} \tau_{s}^{2} \right) + S_{xy} \sigma_{x} \sigma_{y} + S_{xs} \sigma_{x} \tau_{s} + S_{ys} \sigma_{y} \tau_{s}$$
(10)

Elastic strain energy stored in the beam is:

$$U = \int_{V} u_{M} dV \tag{11}$$

Considering 3 plies of veneer and 2 plies of glue line, angle ϕ of fibre direction, bending and torsional moments as:

$$m_x = \frac{M_b}{b}, m_y = 0, m_x = -\frac{M_t}{2b}$$
 (12)

The strained energy is:

$$U = \left(\frac{12}{h^3}\right)^2 \cdot \left(\frac{1}{2} \int_{V} \left(\frac{M_b}{b}\right)^2 bRd\varphi \cdot S_{xx} z^2 dz + \frac{1}{2} \int_{V} \left(-\frac{M_t}{2b}\right)^2 bRd\varphi \cdot S_{xs} z^2 dz - \frac{1}{\sqrt{\frac{M_b}{b}}} \frac{M_t}{2b} bRd\varphi \cdot S_{xs} z^2 dz \right)$$
(13)

Where

$$\int_{0}^{h} S_{ij} z^{2} dz = d_{ij} = \frac{1}{3} \sum_{k=1}^{5} S_{ij}^{k} \left(z_{k}^{3} - z_{k-1}^{3} \right)$$
(14)

Transformed compliance coefficients are expressed with compliance coefficients in fibber direction coordinate system:

$$c = \cos \varphi s = \sin \varphi$$

$$S_{xx} = c^4 S_{11} + s^4 S_{22} + (2S_{12} + S_{66})c^2 s^2 \qquad (15)$$

$$S_{xs} = (2S_{11} - 2S_{12} - S_{66})c^3 s - (2S_{22} - 2S_{12} - S_{66})cs^3$$

$$S_{ss} = (4S_{11} + 4S_{22} - 8S_{12})c^2 s^2 + (c^2 - s^2)S_{66}$$

Compliance coefficients in fibber direction or principal material coordinate system (Figure 6b) are:

$$S_{11} = \frac{1}{E_1} S_{22} = \frac{1}{E_2} S_{66} = \frac{1}{G_{12}} S_{12} = S_{21} = -\frac{V_{12}}{E_1} = -\frac{V_{21}}{E_2}$$
(16)

Using Castigliano's theorem, the deflection of the cantilever end, at the load *P* can be calculated with:

$$\delta = \frac{\partial U}{\partial P} = \left(\frac{12}{h^3}\right)^2 \cdot \left(\begin{array}{c} \frac{1}{b} \int_0^{\pi} M_b \frac{\partial M_b}{\partial P} d_{xx} Rd\varphi + \\ + \frac{1}{4b} \int_0^{\pi} M_t \frac{\partial M_t}{\partial P} d_{xs} Rd\varphi - \\ - \frac{1}{2b} \int_0^{\pi} \left(\frac{\partial M_b}{\partial P} M_t + M_b \frac{\partial M_t}{\partial P}\right) d_{xs} Rd\varphi \right)$$
(17)

2.4 Cantilever deflection using FEM 2.4. Otklon konzole dobiven metodom

konačnih elemenata

Numerical analysis is performed using commercial software SolidWorks 2020 and its Simulation module based on the finite element method (FEM). Five FE models of straight beam cantilever and five models of halfcircle beam cantilever were modelled with dimensions that are defined by design of experiment (Tables 1 and 2) and to be geometrically identical to the real specimens. Models were positioned in Cartesian co-ordinate system where the directions of co-ordinates coincide with three main directions of material axes. Composite material was defined in software using material properties from literature (see chapter 2.1)

The boundary conditions in FEM analysis were defined to correspond as much as possible to the actual experimental conditions. Thus, the results achieved by numerical method could be compared to the experiment results. The left segment for clamping was fixed and force was applied on the line between lateral grooves on the right segment for load. The automatic meshing from Solidworks was used with the fine mesh quality setting in order to obtain accurate results. Figure 7a shows an example of FEM model of straight cantilever with P-type fibre orientation (length 80 mm and

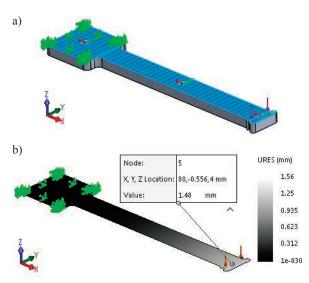


Figure 7 Straight cantilever FEM (L = 80 mm, b = 10 mm, P = 1.01 N), P-type: a) boundary condition, b) deflection result

Slika 7. Ravna konzola FEM (L = 80 mm, b = 10 mm, P = 1,01 N), tip P: a) rubni uvjet, b) otklon

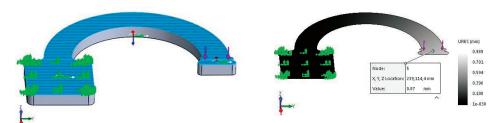


Figure 8 Half-circle cantilever FEM (R = 26 mm, b = 9 mm, P = 1.01 N), P-type: a) boundary condition, b) deflection result **Slika 8.** Polukružna konzola FEM (R = 26 mm, b = 9 mm, P = 1.01 N), tip P: a) rubni uvjet, b) otklon



a)

b)

Figure 9 a) CMM "ZEISS CONTURA G2", b) Measurement of deflection on CMM **Slika 9.** a) CMM "ZEISS CONTURA G2"; b) mjerenje otklona na CMM-u

width 10 mm). Figure 8a shows an example of FEM model of half-circle cantilever with P-type fibre orientation (radius 26 mm and width 9 mm). Figure 7b and Figure 8b show deflection results after finished FEM simulations for the above-mentioned examples.

2.5 Experimental study

2.5. Eksperimentalno istraživanje

CMM "ZEISS CONTURA G2" was used (Figure 9a) for the experimental measurements of plywood cantilever deflections. The CMM was equipped with the continuous scanning probe system "VAST XT". The measuring uncertainty of the CMM is 1.8 µm, which makes it a very accurate measuring device. For the measurement of the defections, the cantilevers specimens were positioned and clamped on the CMM table. The specimens were fixed between two steel parts fastened with bolts. The parts were fixed on the CMM table with magnetic V-block (Figure 9b). At the free end of the specimens, the load was applied by hanging an appropriate weight made from steel nuts (Figure 9b). The deflection of the cantilever was measured as a difference between vertical coordinate reading of the CMM before and after loading. The CMM probing of the cantilever end point where load was applied, before and after loading, was done manually.

3 RESULTS AND DISCUSSION 3. REZULTATI I RASPRAVA

3.1 Results for straight cantilever

3.1. Rezultati za ravnu konzolu

For straight cantilevers, analytical, numerical, experimental and regression model results values, for the end-point deflections for the P-type and L-type fibber orientation, respectively, are presented in Table 3 and 4. The last row shows deviation in results between en experimental and analytical values. Figures 10a and 10b show the above mentioned deflection values on charts.

Analytical and numerical deflection values are very close to each other. Differences are visible only in the second decimal place. These values are, on average, 30 % lower than deflection recorded by the experiment. The differences are lower for the greater cantilever length due to slope contribution to a deflection value.

For P-type straight cantilever, the regression model is:

$$S_{P} = 0.009857 \cdot P^{1.04} \cdot L^{1.8486} \cdot b^{-1.0629}$$
(18)

The regression model for L-type straight cantilever is:

$$\delta_{I} = 0.003132 \cdot P^{1.0787} \cdot L^{1.7658} \cdot b^{-0.9977} \tag{19}$$

For the evaluation of the models, statistical data analysis has been used. Some results are presented in Table 5. The coefficient of determination (R^2), adjusted

Exp. run	1	2	3	4	5	6	7	8	9	10	11	12
y_{exp} , mm	2.40	4.77	5.22	10.72	1.31	3.23	3.06	5.78	3.88	3.79	3.55	3.74
y _{mod} , mm	2.36	4.88	5.15	10.62	1.31	2.71	2.86	5.90	3.88	3.88	3.88	3.88
y_{an} , mm	1.22	2.47	4.13	8.34	0.73	1.48	2.48	5.00	2.64	2.64	2.64	2.64
y _{num} , mm	1.22	2.44	4.10	8.28	0.73	1.48	2.46	4.98	2.61	2.61	2.61	2.61
$y_{exp} - y_{an}$, %	49.0	48.2	20.9	22.2	44.0	54.1	19.0	13.4	32.0	30.4	25.7	29.5
Y _{exp}												

 Table 3 Results for P-type straight cantilever beam

 Tablica 3. Rezultati ispitivanja ravne konzolne grede tipa P

 Table 4 Results for L-type straight cantilever beam

 Tablica 4. Rezultati ispitivanja ravne konzolne grede tipa L

Exp. run	1	2	3	4	5	6	7	8	9	10	11	12
y_{exp} , mm	0.47	1.32	1.08	2.53	0.42	0.56	0.62	1.45	0.99	1.21	0.95	1.10
y_{mod} , mm	0.57	1.22	1.17	2.49	0.34	0.73	0.70	1.49	0.96	0.96	0.96	0.96
y _{an} , mm	0.29	0.59	0.98	1.97	0.17	0.35	0.59	1.18	0.62	0.62	0.62	0.62
y _{num} , mm	0.29	0.59	0.98	1.98	0.17	0.35	0.59	1.19	0.63	0.63	0.63	0.63
$\frac{y_{\rm exp} - y_{\rm an}}{y_{\rm exp} - y_{\rm an}}, 0/0$	38.6	55.5	9.8	21.9	58.8	36.9	5.5	18.1	36.9	48.6	34.3	43.3
${\cal Y}_{exp}$												

 R^2 , standard error, regression sum of squares and the result of *F*-test were used for checking the adequacy of the model. The value of R^2 for the P-type of cantilever, where fibres in outer plies are perpendicular to the length, is quite high ($R^2 = 0.98$). It means that 98 % of cantilever deflection variability was caused by the variation of load *P*, length *L* and width *b* of the cantilever. Only 2 % of the variability was caused by some other factors, which were not included in the experiment (factors that were not controlled). For L-type of cantilever, 12 % of variability was caused by some other factors. It was explained by lower range of deflection (0.42 to 1.44 mm) in comparison to the first type of cantilever (deflection range is: 1.31 - 10.72 as shown in Figure 10a).

3.1 Results for half-circle cantilever

3.1. Rezultati za polukružnu konzolu

For half-circle cantilevers, analytical, numerical, experimental and regression model results values, for the end point deflections, are presented in Table 6. The last columns for each type of cantilever show differences between experimental and analytical values. The differences between analytical and numerical results

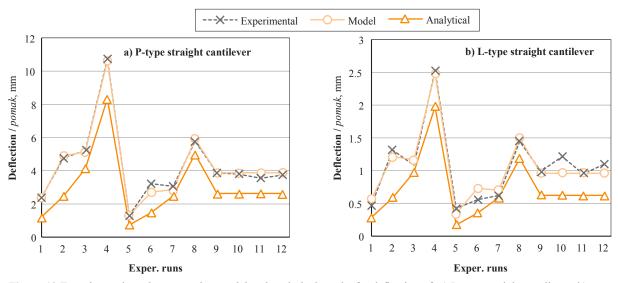


Figure 10 Experimental results, regression model and analytical results for deflection of: a) P-type straight cantilever, b) L-type straight cantilever **Slika 10.** Eksperimentalni rezultati, regresijski model i analitički rezultati otklona: a) ravne konzole tipa P, b) ravne konzole

tipa L

 Table 5 Data analysis for regression model of straight cantilever

 Tablica 5. Analiza podataka za regresijski model ravne konzole

Data / Podatak	P-type	L-type
R^2	0.9859	0.9137
Adjusted R ² / prilagođeni R ²	0.9807	0.8814
Standard error / standardna pogreška	0.0705	0.1785
Regression sum of squares / regresijski zbroj kvadrata	2.7893	2.6998
F _{value}	186.8291	28.2423
F _{table}	$F_0(3;8;0.05)=4.07$	$F_0(3;8;0.05)=4.07$
Model is adequate odgovarajući model	$Yes (F_0 < F)$	$\operatorname{Yes}(F_0 < F)$

are greater than for the straight cantilever, but still the results are very close. The greater differences could be explained by more complex analytical approach. Figure 11 shows graphical representation of deflection values for different methods for P and L type of cantilevers, respectively.

Power regression model for P-type, half-circle cantilever is:

$$\delta_{P} = 0.002107 \cdot P^{1.164} \cdot R^{3.3123} \cdot b^{-1.9705}$$
(20)

and for the L-type, half-circle cantilever is:

Table 6 Results for P-type straight cantilever beam

$$\delta_{I} = 0.00313 \cdot P^{1.0787} \cdot R^{1.766} \cdot b^{-0.9977}$$
(21)

Statistical data analysis is shown in Table 8. Values of R^2 for both types of cantilevers are quite high. Analytical values are, on average, 24 % lower than deflection recorded by the experiment for P-type of halfcircle cantilever and 25 % for L-type.

4 CONCLUSIONS

4. ZAKLJUČAK

Analytically and numerically determined deflections of strait and half-circle cantilever showed very good agreement. Experimentally recorded deflections

Tablica 6. Rezulta	ablica 6. Rezultati ispitivanja polukružne konzolne grede tipa P												
Exp. run	1	2	3	4	5	6	7	8	9	10	11	12	
y_{exp} , mm	0.80	1.88	5.24	10.68	0.61	1.45	2.41	5.21	3.53	3.66	3.34	3.32	
$y_{\rm mod}$, mm	0.99	2.24	4.84	10.97	0.60	1.37	2.95	6.68	2.90	2.90	2.90	2.90	
y_{an} , mm	0.75	1.52	3.18	6.41	0.59	1.18	2.47	4.99	2.17	2.17	2.17	2.17	
y_{num} , mm	0.72	1.45	3.14	6.34	0.46	0.93	1.93	3.90	1.76	1.76	1.76	1.76	
$\frac{y_{\exp} - y_{an}}{y_{\exp}}, \frac{9}{0}$	5.8	19.1	39.4	39.9	4.0	18.4	2.5	4.2	38.5	40.7	35.0	34.7	

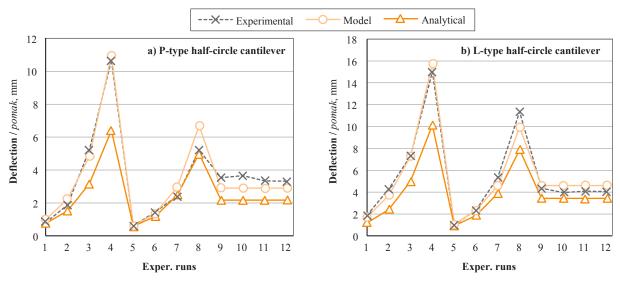


Figure 11 Experimental results, regression model and analytical results for deflection of: a) P-type half-circle cantilever, b) L-type half-circle cantilever

Slika 11. Eksperimentalni rezultati, regresijski model i analitički rezultati otklona: a) polukružne konzole tipa P, b) polukružne konzole tipa L

	-	51			0 1							
Exp. run	1	2	3	4	5	6	7	8	9	10	11	12
y_{exp} , mm	1.90	4.30	7.33	15.01	1.01	2.36	5.36	11.36	4.36	3.96	4.09	4.05
$y_{\rm mod}$, mm	1.71	3.71	7.28	15.76	1.08	2.33	4.58	9.92	4.60	4.60	4.60	4.60
y_{an} , mm	1.19	2.41	5.02	10.14	0.93	1.87	3.90	7.89	3.43	3.43	3.43	3.43
y _{num} , mm	1.20	2.43	5.14	10.37	0.84	1.71	3.60	7.29	3.29	3.29	3.29	3.29
$\frac{y_{\exp} - y_{an}}{y_{\exp}}, \frac{9}{0}$	37.3	44.1	31.5	32.5	8.3	20.7	27.2	30.6	21.3	13.4	16.1	15.3

Table 7 Results for	or L-type sti	raight cantilever beam
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Tablica 7. Rezultati ispitivanja polukružne konzolne grede tipa L

 Table 8 Data analysis for regression model of half-circle cantilever

 Tablica 8. Analiza podataka za regresijski model polukružne konzole

Data / Podatak	P-type	L-type
R^2	0.956	0.977
Adjusted R ² / prilagođeni R ²	0.939	0.968
Standard error / standardna pogreška	0.201	0.131
Regression sum of squares / regresijski zbroj kvadrata	6.915	5.839
$F_{ m value}$	57.327	28.2423
F _{table}	F0(3;8;0.05)=4.07	F0(3;8;0.05)=4.07
Model is adequate / odgovarajući model	Yes (F0 <f)< td=""><td>Yes (F0<f)< td=""></f)<></td></f)<>	Yes (F0 <f)< td=""></f)<>

were approximately 30 % higher than analytical values. The weakness of the analytical and numerical approach lied in the lack of knowledge of the input data for the wood tissue. The mean values were taken from the publicly available literature (Brezović *et al.*, 2003). Another weakness was the presentation of glue line and lack of its properties. Glue line was modelled with a uniform thickness and with the smallest modulus of elasticity.

This study showed that CLPT and Castigliano's theorem could be used to analytically obtain deflection of half-circle, multi-layer composite structure and this approach had never been presented in the publicly available literature as far as the authors were aware.

For strait, three-layer plywood cantilevers, direction of fibres in outer layers has great influence on stiffness properties and deflection values. When fibres in outer layers were perpendicular to the cantilever span (P type), the stiffness was lower and deflection was higher. Experimental deflection values for P type cantilevers were 1.31 to 10.72 mm. For L type cantilevers, the deflections were 0.42 to 1.44 mm.

For half-circle, three-layer plywood cantilevers, direction of fibres in outer layers has medium influence on stiffness properties and deflection values. When fibres in outer layers were perpendicular to the cantilever span, experimentally recorded values were 0.61 to 10.68 mm (P type). For L type, experimentally recorded values were 1.01 to 15.01 mm.

The implementation of design of experiment is proved as an efficient way to obtain regression model for the deflection of plywood beam. Model predicted values are very close to the experimentally obtained values. In the observed experiments, the length of straight cantilever (L) or the radius of half-circle cantilever (R) has the most influence on the beam deflection, more than the force (P) and width of the beam (b). The L or R have the standardized coefficient with the largest absolute value and this means that L or R are the most important independent variable in the regression model.

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The Influence of Different Wood Modifications on Pull-out Force of Rotary Welded Dowels

Utjecaj različitih vrsta modifikacije na izvlačnu silu rotacijski zavarenih moždanika

ORIGINAL SCIENTIFIC PAPER

Izvorni znanstveni rad Received – prispjelo: 27. 1. 2023. Accepted – prihvaćeno: 28. 2. 2023. UDK: 630*83; 684.4.058 https://doi.org/10.5552/drvind.2023.0087 © 2023 by the author(s). Licensee Faculty of Forestry and Wood Technology, University of Zagreb. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license.

ABSTRACT • The increasing use of modified wood justifies the intention to use it in joints made by rotary welding with dowels. Thermal modification, for example, increases the dimensional stability of wood by reducing its hygroscopicity and water absorption, but it is difficult to glue or to rotary weld due to the appearance of cracks. This can be avoided by changing the optimal welding parameters, which on the other hand directly affects the reduction of the pull-out force by more than 25 % depending on the type of the modified base. In the case of welding wooden dowels into wood modified with citric acid, the reduction in pull-out force is even greater. Due to the significant reduction, the use of this type of modification for wood welding is questionable. When citric acid modification is extended to the dowels, the dowel becomes a problem due to its lower tensile strength, which is even lower than the pull-out force of the welded dowel.

KEYWORDS: rotary welding; thermal modification; modification with citric acid; pull-out force; wooden dowels

SAŽETAK • Zavarivanje drva trenjem proces je kojim se spajaju dva ili više elemenata drva. Zbog trenja se stvara toplina koja omekša i rastali strukturu drva. Zavarivanje toplinski modificiranog drva zanimljivo je zbog sve veće primjene takvog drva u praksi. Toplinskom modifikacijom zbog smanjenja higroskopnosti i upijanja vode povećava se stabilnost dimenzija drva, ali zbog pojave pukotina u uzorku može biti otežano lijepljenje drva i rotacijsko zavarivanje moždanika. To se može izbjeći tako da se mijenjaju optimalni parametri zavarivanja, što izravno utječe na smanjenje izvlačne sile. Izvlačna sila moždanika zavarenoga u toplinski modificiranu podlogu smanjuje se za više od 25 %, ovisno o vrsti uzorka. Smanjenje izvlačne sile moždanika zavarenoga u drvo modificirano limunskom kiselinom čak je i veće. Zbog značajnog smanjenja izvlačne sile zavarenog moždanika upitna je primjena modifikacije limunskom kiselinom, kao i topinska modifikacija. Pri modifikaciji moždanika limunskom kiselinom vlačna je čvrstoća moždanika manja od izvlačne sile zavarenog moždanika.

KLJUČNE RIJEČI: rotacijsko zavarivanje; toplinska modifikacija; modifikacija limunskom kiselinom; izvlačna sila; moždanik

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1 INTRODUCTION

1. UVOD

When welding wood by friction, it is possible to join wood elements because the heat changes the structure of wood. Friction caused heat softens and melts the chemical structure of wood (hemicellulose, lignin and accessory substances), and cellulose fibres are intertwined in the melt thus formed. The strength of rotary welded joints is comparable to the strength of glued joints. Belleville et al. (2013) reported that, when heat-welding the birch and maple wood, the hemicellulose breaks down, and the heat affects the lignin polymer causing its depolymerisation. The efficiency of welding is therefore directly related to the properties of the original wood elements, primarily properties of lignin and hemicellulose. Due to lots of research and developments of different wood modification processes, and due to a wide range of applications of modified wood, the authors thought that it would be interesting to research the possibility of welding the modified wood.

Wood modification is a process in which the chemical, physical and technical properties of wood are changed (Hill, 2006). Nowadays, the modification of wood has progressed considerably and is increasingly being used for commercial purposes. It is also necessary to differentiate the modification of wood primarily for increasing its biological resistance and wood modification for changing its mechanical, physical and/or technical properties. Modification is the action of chemical, biological or physical factors in wood, and as this action changes the chemical composition of wood cell walls, modification improves certain properties of wood (Hill, 2006).

The desired properties of the modified wood are obtained by combining the type of heating medium, duration of the process, final temperature, pressure of the process and modified wood species (the main modification parameters). By reducing the coefficient of swelling and shrinking, the stresses in the coatingwood surface system are reduced and the service life of wood, as well as of products made of modified wood, is extended (Hasan and Despot, 2008). In addition to changes in physical and mechanical properties at a temperature of 433 to 493 K, wood resistance to fungi and climatic influences also increases (Kamdem et al., 2002). Heat treatment of beech wood at different temperatures causes a change in the characteristics and chemical components of wood (Windeisen et al., 2007). Wood deformations caused by changes in water content in modified wood can be reduced by up to 80 %, and water absorption by up to 70 % (Patzelt et al., 2002). The same authors stated that the decrease in water absorption is the result of a decrease in wood density and change in the chemical structure of the modified wood. Beech, pine and spruce had the greatest dimensional stability with the same mass loss. Compared to spruce, beech has a greater mass loss under the same conditions of thermal modification. Due to the presence of oxygen, regular air, as a heating medium of modification, causes a greater degree of decomposition of wood matter compared to nitrogen.

If the modification temperatures are higher than 200 °C, or if the modification pressure is elevated at the lower temperatures, significant degradation of the wood structure occurs. The share of cellulose remains almost unchanged, while the share of hemicellulose decreases from 21 % to 1.99 % (200 °C for 10 h). With an increase in the processing time, there may be an increase in the proportion of lignin, since some components formed by degradation are very similar to lignin (Yildiz et al., 2006). The mechanical properties of thermally modified wood are reduced both parallel and perpendicular to the direction of wood fibres. Mechanical properties of modified wood can be evaluated by testing its impact strength. Impact strength of modified wood decreases linearly with the increase of the modification temperature (Rapp and Sailer, 2001; Rapp et al., 2006).

The strength of the welded joint was improved by heating the dowel to 100 °C to a water content of 1.5 % and welding it into the substrate (Pizzi et al., 2004). The thermal modification of wood (200 °C for 48 hours) affects the reduction of the pull-out force of the dowel (Župčić et al., 2009). The same authors reported that the pull-out force of the modified hornbeam is 46 % lower compared to the unmodified one. Due to the increased brittleness of the thermally modified bases, the bases crack, so it is necessary to increase the welding time to avoid the cracks (Župčić et al., 2009). When wood is heated almost without oxygen, hemicelluloses break down first, followed by cellulose and finally lignin (Tjeerdsma et al., 1998). This cellulose decomposition is one of the factors that affect the strength of the welded joint. Thermally modified samples of beech wood can be successfully joined longitudinally using the rotary welding method (Pizzi et al., 2004; Župčić et al., 2011). The thermal modification affects the reduction of strength of the welded joint. By increasing the heat modification temperature from 140 °C to 180 °C, the joint strength decreases. The vibrational welding is also affected by the thermal modification of wood with a significant reduction in shear strength (Vaziri and Sandberg, 2021). The shear strength of the welded joints was lower in the samples that were modified before welding compared to the samples that were initially welded and then thermally modified. Therefore, thermal modification is not suitable for improving the resistance of the welded joint to the influence of water.

The weak water resistance of welded joints is based on uneven swelling of the weld and adjacent wood, which causes high stresses in the welded joint (Vaziri *et al.*, 2019). The strength of the joint made by vibration welding of thermally modified wood is twice lower than the joint of unmodified wood (Boonstra *et al.*, 2006).

Zhang *et al.* (2018) treated base samples with $CuCl_2$ and this treatment resulted in an increase of pullout force of % compared to untreated samples, with the same dowel welding time. The same authors reported that by extending the welding time, the pull-out force decreased. The treatment with $CuCl_2$ affects the strength of the joint due to the formation of more molten materials by the depolymerization and pyrolysis of the wood components (Zhang *et al.*, 2018). However, CuCl₂ has a negative impact on the environment.

Citric acid is one of polycarboxylic acids, together with butanetetracarboxylic acid (BTCA), used to improve the dimensional stability of solid wood and wood panels, as well as to increase the biological resistance of modified solid wood (Peyer *et al.*, 2000; Šefc, 2006; Hasan, 2010). It is ecologically and economically acceptable and does not contain formaldehyde. The tensile strength of wood modified with citric acid is reduced by up to 50 %, and with a decrease in the concentration of citric acid, the drop in tensile strength also decreases (Šefc, 2006). By increasing the concentration of citric acid up to 10.5 %, the biological resistance of beech and pine wood modified with citric acid against rot fungi increases significantly (Hasan, 2010).

This research aimed to determine the possibility of welding thermally modified wood and wood modified with citric acid. Today, modified wood is successfully used in exterior applications for floors and facades, so it is important to understand the possibilities of welding modified wood due to its wide range of application in construction.

2 MATERIALS AND METHODS

2. MATERIJALI I METODE

2.1 Materials

2.1. Materijali

Beech wood (*Fagus sylvatica* L.) was used in the research, randomly selected from the storage of sawn timber, so its origin is unknown. The planks were 50 mm thick without knots, pin knots, irregular heartwood and cracks. The chosen planks were first air-dried and then kiln dried to the final moisture content of (11 ± 1) %. The texture of planks was semi radial / tangential. For wooden dowels production, beech wood grooved rods, 1000 mm in length and 10 mm in diameter, were purchased on the free market.

2.2 Specimens for bases and dowels preparation

2.2. Priprema uzoraka baza i moždanika

The base specimens were made using the technique of sawing, fine planing and cross-cutting to the exact shape and size. Specimens with a cross section of $30 \times 30 \pm 0.1$ mm and a length of 64 ± 0.1 mm were used as a base for welding the dowels parallel to the grain. A hole was drilled in longitudinal direction in the centre of each specimen (Figure 1). Specimens with a cross section of $30 \times 30 \pm 0.1$ mm and a length of 200 ± 0.1 mm were used as a base for welding the dowels perpendicular to the grain. In each specimen, three holes were drilled perpendicular to the grain, spaced 66 mm apart, and the two side holes were moved from the edge of the specimen by 34 mm (Figure 2). All holes were drilled with a Universal HSS spiral drill of 8.5 mm in diameter, with a rotation frequency of 1520 min-¹. The diameter of the holes drilled parallel to the grain was smaller than the diameter of the drill by 0.01 to 0.05 mm, while the diameter of the holes perpendicular to the grain was smaller by 0.06 to 0.1 mm than the diameter of the drill.

The dowels were made by sawing 1000 mm long wooden rods to 120 mm. Each dowel end (cross section) was bevelled for 1 mm at an angle of 45°. The average diameter of the grooved dowels was 10.06 mm measured crosswise at the top of the dowel. The dowels prepared in this way were conditioned in laboratory conditions for more than 30 days before welding and modification.

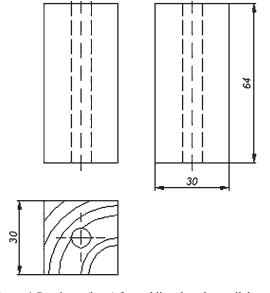


Figure 1 Specimen (base) for welding dowels parallel to grain

Slika 1. Uzorak za zavarivanje moždanika u smjeru vlakanaca

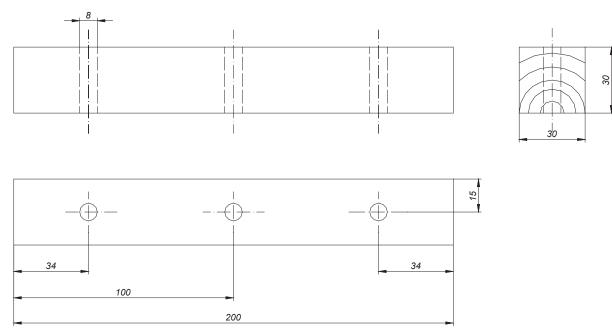


Figure 2 Specimen (base) for welding dowels perpendicular to grain Slika 2. Uzorak za zavarivanje moždanika okomito na smjer vlakanaca

2.3 Preparation of modified specimens2.3. Priprema modificiranih uzoraka

To investigate the influence of selected factors on the pull-out force of the welded dowels, 10 different joints were tested (Table 1). Joints were prepared with differently modified sample bases and/or dowels and different directions of welding or gluing of the dowels. Thermal modification as well as thermo-condansation phase of modification with citric acid were performed in air atmosphere. The procedure was carried out by gradual heating of the specimens (bases and/or dowels) up to a temperature of (103 ± 2) °C to achieve 0 % moisture content. Then the temperature was linearly increased to 160 °C over a period of 40 minutes. At a temperature of 160 °C, the wood was thermally modified for 10 hours. After 10 h, the temperature was linearly decreased over the next 40 min to (103 ± 2) °C, and the modified specimens were cooled to room temperature in a desiccator over silica gel. After modification, samples were weighed on a precise laboratory balance (±0.1 mg), and the moisture content and mass loss of thermally modified wood were determined. The average mass loss caused by thermal modification was 2.6 % (1.4 % to 3.5 %), and after 60 days of conditioning in laboratory conditions the average moisture content in the thermally modified specimens was 6.4 % (min, 4.2 % max. 7.7 % measured according to HRN ISO 13061-1:2015). After conditioning, the dowels were welded into the bases.

An aqueous solution of 10.5 % of citric acid was prepared with the addition of 7.5 % sodium hypophos-

phite, SHP (NaH₂PO₂×H₂O) as a catalyst. The second set of specimens (bases and dowels) were immersed in the prepared solution and impregnated by full cell procedure (vacuumed in the chamber for 2 h, after which the pressure in the chamber was raised to 4 bar and maintained for the next 2 h). After finishing the impregnation, the excess solution from the specimens was wiped with a cloth, and the impregnated specimens were left for 10 days in laboratory conditions for drying and conditioning. After drying in laboratory conditions, the specimens (bases and dowels) were placed in an oven dryer and the temperature was gradually (over 6 hours) increased to (103 ± 2) °C. Care was taken to prevent the sudden drying of the specimens and the appearance of cracks. Since the drying went well, the temperature was linearly increased during the next 40 minutes to the final thermo-condensation temperature of 160 °C and maintained for 10 h. After the completion of thermo-condensation (modification of the specimens), the temperature linearly decreased to (103 ± 2) °C over the next 40 minutes. The final cooling of the modified specimens to room temperature was carried out in a desiccator over silica gel. After 60 days of conditioning in laboratory conditions, the average moisture content in the specimens (bases and dowels) modified with citric acid was 6.0 % (min. 5.1 % max. 6.6 % measured according to HRN ISO 13061-1:2015).

Modified and unmodified dowels were glued and welded into modified and unmodified bases as described in Table 1. For each parameter combination, 36 joints were glued or welded.

Table 1 List of test combinations and sample codes
Tablica 1. Popis ispitanih kombinacija i označivanja uzoraka

O. nu. <i>Red. br.</i>	Test description /Opis ispitivanja	Code Oznaka
1	Unmodified dowels welded into thermally modified base perpendicular to wood grain <i>nemodificirani moždanici zavareni u termički modificiranu podlogu okomito na smjer vlakanaca</i>	MRTV
2	Unmodified dowels glued into thermally modified base perpendicular to wood grain nemodificirani moždanici zalijepljeni u termički modificiranu podlogu okomito na smjer vlakanaca	MRTLJ
3	Unmodified dowels welded into thermally modified base parallel to wood grain <i>nemodificirani moždanici zavareni u termički modificiranu podlogu paralelno sa smjerom vlakanaca</i>	MPV
4	Unmodified dowels glued into thermally modified base parallel to wood grain <i>nemodificirani moždanici zalijepljeni u termički modificiranu podlogu paralelno sa smjerom vlakanaca</i>	MPLJ
5	5 Unmodified dowels welded into base modified with citric acid perpendicular to wood grain nemodificirani moždanici zavareni u podlogu modificiranu limunskom kiselinom okomito na smjer vlakanaca	
6	Unmodified dowels welded into base modified with citric acid parallel to wood grain nemodificirani moždanici zavareni u podlogu modificiranu limunskom kiselinom paralelno sa smjerom vlakanaca	LKPV
7	Unmodified dowels glued into base modified with citric acid parallel to wood grain nemodificirani moždanici zalijepljeni u podlogu modificiranu limunskom kiselinom paralelno sa smjerom vlakanaca	LKPLJ
8	Dowels modified with citric acid welded into unmodified base parallel to wood grain limunskom kiselinom modificirani moždanici zavareni u nemodificiranu podlogu paralelno sa smjerom vlakanaca	MLKPV
9	Control test (8.5 mm) unmodified dowels welded into unmodified bases perpendicular to wood grain <i>kontrolno ispitivanje (8,5 mm): nemodificirani moždanici zavareni u nemodificiranu podlogu okomito na smjer vlakanaca</i>	8,5 RTV
10	Control test (8.5 mm) unmodified dowels welded into unmodified bases parallel to wood grain <i>kontrolno ispitivanje (8,5 mm): nemodificirani moždanici zavareni u nemodificiranu podlogu u smjeru vlakanaca</i>	8,5 PV

2.4 Production of test joints by rotary welding

2.4. Izrada ispitnih spojeva rotacijskim zavarivanjem

All welded joints were made using the rotary welding technique, where the dowel rotates at a certain frequency and is welded into stationary base. The dowels were welded parallel and perpendicular to the wood grain. The rotation frequency of the dowels was 1520 min⁻¹, the welding depth was 20 mm, and the duration of welding was from 0.8 to 1 s (Table 2), depending on the base or dowel modification type. Rotary welding was performed with an average interference fit of 1.6 mm in order to keep the welding times within optimal limits. It was not possible to use a larger interference fit

due to the appearance of cracks in the modified bases or breakage of the modified dowels during welding. After the rotation was stopped, the pressure on the dowel was maintained for 5 seconds.

2.5 Production of test joints by gluing 2.5. Izrada uzoraka tehnikom lijepljenja

The samples intended for gluing were of the same dimensions as the samples for welding, except the holes were 10 mm in diameter. The average interference fit during gluing of the dowels was 0.16 mm. One-component waterproof PVAC bond Pattex super 3 (Henkel, Hungary) was used. The gluing process consisted of applying the glue on the dowel and inserting it partially into the hole. Then, using a stopper, the dowel

Type of modification and section Vrsta modifikacije i presjeka	Average duration of welding, s <i>Prosječno trajanje zavarivanja</i> , s	Feed per revolution, mm <i>Pomak po okretaju,</i> mm
MPV	1.0	0.79
MRTV	0.9	0.88
LKPV	0.9	0.88
LKRTV	0.8	0.99
MLKPV	1.0	0.79
8,5 PV	0.8	0.99
8,5 RTV	0.8	0.99

Table 2 Data on welding durationTablica 2. Podatci o trajanju zavarivanja

was pressed to a depth of 20 mm. After pressing, the excess glue was removed. The mass of the applied glue was determined by measuring the mass of the glued joints before applying the glue and after gluing (after removing the excess glue from the joint). The average application of glue for grooved dowels was 244 g/m² per joint. After gluing the dowels, the glued joints were conditioned in laboratory conditions for a minimum of seven days.

2.6 Testing method and data analysis2.6. Metoda ispitivanja i analiza rezultata

The testing of the undamaged glued and welded joints was carried out on a universal tensile testing machine after its visual control. All prepared joints were conditioned for seven days before they were tested on a computer controlled universal / tensile testing machine Shimadzu AGX-V (AUTOGRAPH Precision Universal Tester AGX-V, SHIMADZU Corporation, Kyoto, Japan). The joints were tested using joint jaws for precise positioning. The movement of jaws during the test was 5 mm/min.

The data obtained from the measurements were processed in the StatSoft Statistica 8.0 software package. If the condition of normality of distribution and homogeneity of variance was satisfied (Table 4), the differences between individual groups of samples were tested by Student's T-test or by analysis of variance. If the condition of homogeneity of variance was not met (Table 7; F-test and Levene's test), the Mann-Whitney U-test or the Kruskal-Wallis test was used to confirm whether or not there was a statistically significant difference between individual groups of samples. Post-hoc tests established statistically significant differences between individual groups of samples if they existed (Tables 5 and 8). The difference greater than 5 % was considered significant. Presentations of comparisons were made using box and whisker graphs (Figure 3 and 4).

3 RESULTS AND DISCUSSION 3. REZULTATI I RASPRAVA

melting temperature is reached.

During welding, a part of the wooden tissue of the dowel and base, which is in the zone of an interference fit, is melted (black line in Figure 3). The tip of the dowel is turned upwards, and it is visible that the welded joint becomes conical (Figure 3). Conical shape of the welded joint incurs due to tear off of the tip of the dowel during the beginning of the welding, while no

During welding, modified wood melts much less than unmodified wood. The border of the dowel and the base in the welded joint in Figure 4 is the crack, and the thickness of the melt of thermally modified dowel is visibly smaller than the thickness of the melt of unmodified base (Figure 4).



Figure 3 Sections of welded unmodified dowel into unmodified base; tip of the dowel is turned upwards (Župčić, 2010) Slika 3. Presjeci zavarenoga nemodificiranog moždanika u

nemodificiranu podlogu; vrh moždanika usmjeren je prema gore (Župčić, 2010.)

An average density of unmodified wood of bases was 0.69 g/cm³ (min. 0.62 and max. 0.74 g/cm³; according to HRN ISO 13061-2:2015). Average density of thermally modified wood (bases) was (0.63 ± 0.04) g/ cm³, while the average density of wood modified with citric acid was (0.67 ± 0.03) g/cm³. Thermally modified wood has increased dimensional stability, which is certainly a positive characteristic, but also increased brittleness, which is a negative characteristic when wood is to be used for rotary welding. The fragility of wood during rotary welding requires an extension of the duration of the welding process, and it requires the reduction of the interference fit. The optimal interference fit for rotary welding of 10 mm diameter dowels is 2 mm (Pizzi *et al.*,

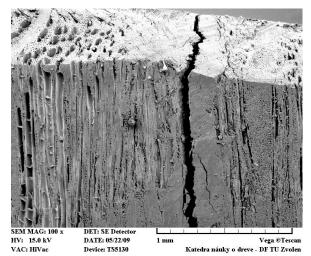


Figure 4 SEM micrograph of transversal section of welded thermally modified dowel into unmodified base: Left of crack - dowel; Right of crack - base (Župčić, 2010) **Slika 4.** SEM mikrografija transverzalnog presjeka zavarenog termički modiciranog moždanika u nemodificiranu podlogu: lijevo od pukotine je moždanik, a desno od pukotine je podloga (Župčić, 2010.)

2004). The welding time also affects the strength of the rotationally and vibrationally welded joints (Župčić *et al.*, 2011; Amirou *et al.*, 2020). Within this investigation, an interference fit of 2 mm was not adequate to ensure the optimal movement of the dowel during welding of the modified wood. Therefore, the interference fit was reduced to 1.6 mm, so that the welding time of the modified and unmodified specimens could be approximately the same, considering that the extension of the welding time reduces the strength of the joint. It was not possible to use a larger interference fit due to the appearance of cracks in the modified bases or the breakage of dowels during welding.

Welding the dowels modified with acid into the modified bases was difficult to perform due to occurrences of cracks and splits in the bases or fractures of the dowels. Due to the increased brittleness of the wood modified with citric acid, the dowels crumbled, and the bases split during welding. Splitting of the bases occurred often at the very beginning of welding due to the pressure of the dowel on the walls of the holes. For these reasons, modified dowels were successfully welded only in unmodified bases (Figure 4; but other problems arose during the pull-out test), and welding of modified dowels into modified bases was not carried through.

In addition to the increase in wood brittleness, it was observed that, when welding modified wood, less melt is coming out of the joint and more broken pieces of wood occur. A similar phenomenon occurs with vibrational welding of wood. During vibrational welding, the weld lines of heat-treated wood show intertwined fibres with none or a very small amount of the molten material that is usually observed when welding unmodified wood. In weld lines, obtained by hydrothermal processes during welding, an increase in stiffness and brittleness of the wood cells is observed (Boonstra *et al.*, 2006).

Statistical analysis revealed a statistically significant reduction in pull-out force of welded dowels compared to glued ones in the modified bases, both in a parallel and perpendicular direction to the grain (Tables 3, 4 and 5; 6, 7 and 8). The pull-out force of welded dowels parallel to the grain is 27 % lower, while the pull-out force of welded dowels perpendicular to the grain is 45 % lower than that of glued dowels. Higher pull-out force values were achieved by gluing and welding the dowels parallel to the grain compared to the perpendicular direction, which was expected on the basis of previous knowledge. The results of the research (Figure 5, Table 3) show an additional reduction in the pull-out force of glued and welded joints of specimens modified with citric acid compared to the thermally modified samples, as it was proven by Poljak (2008) who conducted a similar research.

A comparison of the pull-out forces obtained by welding thermally modified and unmodified wood parallel to the grain (Table 6, Figure 6) indicates a 25 % reduction in the pull-out force of thermally modified wood. Specimens modified with citric acid and welded perpendicular to the grain achieve a 32 % reduction in pull-out force compared to unmodified ones. Pull-out force of welded joints of thermally modified wood perpendicular to the grain is reduced by more than 36 % compared to pull-out force of unmodified joints. Welded joints modified with citric acid achieve a 4 % reduction in pull-out force compared to unmodified ones. Such a small reduction in pull-out force indicates an illogical result. Namely, it was expected that the

Code Oznaka	Number of welded/glued and tested joints Broj zavarenih/zalijepljenih i ispitanih spojeva	Mean pull-out force, N Srednja vrijednost izvlačne sile, N	Std. Dev, N	Min, N	Max, N
MPV	36	3255.1	501.3	2606	4493
MRTV	25	5198.4	481.5	1118	2989
MPLJ	31	4477.6	511.5	3633	5599
MRTLJ	12	4058.6	408.5	3586	4772
LKPV	29	2948.3	351.1	2342	3472
LKRTV	27	3333.5	333.1	2786	4078
LKPLJ	18	3606.3	537.6	2863	4817

 Table 3 Descriptive statistics of pull-out force depending on modification type and grain orientation of welded/glued joints

 Tablica 3. Deskriptivna statistika izvlačne sile u ovisnosti o modifikaciji drva i smjeru vlakanaca zavarenih/slijepljenih spojeva

 Table 4 Levene test of homogeneity of variances of welded/glued joints pull-out forces

 Tablica 4. Testiranje homogenosti varijance (Leveneov test) izvlačne sile zavarenih/slijepljenih spojeva

							5	
Variable	SS	df	MS	SS	df	MS	F	
Varijabla	effect	Effect	Effect	Error	Error	Error	ľ	P
Pull-out force, N <i>izvlačna sila</i> , N	560113.1	6	93352.18	12561687	171	73460.16	1.270787	0.273268

*Orange colour means statistically significant difference at p < 0.05000. / Narančasto su otisnute statistički značajne razlike pri p < 0.05000.

Code	(1)	(2)	(3)	(4)	(5)	(6)	(7)		
Oznaka	M=3255.2	M=2198.4	M=4058.6	M=4058.6	M=2948.3	M=3333.5	M=3606.3		
MPV (1)		0.000000	0.000000	0.000191	0.297686	0.998258	0.311292		
MRTV (2)	0.000000		0.000000	0.000000	0.000008	0.000000	0.000000		
MPLJ (3)	0.000000	0.000000		0.295059	0.000000	0.000000	0.000001		
MRTLJ (4)	0.000000	0.000000	0.295059		0.000000	0.002571	0.314365		
LKPV (5)	0.297686	0.000008	0.000000	0.000000		0.129865	0.001165		
LKRTV (6)	0.998258	0.000000	0.000000	0.000571	0.129865		0.690699		
LKPLJ (7)	0.311292	0.000000	0.000001	0.314356	0.001165	0.690699			

 Table 5 Multiple post hoc test of welded/glued joints pull-out forces

 Tablica 5. Višestruki *post hoc* test izvlačne sile zavarenih/slijepljenih spojeva

*Orange colour means statistically significant difference at p < 0.05000. / Narančasto su otisnute statistički značajne razlike pri p < 0.05000.

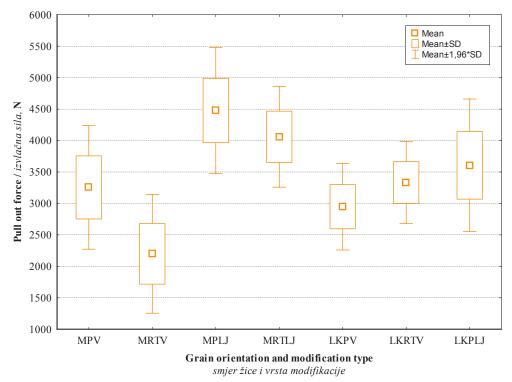


Figure 5 Influence of modification type and grain orientation on pull-out force of welded/glued joints **Slika 5.** Utjecaj vrste modifikacije drva i smjera žice na izvlačnu silu zavarenih/slijepljenih spojeva

LKRTV samples would achieve a lower pull-out force compared to the MRTV samples. Comparing LKPV and LKRTV samples, the pull-out force of LKRTV samples is slightly increased (statistically not significant; Table 8), which was not expected. This increase in pull-out force is affected by the growth rings width, density of wood and the reduction of welding time by 0.1 s. Wood welding is a process that depends on nu-

 Table 6 Descriptive statistics of pull-out force depending on modification type and grain orientation of welded joints

 Tablica 6. Deskriptivna statistika izvlačne sile u ovisnosti o vrsti modifikacije i smjeru žice zavarenih spojeva

Code Oznaka	Mean Pull-out force, F, N Srednja vrijednost izvlačne sile, N	Number of welded and tested joints Broj zavarenih i ispitanih spojeva	Std. Dev. (F), N	Min (<i>F</i>), N	Max (F), N	Q25 (F), N	Median (F), N	Q75 (F), N
8.5 PV	4346.8	30	493.4	3472	5305	4013	4390	4699
MPV	3255.2	36	501.3	2606	4493	2908	3128	3510
LKPV	2948.3	29	351.1	2342	3472	2657	2945	3246
MLKPV	2586.4	25	966.4	774	4063	1886	2742	3269
8.5 RTV	3455.9	30	274.4	3057	3940	3222	3438	3660
MRTV	2198.4	25	481.5	1118	2989	1950	2179	2571
LKRTV	3333.5	27	333.1	2786	4078	3087	3319	3499

Table 7 Levene test of homogeneity of variances of welded joints pull-out force
Tablica 7. Testiranje homogenosti varijance (Leveneov test) izvlačne sile zavarenih spojeva

Variable	SS	df	MS	SS	df	MS	F	
Varijabla	effect	Effect	Effect	Error	Error	Error	Г	p
Pull-out force, N	5070116	6	879686.0	17146604	195	87931.30	10.00424	0.000000
<i>izvlačna sila,</i> N	5278116	0	8/9080.0	1/140004	195	87931.30	10.00424	0.000000

*Orange colour means statistically significant difference at p < 0.05000. / Narančasto su otisnute statistički značajne razlike pri p < 0.05000.

	Tablica 8. visestruka usporedba rangova izviačne sile zavarenih spojeva										
	Call		Kruskal-Wallis test H (6. <i>N</i> = 202) = 119.8847 <i>p</i> = 0.000								
	Code Oznaka	8,5 PV	MPV	LKPV	MLKPV	8,5 RTV	MRTV	LKRTV			
	Оглака	R:181.03	R:101.96	R:74.793	R:65.980	R:128.33	R:26.180	R:114.02			
	8.5 PV		0.000001	0.000000	0.000000	0.010083	0.000000	0.000325			
	MPV	0.000001		1.000000	0.379561	1.000000	0.000013	1.000000			
	LKPV	0.000000	1.000000		1.000000	0.009163	0.048516	0.254168			
	MLKPV	0.000000	0.379561	1.000000		0.001719	0.337619	0.064440			
	8.5 RTV	0.000000	1.000000	0.009163	0.001719		0.000000	1.000000			
	MRTV	0.010083	0.000013	0.048516	0.337619	0.000000		0.000001			
	LKRTV	0.000325	1.000000	0.254168	0.064440	1.000000	0.000001				

Table 8 Multiple comparisons of p values of welded joints pull-out force Tablico & Vičestruka usperadba rangova izvlačna sila zave

*Orange colour means statistically significant difference. / Narančasto su otisnute statistički značajne razlike.

merous welding parameters (Pizzi et al., 2004; Leban et al., 2008; Župčić, 2010), such as the duration of the welding process, interference fit, growth rings width, wood density, rotation frequency, welding depth, etc. The average growth rings width of LKPV bases was 2.5 mm and of LKRTV bases 3.0 mm. The average density of LKPV bases was 0.65 g/cm3, while the average density of LKRTV bases was 0.69 g/cm³. Increasing the growth rings width of hardwoods increases the density of wood, and the increase in wood density results in a slight increase in the pull-out force (Župčić, 2010), but not significant. Therefore, the density of wood and the width of the growth rings can result in a slight increase in the pull-out force of the welded dowels (Table 6, Figure 6). The results of this research indicate a significant reduction of the pull-out force of welded joints of thermally modified wood and wood modified with citric acid, which raises the question of the justification of welding the modified wood (Tables 5 and 8).

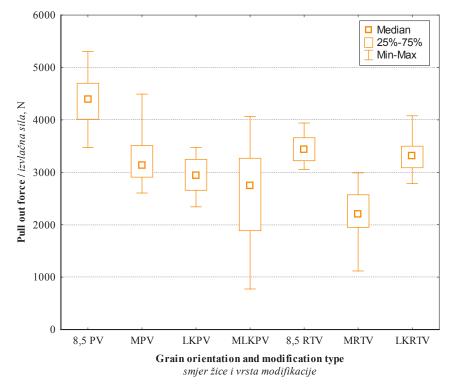


Figure 6 Influence of modification type and grain orientation on pull-out force of welded joints Slika 6. Utjecaj vrste modifikacije drva i smjera žice na izvlačnu silu zavarenih spojeva

Dowels modified with citric acid can be successfully welded to an unmodified base (MLKPV). During welding, there were no major cracks or splits in the bases. As a result of the modification, some dowels were slightly curved and deflected, which created major problems during welding. If the deflection of the dowel was greater than 1 mm, it was not welded. During pull-out testing, only three, out of 25 welded dowels, were pulled out of the bases. All other dowels were weaker than the welded joint, so the pull-out force could not be measured. High data variability of measured pull-out force is due to the different tensile strength of the modified dowels. The tensile strength of the dowels varied from a minimum of 774 N to a maximum of 4063 N, and the average pull-out force of three dowels that withstood the tension during testing (no fracture occurred in the dowel) was 3420 N, which is comparable to the MRTV and LKRTV samples (Table 6, Figure 6).

4 CONCLUSIONS

4. ZAKLJUCAK

The untreated dowels can be welded or glued into a thermally modified base or a base modified with citric acid. If both the base and the dowel are modified with citric acid, major errors (splits in the bases or breakage of the dowels) occur during welding.

Thermal modification causes a decrease in the pull-out force of welded and glued dowels parallel and perpendicular to the grain. Welded and glued dowels parallel to the grain achieve higher pull-out forces compared to welded and glued dowels perpendicular to the grain.

Modification with citric acid in glued or welded samples parallel and perpendicular to the grain reduces the pull-out force compared to thermally modified samples.

The strength of the welded joints of dowels modified with citric acid is higher than the tensile strength of the modified dowels.

Due to the significant reduction of pull-out force of dowels welded in thermally modified wood or wood modified with citric acid, their use is not recommended for welded joints.

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Factors Influencing Behaviour of Solid Wood Bending Process

Čimbenici koji utječu na proces savijanja cjelovitog drva

REVIEW PAPER

Pregledni rad Received – prispjelo: 24. 2. 2022. Accepted – prihvaćeno: 28. 6. 2022. UDK: 674.028 https://doi.org/10.5552/drvind.2023.0020 © 2023 by the author(s). Licensee Faculty of Forestry and Wood Technology, University of Zagreb. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license.

ABSTRACT • The effect of temperature and moisture on the behaviour of solid wood is a well-known fact that directly affects wood creep as well. Wood creep includes three types of behaviour, such as viscoelastic creep, mechano-sorptive creep, and pseudo-creep and recovery. All these types can occur simultaneously, and it is sometimes complicated for researchers to isolate or distinct one from another. This paper presents a review of literature on wood rheology and creep properties, as well as factors that influence them, mainly time, temperature, and moisture content. The study of the viscoelasticity and wood creep is very important for gaining knowledge to be applied in solid wood bending.

KEYWORDS: solid wood; creep; rheology; viscoelastic; solid wood bending

SAŽETAK • Utjecaj temperature i vlage na ponašanje drva dobro je poznata činjenica koja ima izravan utjecaj i na puzanje drva. Puzanje drva obuhvaća tri tipa ponašanja: viskoelastično puzanje, mehaničko puzanje uzrokovano sorpcijom vode te prividno puzanje i oporavak. Svi ti tipovi ponašanja drva mogu se pojaviti istodobno, a istraživačima je katkad komplicirano izolirati ili razlikovati jedan tip od drugoga. Ovim je radom prikazan pregled literature vezane za reologiju i svojstva puzanja drva, kao i čimbenika koji na njih utječu, a uglavnom su to vrijeme, temperatura i sadržaj vode u drvu. Proučavanje viskoelastičnosti i puzanja drva iznimno je važno radi stjecanja specifičnih znanja potrebnih za istraživanja i razvoj tehnološkog procesa savijanja cjelovitog drva.

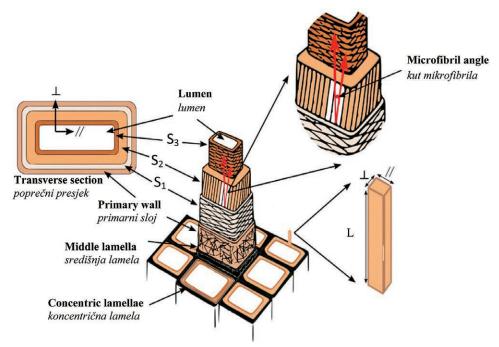
KLJUČNE RIJEČI: cjelovito drvo; puzanje; reologija; viskoelastičnost; savijanje cjelovitog drva

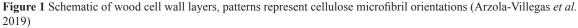
1 INTRODUCTION

1. UVOD

Wood is a 3-component fibre-reinforced biocomposite. Its cells are multi-layered tubes with closed ends. Individual cells have four distinct cell wall layers. Those layers are primary, S1, S2, and S3 and each of them is composed of a combination of cellulose microfibrils, lignin and hemicelluloses. Microfibrils arrangement is the basic of dividing cell wall layers (Cave and Walker, 1994). Lignin is an amorphous phenol, and the cellulose and hemicellulose are linear polysaccharides (Tabet and Aziz, 2013). According to Arzola-Villegas *et al.* (2019), primary wall and middle lamella are grouped into a layer called the compound middle lamella due to them being almost identical. The middle lamella provides adhesion between the cells and interconnects them. It is mostly made of lignin

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Slika 1. Shema slojeva stanične stijenke drva; uzorak predočuje orijentaciju celuloznih mikrofibrila (Arzola-Villegas *et al.*, 2019.)

with some hemicelluloses. Schematic of wood cell wall layers in Figure 1 shows the patterns that represent the orientation of cellulose microfibril.

Navi and Sandberg (2012) stated that the S2 layer is thicker than S1 and S3 and that therefore it contributes the most to the mechanical and physical properties of the cell wall. For these reasons, the term microfibril angle (MFA) is usually applied to the orientation of cellulose microfibrils in the S2 layer in literature. It refers to the angle between the direction of cellulose microfibrils and the longitudinal cell axis. According to Barnett and Bonham (2003), wood mechanical properties are profoundly affected by differences in microfibril angle since the S2 layer represents the cell wall major component. Tensile strength and stiffness quickly decrease as the MFA increases (Mary Treacy et al., 2001). This means that wooden elements with long cells and low microfibril angles would make great material for bending due to better tensile strength, which can cause problems.

Bending of wood is the type of processing with certain levels of mechanical destruction. It is often used in manufacture of furniture, ships and boats, sports equipment, fishing rods, bows and other kind of tool equipment. Higher material utilization is the most important advantage of wood bending. Some of other advantages are small investments in technology, and higher strength and stiffness of bent wood elements than those of sawn elements. For example, chairs produced of bent elements have greater load-bearing capacity (e.g., legs, back rest, and arm rest) than those of sawn elements. Greater strength of bent elements is due to continuous grain slope. Sawn elements have their grain slope cut off in certain parts, which lowers their strength and load-bearing capacity.

When solid wood bends, it stretches on the convex (outer) side of bent piece and compresses on the concave (inside) side. Therefore, convex side is longer than the concave side. Bent elements tend to return to their original position due to high remaining stresses caused by deformation of wood. For these reasons wood is softened by moisture and heat or in some cases with chemicals because that way stress development is limited. It allows wood elements to retain their bent shape. Wood, even after softening or plasticizing, cannot be stretched very much but it can be significantly compressed, which is the reason why manufacturers are compressing wood while at the same time preventing stretching along the outer (convex) side (usually by using a metal strip). Some prior knowledge of wood creep and rheology of materials (mainly wood) is required to bend wood properly and precisely without a high rate of damage and discard.

Wood is a natural polymer, and it creeps under imposed stress, and it is a viscoelastic mechanical problem that is mostly encountered during processing and utilization. Wood creeps during the drying of solid wood, as well as during long-term loading of wood members such as roof frames, beams, columns, and walls. It is necessary to understand wood creep properties and the influence of creep on wood and wood products in long-term service conditions to make rational and efficient use of it (Jin *et al.*, 2016).

Pustaić and Cukor (2009) called material creep "a phenomenon where stresses and strains that occur under the load of a deformable body change over time even if the load is time-invariant (constant)". On the other hand, Curtu et al. (2015) defined the rheological behaviour as the system that strains under external load in a certain amount of time and under the influence of environmental factors. This phenomenon was also divided by Pustaić and Cukor (2009) in two forms; the first form is called creep – the change in deformation over time, while the second form is called relaxation the change in stress. Fridley (1992a); Fridley (1992b) characterized wood as viscoelastic material governed by creep behaviour. Creep can be plastic and elastic, which means that, with plastic creep, the deformations are basically irreversible (only slight decrease in deflection) after unloading the element, while with elastic creep, deformations decrease over time after removing the load from the element and they completely disappear after some time. Hunt (1999) claims that for many structural applications the most important mechanical property of wood is its resistance to deflection, including elastic and creep deflection. Many factors affect rheological phenomenon. Curtu et al. (2015) listed some of them to be temperature and air humidity or moisture content (MC) of rheological system (in this case wood MC), various radiations in term of intensity, duration, and type - UV, IR, X, geometry of the elements; loadings in terms of intensity, variation, duration; defects; aggressive environment; composition, material properties; and combinations of these factors. Rheology science is based on the theories of the strength of materials, thermodynamics, chemistry and materials science, but in terms of application, it provides a personalized analysis or diagnosis according to the condition of the structures/systems used (Curtu et al., 2015). Hunt (1999) suggests that creep includes three distinct types of behaviour, which are difficult to separate because they can all operate simultaneously. The three types mentioned by Hunt (1999) are timedependent (viscoelastic) creep, mechano-sorptive (moisture-change) creep, and the pseudo-creep and recovery ascribed to differential swelling and shrinkage.

2 MECHANICS AND RHEOLOGY OF WOOD

2. MEHANIKA I REOLOGIJA DRVA

The study of strain behaviour of polymeric materials, which is time-dependent, is called creep and it is defined as continuous deformation in time when exposed to a continuous load (Peng *et al.*, 2017). In terms of creep behaviour, it is well known that, when stress is applied, an immediate elastic strain appears and in case of longer period exposure, long-term strain is developed. Navi and Stanzl-Tschegg (2009) and Morreale et al. (2015) described wood as a sustainable building material, which shows creep behaviour due to its viscoelastic nature. Creep of wood as viscoelastic material occurs as a combination of elastic deformation and viscous flow, known as viscoelastic deformation (Bodig and Jayne, 1993). As already mentioned in the introduction, a variety of factors influence wood creep behaviour, some of them being stress level, composite formulation, temperature, and MC (Liu, 1993; Chen and Lin, 1997; Hogan and Niklas, 2004; Zhang et al., 2007). The factors listed above are a more simplified explanation of rheological factors than that of Curtu et al. (2015) presented in the previous chapter. Leicester (1971) reported that, while drying under a load, the deflection increase is more influenced by MC than by time. Hunt (1999) suggests that the main design parameter for timber is deflection which is the addition of two types of behaviour, namely elastic deflection and creep. The second component of deflection, creep, is of two types as already mentioned earlier in this paper: viscoelastic and mechano-sorptive. These two types have traditionally been considered independent and additive but contrary to previous views, the experimental results of Hunt (1999) led to the conclusion that timedependent creep and mechano-sorptive creep are different means of reaching the same creep result. In addition to the time-dependent creep and mechano-sorptive creep, Hunt (1999) also mentions a pseudo-creep and recovery phenomenon, which is manifested during continued moisture cycling, in which the creep deflection eventually increases during desorption but decreases during sorption. He ascribed this to differences in the normal longitudinal swelling and shrinkage of wood as stated by Hunt and Shelton (1988) who claimed that "a tensile strain resulted in a smaller shrinkage coefficient, while a compression strain resulted in a larger one". This indicates that pseudo-creep and recovery are approximately a reversible phenomenon, contrary to the other two types of creep that are irreversible while the loading is maintained. Curtu et al. (2015) explained the creep phenomenon that appeared in the timber by the development of dislocation between the molecule chains and the destruction of the primary and secondary links, by occurrence of cracks and shears between the wood fibres. On the other hand, Kollmann (1968) stated that the elastic properties of wood are influenced considerably by knots, as they have cross grain or interlocked fibres. Furthermore, Hunt (1999) considers that deflection has acquired greater importance since the increased use of 'plantation grown' timber, which means that more commercial timber is fast grown (i.e., wide growth rings) and is cropped at a sufficiently early age to contain a significant proportion of 'juvenile' wood. He also claims that

juvenile wood can creep up to five times as much as mature wood and that all these factors mentioned result in a material that has lower elastic modulus and creeps significantly more than slow-grown mature wood. Kollmann (1968) mentions investigations carried out by Kellog (1960), which indicate that ultimate tensile strain of wood including accumulated creep increases after repeated stressing in tension parallel to the grain and that there is an indication that this increase in ultimate strain is a result of the increased strain due to the creep that occurred during the cycling period.

The general rheological model was developed under the assumption of strain which is divided into parts, meaning that the total mechano-sorptive creep strain is the sum of all the above strains: elastic strain, viscoelastic strain at constant MC, free shrinkage/ swelling strain, mechano-sorptive strain, and thermal expansion/contraction strain (Vici *et al.*, 2006; Guo, 2009). However, Peng *et al.* (2017) claim that this rheological model and its application is still not understood well enough due to wood viscoelastic properties that depend on climate conditions, complex anatomic structure, stress level, and load model.

3 INFLUENCE OF MOISTURE AND TEMPERATURE ON WOOD CREEP 3. UTJECAJ VLAGE I TEMPERATURE NA PUZANJE DRVA

Wood adsorbs and desorbs moisture with changes in conditions such as relative humidity and temperature, which is the reason why it is considered a hygroscopic material. The changes in MC of wood lead to swelling and shrinkage, which are dimensional changes that happen when wood adsorbs or desorbs moisture. For that reason, it is expected that MC and especially its changes would have effect on wood creep. Hunt (1999) named a second type of creep, which is mentioned and associated with transient moisture-content changes, as mechano-sorptive creep, while Kaboorani et al. (2013) named dual effect of wood moisture and the load mechanical absorption effect. Armstrong and Kingston (1960) were the first ones that discovered the effect of moisture changes on creep and reported it. Hunt (1999) summarized this phenomenon in three statements: "the deflection of wood under load increases massively during moisture changes, whether sorption or desorption, and the final creep compliance is greater than it would be expected at either the lower or the higher MC"; "the final deflection depends mainly on the size of the moisture step and is little affected by its duration"; "and while the moisture is cycled within a given range there is a gradual decrease in creep rate; any increase to a yet higher MC causes the creep rate to increase to the original highest rate". Jin et al. (2016) put it simply by saying that moisture in wood, acting as a plasticizer, strongly affects the wood viscoelastic properties. It is well known that as the wood dries below fibre-saturation point its strength increases and that above fibre-saturation point the effect of MC on static strength is negligible. Following these claims, Kollmann (1968) suggests that above the fibre saturation point free liquid water filling the coarser capillaries in vessels, tracheids and other elements of the wooden tissues does not affect strength and elastic properties. This would suggest that, when wood is exposed to moisture changes, deformation would be higher than when it is exposed to constant environmental conditions for the same time (Bazant and Meiri, 1985; Nakano, 1999). It can be concluded that MC would have an impact on creep properties of wood as well as its lowered mechanical properties. Jin et al. (2016) suggests that moisture in wood (when MC < 30%) could affect the internal hydrogen bonds between wood polymers, which would directly influence wood plasticity and deformation. When wood is under load, adsorption and desorption cause additional deflection, hydrogen bonds break during desorption, which leads to an increased response in strain (Gibson, 1965).

Hoffmeyer and Davidson (1989) related the process of forming, breaking, and reforming of hydrogen bonds to slip planes in the cell walls. Slip planes form faster and at lower stresses when exposed to varying moisture conditions than at constant MC.

Hsieh and Chang (2018) separated their results into two distinct groups with MC higher or lower than the equilibrium moisture content (EMC) and found that the mechano-sorptive effect is time-independent. It is well known that temperature itself, as well as changes in temperature, have influence on strength, elasticity, and plasticity of wood. Kollmann (1968) claims that strength and stiffness of wood decrease with increasing temperature due to thermal expansion of the crystal lattice of the cellulose and due to the increased intensity of the thermal molecular oscillations. Researchers often have problems when conducting creep experiments with increasing temperature because retaining constant MC at higher temperatures is very difficult due to changes in MC that take place simultaneously with changes in the wood temperature; for these reasons, achieving desired MC levels at higher temperatures during creep tests was shown to be a technical difficulty (Jin et al., 2016). Jin et al. (2016) conducted experiments on small birch wood samples conditioned at six MCs (0 %, 6 %, 12 %, 18 %, 24 % and 120 %) and temperatures ranging from 5 °C to 105 °C in increments of 10 °C. Their results of instantaneous compliance (IC), (which is reciprocal of modulus of elasticity), are shown in Figure 2. Based on data shown in Figure 2, it can be concluded that with increasing MC, the instantaneous compliance at the same

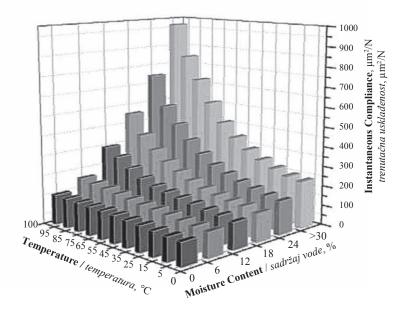


Figure 2 Instantaneous compliance of samples at 6 MCs and 11 temperatures (Jin *et al.*, 2016) **Slika 2.** Trenutačna usklađenost uzoraka pri šest različitih sadržaja vode i 11 različitih temperatura (Jin *et al.*, 2016.)

temperature increased consistently, which agrees with previous research conducted by (Tissaoui, 1996; Moutee, 2006; Engelund *et al.*, 2012) and with Madsen (1992) who claimed that higher temperatures produce more creep, while at the same time samples were more affected by changes in MC than by temperature in a given range.

Yang et al. (2004) mention dependence of temperature on mechanical behaviour of wood (as a natural polymer) by saying that mobility of their molecular chain parts and chain segments are completely frozen when polymer components of wood are in the glassy state and that thermal motion only occurs at fixed positions. This again means that, when temperature is increased, molecular chains become loosened because of the repulsion between chains (Yang et al., 2004; Engelund and Salmén, 2012). Dwianto et al. (1998b) divided these structural changes at molecular level in wood under steaming in three processes depending on treatment conditions. The first process they mention is hemicelluloses degradation, the second process is formation of cross-linkages between cell wall polymers and the last process is decomposition of hemicelluloses and lignin. They suggest that the difference in structural changes between the mentioned processes can be determined by creep measurement rather than by stress relaxation measurement. Results of Dwianto et al. (2000) research suggest that, with increasing prestreaming time, degradation rate of cell wall polymers accelerated. They recorded large deformations at stress level of 0.71 of maximum compressive strength above 160 °C and concluded that creep deformation in those cases was sensitive to any degradation of cell wall

polymers and that increasing temperature noticeably accelerated the increase of creep compliance while steaming above the mentioned temperature. Data from Armstrong and Kingston (1960); Gibson (1965); Hoffmeyer and Davidson (1989); Madsen (1992); Hsieh and Chang (2018); Peng *et al.* (2017) agree with the statement that changes in MC contribute more to creep than the initial MC. Hsieh and Chang (2018) reported that higher strain at same time point was noticed at wood samples with higher MC, as well as higher creep strain increment caused by higher MCs and higher desorption rates.

Wood creep knowledge in relation to moisture and temperature is also important while drying wood. For example, Zhan and Avramidis (2011) obtained valuable data in their research for kiln operators to choose the correct control strategy and theoretically valuable to determine the mechano-sorptive creep development mechanism during timber drying processes, which can help in obtaining a better description of the wood drying stress and drying strain.

4 TIME AS A FACTOR INFLUENCING WOOD CREEP 4. VRIJEME KAO JEDAN OD ČIMBENIKA KOJI UTJEČU NA PUZANJE DRVA

Creep phenomenon also depends on time, and it appears in many other materials, not only wood, and it is called time-dependent creep (Hunt, 1999). Hunt also claims that wood, as any other material, when considering its behaviour in terms of time requires temperature and MC as well as other relevant variables to remain

constant. Sun and Frazier (2007) suggest that, in order to ease moisture control problem, absolutely dry wood rheology offers experimental advantage. Franck (2021) suggests that more detailed picture of time-related influence is needed to understand this behaviour fully for this information is not accessible experimentally. Furthermore, Franck (2021) claims that measurements consisting of a wide range of temperature are easy to make but when speaking in term of time changes that occur in less than a second or when time span is a few weeks long, such measurements become complicated.

Burgers (1948), Burgers and Blair (1949) and Curtu et al. (2015) mentioned Burgers model presented by Eq. (1), which characterises rheological deformation of wood based on its behaviour in terms of time:

$$\varepsilon = \varepsilon_e + \varepsilon_{ei} + \varepsilon_c = \frac{\sigma}{E_1} + \frac{\sigma}{E_2} \left(1 - \varepsilon^{\frac{E_i}{\lambda_2}} \right) + \frac{\sigma^* t}{\lambda_1} \quad (1)$$
Where, ε - strain (%)
 ε_e - elastic strain (%)
 ε_{ei} - delayed elastic strain (%)
 ε_c - flow strain (%)
 σ - stress (MPa)
 E - Young's Modulus of material (MPa)
 t - time (s)/(h)/(days)
 λ - viscosity (Pa*s)

E,

E_

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E

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Yuan-rong et al. (2008) described Burgers body as a simple model that describes creep behaviour of wood and noted that elasticity, viscoelasticity and creep, as parts of wood creep, can be determined from this equation. However, he stated that it can only be applied to the initial and second stage of creep, and that it cannot be applied to the end and breakage stage. Schniewind and Barrett (1972) suggest that wood can be considered as a linearly elastic material under some conditions, and as a linear viscoelastic material under other conditions for the purpose of stress analysis. When wood is exposed to sufficiently high temperatures, MCs, and stresses, it starts to show nonlinear behaviour (Bach, 1965). Data provided by Echenique-Manrique (1969) on stress relaxation shows that there are signs of non-linearity even at low levels of initial strain, but that the degree itself is very small over a large range of initial strain values, which means that there is no reason to discard the concept of wood as a linear viscoelastic material. Following these claims, Franck (2021) suggests that the solution arose from the experimental findings, which shows that time and temperature of time-dependent processes have similar effects on the rheological properties of linear viscoelastic materials. Franck (2021) describes master curves as a helpful way for understanding rheological behaviour of a polymer: viscoelastic properties depend on two main variables (time and temperature), which are separated by the super-position process that expresses the properties in terms of a single function for each. The time dependence of the material at a constant reference temperature is shown by the master curves, while the variation of the shift factor with temperature shows temperature dependence of the viscoelastic properties. Sun and Frazier (2007) conducted research on small southern yellow pine (Pinus spp.) and yellow poplar (Liriodendron tulipifera) dry wood samples. They applied

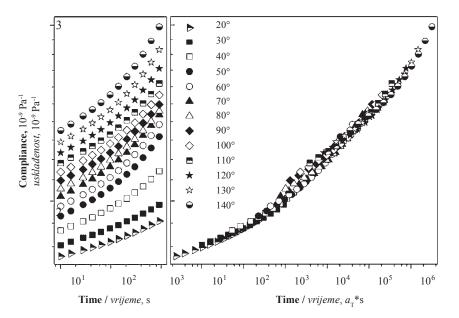


Figure 3 Raw creep compliance of southern pine from 20 °C to 140 °C (left). Master curve that results from simply shifting raw data in logarithmic time scale with a multiplicative shift factor (a_{τ}) (no vertical shifting employed) (right). (Sun and Frazier, 2007)

Slika 3. Neobrađeni podatci o puzanju uzoraka crvenog bora pri temperaturama od 20 do 140 °C (lijevo). Glavna krivulja koja je rezultat jednostavnog pomicanja neobrađenih podataka u logaritamskoj vremenskoj skali s multiplikativnim faktorom pomaka (a_r) (bez vertikalnog pomaka) (desno) (Sun and Frazier, 2007.)

creep bending stress in tangential direction over a $10 \,^{\circ}$ C to $170 \,^{\circ}$ C temperature range for 30 min. Their results of raw creep compliance are shown in Figure 3, where it can be seen that the master curve is not smooth, meaning that the temperature dependence of the creep relaxation is not uniform across a given temperature range.

They concluded that the principle of time/temperature equivalence was valid for the dry wood creep response over a given temperature range, but it was true only for specimens that received a prior thermal treatment from 100 °C to 170 °C (for 30 min), at which specimens lost all moisture. The creep compliance is an established metric of the rate at which strain increases for a constant applied stress of viscoelastic materials (Tweedie and Van Vliet, 2006).

Ferry (1980) suggests that smooth master curve appears when time and temperature effects are identical. When smooth master curve occurs in a material, it is considered thermorheologically simple, but in this case specimens are thermorheologically complex due to failure of achieving smooth master curve (Sun and Frazier, 2007). According to Franck (2021), the master curve shows the time dependence (in terms of frequency) of the material at a constant reference temperature; the temperature dependence of the viscoelastic properties is shown by the variation of the shift factor with temperature.

5 APPLICATION OF RHEOLOGY IN SOLID WOOD BENDING PROCESS 5. PRIMJENA REOLOGIJE U PROCESU SAVIJANJA CJELOVITOG DRVA

As already mentioned in the introduction, when solid wood is bent, it stretches on convex side and compresses on concave side. This results in convex side being longer than concave side and that difference in length causes stresses to accumulate. These stresses then tend to bring back solid pieces to their original form; this phenomenon is commonly called springback effect. The reason why wood is being softened is to restrict development of the mentioned stresses. During the bending process, it is desirable for wood to creep/bend more due to the nature of the final product. Both high temperature and MC influence creep properties as already described in previous chapters. That is the reason why wood is exposed to high temperatures and MC due to temporary reduction in MOE of wood (which is preferred for its easier bending).

Navi and Sandberg (2012) explained the purpose of plasticization treatments. To make the curve, wood needs to be sufficiently softened so it can withstand the necessary compressive deformation without fracture. Furthermore, a combination of heat and moisture is an effective way of softening wood as wet wood is more plastic than dry wood and hot wood more plastic than cold wood.

Sandberg *et al.* (2013) stated that the main reason for the difficulty in bending solid wood is the low strain to failure in tension (about 1-2 %). However, after wood is plasticized, it becomes more plastic or semi-plastic, and this means that it can be softened and formed to keep its shape after cooling. Wood compressibility is greatly increased in the longitudinal direction after plasticizing, as much as 30-40 %, although its ability to lengthen under tension is not significantly affected.

Zemiar et al. (1997) stated that plastic deformability of wood increases with the decrease in MOE. Thermal plasticizing is a process of exposing wood to temperature and moisture in order to increase the plasticity of wood, and its main target is a temporary change in the mechanical and physical properties of wood. According to Báder and Németh (2019), the best pliability during bending is achieved when the moisture content of pleated wood is close to its fibre saturation point. According to Taylor (2008), the best MC for bending is around 25 to 30 %, and around 2 minutes of steaming per millimetre of width. Plasticisation of wood can also be done with chemicals such as urea and liquid ammonia, but it will not be described because it is beyond the scope of this paper. Gáborík and Zemiar (1997) suggest that it is most desirable to optimize the degree of plasticity with the degradation of components of the lignin-cellulose matrix. Gašparík and Barcík (2014) suggest that softening lignin, which is the main component of the middle lamella, is an important part of plasticizing wood because lignin properties reflect wood plastic properties. While steaming is the most commonly used method of softening wood, many manufacturing companies use the combination of steaming and high frequency (HF) process to unite the heating, plasticizing and drying in a single sequence. Bent samples are partially dried in (HF) press after bending. After unloading, the press samples are further fixated with wooden or metal tools to keep them firm to prevent the spring-back effect during drying of samples.

As already mentioned in the introduction, extensive knowledge of wood rheology and wood viscoelastic properties and a lot of trial and error tests is required to bend wood properly. Beech is the most common species of wood used for bending because of its good bending properties and its common use in manufacture of furniture. Likewise, oak wood is also a desirable species for bending because it is also often used in manufacture of furniture but is more complicated to bend than beech. Considering that, as expected, most of the literature describes and gives data on beech wood (*Fagus sylvatica*) bending. While in essence the process of plasticizing oak wood by heat and moisture is the same as that of beech wood, parameters such as initial MC before

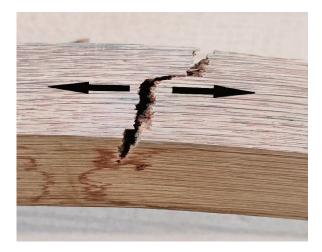


Figure 4 Transverse cracks on convex side (tension) (photo: Mikšik, 2021) Slika 4. Poprečne pukotine na konveksnoj strani uzorka (tenzija) (fotografija: Mikšik, 2021.)

steaming, temperature of steaming, duration of steaming and press parameters vary. Many factors affect wood bending; some of them are radius of bending, species, MC, thickness, and width of wood, steaming time, fibre direction and defects (Niemiec and Brown, 1995). Niemiec and Brown also claim that wood selected for bending must be defect-free and have straight fibres, but in practice it is not always the case since wood pieces with knots and curled fibres can also be bent properly. Wangaard (1952) conducted an experiment on loss of modulus of rupture for beech and oak wood bent at a radius of 20 cm and concluded that the loss was 32.1 % for beech and 26.1 % for oak. Furthermore, he also stated that, with the increase of bending radius, the strength loss of beech wood decreased. Büyüksarı and As (2012) concluded in their research that the density of bent samples of oak wood and beech wood increased with the decreasing bending radius, which is logical due to the compression on the concave side of samples. They also stated that the increase of density was greater in oak wood than beech wood for all bending radii.

According to Peck (1957), compressive failures can occur if the plasticized wood is compressed excessively, if stresses are concentrated because of some defects and if the lines of weakness encourage shear failure. According to Báder *et al.* (2019), compression in longitudinal direction induces changes in wood tissue, which results in better bendability.

On the other hand, Stevens and Turner (1970) suggest that controlling the length and longitudinal tensile strain in the vicinity of the convex surface of a wood specimen during a bending process is important, because too small end-force causes longitudinal tensile failure, while an excessively large end-force causes premature longitudinal compressive failure. The smallest radius of curvature for any piece of wood is reached when both the inner and outer surfaces are on the point



Figure 5 Structure collapse on concave side (compression) (photo: Mikšik, 2021)
Slika 5. Urušavanje strukture na konkavnoj strani uzorka (kompresija) (fotografija: Mikšik, 2021.)

of fracturing. Adjusting end-stops can be used to regulate the amount of end-force and thereby impose the maximum compression on the concave surface of a bend, without, at the same time, inducing stress on the fibres near the convex surface (to strain them beyond the limit). Some of the wood defects encountered in literature so far are transverse cracks on convex side, structure collapse on concave side, defects around knots, cross section cracks, surface splitting of layers, cracks due to current breakdown in HF press, longitudinal crack on both sides and discoloration caused by metal strap in contact with woods that contain tannin.

6 CONCLUSIONS 6. ZAKLJUČAK

It can be concluded that both temperature and MC affect wood creep. It is more affected by changes in MC than by constant MC. Time-dependent creep also affects wood but to measure it requires temperature, moisture content and other important variables to remain constant. It is hard to conduct creep experiments with increasing temperature because retaining constant MC at higher temperatures is very difficult due to changes in MC, which take place simultaneously with changes in the temperature. The sum of the elastic strain, viscoelastic strain at constant moisture content, free shrinkage/swelling strain, mechano-sorptive strain, and thermal expansion/contraction strain makes mechano-sorptive creep stain. In order to bend wood more easily, it needs to be plasticized (treated with high temperature and moisture) properly to avoid unwanted defects. Bending methods and parameters for beech have already been determined in detail, which cannot be said for oak. For these reasons, our further studies will focus on finding optimal drying mode and optimal bending parameters (MC, thickness,

width of wood, steaming time) for oak. Furthermore, experiments related to minimum radius for bending oak will be conducted. These parameters, if established properly, will have great industrial relevance for manufacturers that plan on producing furniture of bent solid oak wood in the future.

Acknowledgements – Zahvala

The article was published as part of the project "Development of innovative products from modified Slavonian oak" (KK.01.2.1.02.0031) by Spin Valis d.d. and partner University of Zagreb, Faculty of Forestry and Wood Technology. The total value of the project is HRK 55,064,343.84, while the amount co-financed by the EU is HRK 23,941,527.32. The project was co-financed by the European Union from the Operational Program Competitiveness and Cohesion 2014 - 2020, European Fund for Regional Development.

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Additive Technologies and Their Applications in Furniture Design and Manufacturing

Aditivne tehnologije i njihova primjena u dizajnu i proizvodnji namještaja

REVIEW PAPER

Pregledni rad Received – prispjelo: 27. 1. 2022. Accepted – prihvaćeno: 3. 10. 2022. UDK: 684.4 https://doi.org/10.5552/drvind.2023.0012 © 2023 by the author(s). Licensee Faculty of Forestry and Wood Technology, University of Zagreb. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license.

ABSTRACT • This paper deals with an overview of additive manufacturing and its segment - 3D printing, which is today rapidly and widely used (Agashe et al., 2020) for personal and high-capacity production. The paper discusses the possible positive factors such as small and personalized production series, cheaper design and production process, complex geometry, bionic structures (whose surfaces are complicated to make, and are copy of biological organisms) and negative factors such as lack of educated specialists and trainings. Those facts are affecting the implementation of these technologies in different segments of the design, product development and furniture production. The impacts of new technologies on the design and production of rapid prototypes and finished products in furniture industry are analyzed. The positive results of using additive manufacturing indicate that, in spite of minor obstacles and problems with connecting different production processes, additive production will have a significant place in the future of furniture design and production. The most important advantages of 3D printing is fast prototyping, one piece production, free form designing and the use of bio-based materials and their possibility of recycling.

KEYWORDS: *additive manufacturing; furniture modelling; 3D printing; furniture design; bio – based materials*

SAŽETAK • U radu se opisuje aditivna tehnologija 3D printanja koja se danas ubrzano i široko primjenjuje (Agashe et al., 2020.) u maloj i industrijskoj proizvodnji. U radu se opisuju mogući pozitivni čimbenici kao što su male i personalizirane proizvodne serije, jeftiniji dizajn i proizvodni proces, složena geometrija, bioničke strukture (površine složene za izradu, a kopije su bioloških organizama), ali i negativni čimbenici poput nedostatka obuke i educiranih stručnjaka. Navedeni čimbenici utječu na implementaciju tih tehnologija u različitim segmentima dizajna, razvoja proizvoda i proizvodnje namještaja. Analizirani su utjecaji novih tehnologija na dizajn i proizvodnju brzih prototipova i gotovih proizvoda u industriji namještaja. Pozitivni rezultati primjene aditivne tehnologije pokazuju da će ona, usprkos manjim preprekama i problemima povezivanja različitih proizvodnih procesa, imati važno mjesto u budućnosti dizajniranja i proizvodnje namještaja. Najvažnije prednosti 3D printanja jesu brza

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izrada prototipova, proizvodnja u jednom komadu, dizajn slobodnih formi i upotreba proizvodnih materijala te mogućnost njihova recikliranja.

KLJUČNE RIJEČI: aditivna proizvodnja; modeliranje namještaja; 3D printanje; dizajn namještaja; prirodni materijali

1 INTRODUCTION

1. UVOD

Additive manufacturing (AM) is a specialist and complex manufacturing niche. The development of additive manufacturing has huge influence on the production process in the furniture sector. 3D printing offers almost unlimited possibilities for creativity. In terms of technology, it is completely different from the traditional subtractive type of production (Kietzmann et al., 2015) and gives it a lot of advantages especially in design. The possible uses of 3D printing are: fast prototype, one piece production, complete product manufacturing, education and testing of ergonomics. Therefore, it affects production from the initial stages - from base model to the final stages of finished products. To be able to use additive manufacturing, it is necessary to think differently. It is necessary to determine the purpose of using available technology and material to be able to achieve full benefits of additive technology. There are doubts about the meaning of the terms AM and 3D printing. Both terms are often used interchangeably, and they are considered synonymous by some authors (Weller et al., 2015). On the contrary, other authors consider AT and 3D printing to be two different terms (Gibson et al., 2010; Mellor et al., 2014). As stated by Ford et al. (2016) AT is not just one technology but covers a range of technologies. Both, the term additive manufacturing, and the term 3D printing are used when talking about three-dimensional printing of prototypes or real pieces of furniture. Because of almost unlimited application, the manufacturing has entered the era of artificial intelligence, digitalization, and networking (Xu et al. 2019). The versatility and more use of AM are confirmed by Berman (2012).

Regardless of whether it is the production of furniture, clothing (Sun and Zhao, 2017), medical implants (Schubert *et al.*, 2013), cars, jewellery, or artificial organs (which use layers of human cells), this technology is in rapid development and is already widely used (Ngoa *et al.*, 2018). Recent advances in research and development have already enabled its application for private use in households. Additionally, also design education and courses are adapted to the new compatible technologies that include 3D rapid prototyping (Yulvan, 2020). 3D printing enables consumers around the world to transform digital data into physical products and vice versa, thus facilitating access to innovation (Rindfleisch *et al.*, 2017). This allows the user to progress in the process of developing new products, in the way that he/she can use digital tools, such as 3D scanners and 3D printers, to collect and/or generate data, and in the end to create and offer a range of new products on the market. A positive feature is also that 3D printing allows anybody with lower manual skills to produce very demanding and complicated products (as craftsmen once did), which makes such furniture or furniture parts unique.

2 ADDITIVE MANUFACTURING AND OCCURRENCE OF 3D PRINTING

2. ADITIVNA PROIZVODNJA I POJAVA 3D PRINTANJA

2.1 Historical review of additive manufacturing

2.1. Povijesni prikaz aditivne proizvodnje

Production of 3D printers began in the 1980s with the development as an auxiliary simple technology for making rapid prototypes. As well as moulding and welding, printing is also considered as additive manufacturing due to the process of adding material, which creates a new body. Previously applied procedures are based on cutting and removing material. In the period from early 1980s to 1988, various 3D printing processes were designed thanks to the high demand for rapid prototyping. The first ideas and key detailed information for creating the process of stereolithography were given in the 1980s by the Japanese physician Hideo Kodama, who invented the laser approach to curing with one laser beam using photopolymerization.

In the same years, Oliver de Witt together with Jean Cloude Andre worked on research and invention of 3D printers, and in 1986 a patent was granted for the stereolithographic process. The development was not completed due to the uncertainty of the application of this technology. At the same time, Charles Hull successfully patented stereolithography, *i.e.*, solidification or resin printing. It is an ongoing process consisting of curing or solidifying a photosensitive polymer in contact with an ultraviolet laser and an adequate resin (Wong and Hernandez, 2012).

2.2 Preparation of CAD models for 3D prototyping

2.2. Transformacija CAD modela za izradu 3D prototipa

A suitable computer program, equipment and adequate materials are needed for 3D printing (Kralj,

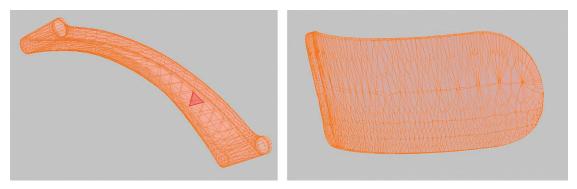


Figure 1 STL file format of chair backrest made of lines and triangles; (Jarža, L.: 3D model photography, 2021) **Slika 1.** STL datoteka modela naslona stolice sastavljenoga od linija i trokuta (slika 3D modela: Jarža, L., 2021.)

2017). The first step is to create the wanted model in the program, then the model needs to be converted into a readable device format for 3D printer. The basic 3D model is converted in STL format to get a readable format that consists of lines and surfaces combined into triangles. With terms stereolithography, the abbreviation STL standing for "Standard Triangle Language" and "Standard Tessellation Language" is also used. This is a universal format used to transfer information from modelling programs and can be exported from and to most CAD software packages. The process of creating a STL file is to convert the continuous geometry in a CAD file into triangles or a list of triplet coordinates x, y and zcoordinates and a normal vector into triangles (Wong and Hernandez, 2012). Each triangle, which represents a given area, is characterized by three vertices and the corresponding unit normals (Pascucci et al., 2018). The STL format only supports a three-dimensional description of surface geometry without generating print information such as texture or colour (Hiller and Lipson, 2009). Depending on the complexity and size of the model, each vertex is part of three or more triangles and could be read as a 3D body. Szilvśi-Nagy and Matyasi (2003) provide a method for detecting deficiencies in the representation of the surface when using the STL format. Figure 1 shows a 3D body of a chair backrest which is made of the above-mentioned triangles. They are an integral part of a model and important for further usability of the files. If the link is "split", it is not visible to the printer when reading the format. For the print format to be functional, it must be non-specific or adaptable to each printing machine and have the ability to read all elements of the model, including models default colours and materials. Also, because of the rapid advancement of this technology, the format should be "up-to-date" or readable regardless of computer software updates. Groenendyk and Gallant (2012) write about the challenges of preparing files for printing, and Wu and Cheung (2005) about the possibilities for enhancing the printing format.

The advantages of this format (Hiller and Lipson, 2009) include simplicity, portability (format), and low-requirements of working computer memory, while the disadvantages are geometry leaks, incompatibility with colours and materials, poor scalability, and lack of auxiliary information for printing. Based on these disadvantages, they suggest new solutions for use in 3D printing. Their proposal is XML based and AMF format (they give precision, multiple materials and multiple colours at once) but the success of a file format depends on its adoption by users. Nevertheless, today with the development of new materials and hardware, these disadvantages have been reduced, including the lower cost, and better properties of materials, more colours, *etc*.

3 TECHNOLOGY OF 3D PRINTING 3. PRIMJENA TEHNOLOGIJA 3D ISPISA

Additive manufacturing could be divided into two categories: regarding the material required for printing or the type of technology. Thus, the best known are SLS (selective laser sintering); SLM (selective laser melting); EBM (electronic beam melting which uses the powder materials for printing); SLA (stereolithography - the first invented using photopolymers and UV light); LOM (laminated object manufacturing - not so used because of the amount of needed material and difficult removal of residual material, INKJET, LENS (laser engineered net shaping); FDM (fused deposition modelling) and others described by (Kariž et al., 2017). There are many technologies for a variety of applications, but for the furniture production and product development, FDM is the most frequently used due to the possibility of joining the wood with polymers. This method uses the thermoplastic polymers, since they are compatible with clay, metal, wood, and others. This technology uses heat for energy and has a lot of advantages including cheap printers, simultaneous printing with multiple materials, etc. SLS uses powder, resulting in high accuracy, strength, stiffness, and high density and can also be used in furniture production. Figure 2 shows the examples of FDM and SLS printed models.



Figure 2 Examples of printed models: a) SLS technology printed models (https://3dprint.com/); b) FDM technology printed model (https://www.printyourmind3d.ca/products/print-your-mind-3d-woodfill-filament?variant=43074437318); c) FDM technology printed model (https://www.forust.com/)

Slika 2. Primjeri isprintanih modela: a) model ispisan SLS tehnologijom (https://3dprint.com/); b) model ispisan FDM tehnologijom (https://www.printyourmind3d.ca/products/print-your-mind-3d-woodfill-filament?variant=43074437318); c) model ispisan FDM tehnologijom (https://www.forust.com/)

4 APPLICABLE MATERIALS IN 3D PRINTING 4. MATERIJALI ZA PRIMJENU U 3D PRINTANJU

Innovation and progress mean innovation in both technology and application of new materials. Additive manufacturing printing materials are available in different forms as powder, filaments, pellets, granules, resins, etc. The choice depends on the application and properties. Various materials are available on the market, such as polymers, metal-based materials, wood based, ceramic, concrete, etc. Some of these polymers are (polylactic acid), (*2012), ABS (acrylonitrile butadiene styrene), PVA (polyactic) PC (polycarbonate), nylon, and polystyrene.

Frequency of use of different materials depends on the requirements of users and the purpose. Polylactic acid or polylactide (PLA) is a widely used plastic filament material, and it is biodegradable and adaptable to all FDM printers. ABS is currently the most flexible material for various purposes available on the market. It is used in the form of filament and is suitable for melting and hardening, also available in all colours. Nylon or polyamide is used in powder form or filament form. Ashraf et al. (2018) write about metals and stainless steel in 3D printing. There is also the possibility of printing with powder such as ceramic (Travitzky et al., 2014) or concrete (Nadarajah, 2018). Saad (2016) and Rael (2018) indicate the recommendations for choosing the material depending on the technology used. San Fratello and Rael (2020) describe how to use innovative materials like clay and salt or even soil for 3D printing.

Although synthetic polymers have to be recycled, recycling rates are very low (Garmulewicz *et al.*, 2018). There is an interest for using other sustainable and environmentally friendly materials (Sabbatini *et al.*, 2021). Wood residues make a high percentage of

waste in wood industry. It can be useful as a material for further use and recycling. It can be used for making wood filaments for 3D printing and not only for ecological reasons (Pringle *et al.*, 2017).

From the designer's or even the customer's point of view, 3D printed product should look like wood and it is less important whether the printed model is real wood. However, for the wood industry, the possibility of recycling wood waste is more important than the mentioned aesthetic impression. Additive manufacturing would be one of the most positive ways of disposing wood waste and wood mass, which mainly ends up in thermal power plants. Numerous studies have shown a positive trend towards lignocellulosic fillers that are increasingly replacing synthetic fibres, emphasizing their mechanical properties, such as low density, good thermal insulation, low cost, and availability (Ayrilmis et al., 2019). Cellulose does not cause allergies, and withstands high processing temperatures between 175-250 °C needed for 3D printing. Such printed elements have the possibility of further processing, carving, sanding, painting, etc. With the use of wood waste, the use of filament of organic origin can also further increase the environmental benefits (Das et al., 2021). In addition to being widely available and renewable material, reusing and recycling would give the waste a new purpose and thus gain in value, which is of extreme importance in the context of the world's climate crisis.

Wood filaments are made by mixing wood sawdust, powder, or particles with polymers, after which the material is extruded. Wood particles must be of a certain fineness and texture to enable its mixing into the polymer (Wimmer *et al.*, 2015). Extrusion deposition and polymer binding are layering technologies that use wood materials (Das *et al.*, 2021). An overview of the problems and advantages of using wood in 3D printing is explained by Gardner and Wang (2019).

The research of Wahab et al. (2013) confirms the sustainability of wood waste - sawdust for use with 3D printing manufacturing with the fact that the fineness of the powder increases the model surface quality. They also state that increasing the sawdust content reduces the properties of hardness and dimensional accuracy. Several authors explore the possibilities of using wood waste for 3D printing depending on its ratio (%) in the filament (Kariž et al., 2016, 2018b). The research was conducted on residues of beech wood (Fagus sylvatica) (Kariž et al., 2017; 2018a; Ayrilmis et al., 2019; Rosenthal et al., 2018); Chinese poplar (Populus lasiocarpa) - Chinese aspen (Populus adenopoda) (Tao et al., 2019), spruce (Picea abies) (Henke and Treml, 2013); European poplar (Populus), (Bi et al., 2018b); pine (Pinus) (Le Guen et al., 2019), (Wechsler and Hiziroglu, 2007) and olive wood (Olea europaea) (Smardzewski et al., 2018).

Kariž *et al.* (2017) investigated the impact of beech particles present in wood content on 3D printing. The wood particles in the filament reduced the density of the printed parts due to the lower density of the wood compared to synthetic polymers. The surface of the printed parts without the addition of wood is smoother and without cavities. The increase of wood particles increased the roughness of the printed element. Nevertheless, their results showed that wood can be used as a component in 3D printing materials and that a small percentage of wood dust strengthens the filament structure by up to 10 %. But also, with increasing percentage of wood dust, tensile strength decreases.

Between the layers, certain stresses may occur due to shrinkage / swelling *e.g.*, stresses during cooling of molten polymers and bending of the product (Kariž *et al.*, 2017). The influence of moisture content from wood powder and air on 3D printed wood was described by Kariž *et al.* (2018b) and Ayrilmis *et al.* (2019). Grujović *et al.* (2016) prove that 3D printed furniture connecting elements are useful for wide application. That would reduce the cost of making small series of complex furniture elements. Research on the production of extruded wood concrete, whose composition is limestone, cement, and untreated coniferous wood chips, has also shown positive results (Henke *et al.*, 2016).

Considering that the 3D printing is a relatively new technology, the production of adequate wood filament requires a lot of research focusing on issues such as types of wood used, particle size, type of natural polymers, *etc.* Table 1 presents the advantages and disadvantages of wood in the application of additive manufacturing and 3D printing based on previous research (Wimmer *et al.*, 2015; Henke, *et al.*, 2016; Bi *et al.* 2018a; Das *et al.*, 2021; Wahab *et al.*, 2013; Ayrilmis *et al.*, 2019; Rosenthal *et al.*, 2017; Kariž *et al.*, 2017).

5 IMPLEMENTATION OF AM IN FURNITURE PRODUCTION

5. IMPLEMENTACIJA ADITIVNE PROIZVODNJE U PROIZVODNJI NAMJEŠTAJA

The choice of adoption of new technologies in a company depends on the needs and priorities of the production department. There are good reasons for and against the introduction of 3D printing. Aydin (2015), based on a real projects overview, emphasizes innovation as key in the development of this industry. For small-and medium-sized companies' benefits, challenges and business factors have significant influence on the adoption of the additive manufacturing (Kulkarni *et al.*, 2020). According to the production department, technology and costs are the most important implementation factors, while the marketing and development department consider future market placement as the priority, followed by the impact on the environment (Yeh and Chen, 2018). Pascucci *et al.* (2018)

Table 1 Advantages and disadvantages of wood used in 3D printin	g
Tablica 1. Prednosti i nedostatci upotrebe drva u 3D printanju	

Wood filaments in 3D printing / Filamenti na bazi drva u 3D printanju		
Advantages / Prednosti	Disadvantages / Nedostatci	
Ecological aspects, recyclability, and biodegradability	Technical difficulties when printing -	
ekološki aspekti, neštetnost za okoliš,	filling and blocking nozzles with wood powder	
mogućnost recikliranja i biorazgradivost	punjenje i blokiranje sapnica drvnim prahom	
Reducing the cost of purchasing materials possibility of	Higher percentage of wood reduce mechanical properties,	
making it inside the factory	and surface quality	
smanjenje troškova nabave materijala za ispis, mogućnost	veći postotak drva smanjuje	
izrade unutar tvornice	mehanička svojstva predmeta i kvalitetu površine	
Sustainability of use of 3D printing manufacturing	Low wood compatibility with printing – materials	
održivost proizvodnje 3D printanja	niska kompatibilnost drva s ostalim materijalima	
Easy workability, sanding, colouring of the printed element	Rougher surface	
laka obradivost ispisanog elementa	hrapavija površina	
Aesthetic characteristics (appearance of real wood)		
estetska obilježja (izgled pravog drva)		

determined the key factors common to all productions in terms of implementation of additive manufacturing in production processes. These are: the purpose of use, company size, time of use, type of material and transition from conventional production techniques. Additive manufacturing must be interconnected with market and production needs. The plan for the implementation of additive manufacturing in the actual production is described by Mellor et al. (2014), and as the main characteristics for the introduction of technology they state: the degree of adaptability, increased functionality through design and the possibility of producing small quantities. The reasons against are the high price of large-scale printing machines despite the use of cheap recycled material and the slowness of the technology, e.g. 24 hours for one backrest.

The mentioned value can be associated with the increase of competitiveness. The product can be quickly improved with new properties or design details. Every aesthetic supplement that makes the product recognizable and more interesting increases the interest of customers and the product gains in value. Most often, these are examples of furniture parts that are not available on the market, and due to their design, they must be special and different. The possible uses in production that could be important for the implementation are: full size prototyping and printing furniture in large scales; printing elements of furniture; replacement of furniture pieces; ergonomy testing; printing furniture joints, reverse engineering and rapid prototyping. These few examples are shown in Figure 3. The example for the replacement parts is presented by 3D printed elements shown in Figure 3d. It describes the furniture part whose solution did not exist on the market and a new one was created. Innovative elements are very suitable for 3D printing (Podskarbi et al., 2016; Krzyżaniak et al., 2020). Those made with 3D printing may enrich the product not only with an aesthetic value but also with a new function. They are printable with different materials, interchangeable, available in all places where there is the possibility of 3D printing, even in households. This reduces the cost of ordering and delivery time and allows the possibility of personalization. Users who want to be unique are the most common target group to be recipients of such furniture. Interesting examples of 3D printed elements incorporated into the design of furniture are shown in the works of Pandolfo (2016) and Jarža (2016).

Aiman *et al.* (2020) compare furniture joints made of existing material and traditional production type and Fused Deposition Modelling (FDM) fabrication method. Results showed that the elements made

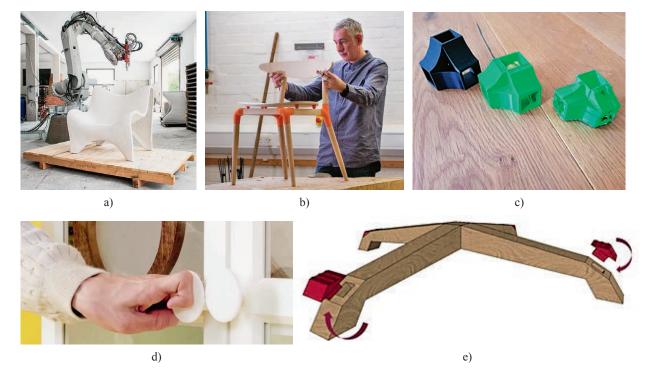


Figure 3 Uses of 3D printing in production: a) 3D printed large scale chair (https://parametrichouse.com/3d-printed-furniture/); b) Part of furniture (https://ultimaker.com/learn/3d-printing-in-furniture-design); c) Joints development (Prekrat, S., photography); d) Parts for testing of ergonomy (https://3dprinting.com/3d-printing-use-cases/how-the-furniture-industry-benefitsfrom-3d-printing/); e) Replacement of parts (Jarža, L., photo of rendered 3D model)

Slika 3. Primjena 3D ispisa u proizvodnji: a) 3D printana stolica stvarne veličine (https://parametrichouse.com/3d-printedfurniture/); b) dijelovi namještaja (https://ultimaker.com/learn/3d-printing-in-furniture-design); c) razvoj elemenata za sastavljanje namještaja (fotografija: Prekrat, S.); d) elementi za ergonomsko testiranje (https://3dprinting.com/3d-printing-use-cases/ how-the-furniture-industry-benefits-from-3d-printing/); e) zamjenski dijelovi (fotografija izvedenog 3D modela: Jarža, L.)



Figure 4 3D printed element created by reverse engineering (https://3dprint.com/45399/marie-antoinette-museum-chair/) Slika 4. 3D printani elementi kreirani povratnim inženjeringom (https://3dprint.com/45399/marie-antoinettemuseum-chair/)

from waste materials printed by FDM method are fully functional and of suitable quality. This way of reusing and recycling the residue from production (which is thrown away in large quantities) could be used in production of filaments for 3D printers.

In traditional (convectional) production some products and elements have become a thing of the past, they are no longer produced, technologies are outdated or not in line with the new trends. 3D print is part of reversible engineering. The example of the application of this technology is the restoration of furniture elements (Figure 4). It includes three-dimensional scanning of elements, digitization of models and creation of documentation for CAD programs that support the format for 3D printing (Celent *et al.*, 2016). The advantage of using this manufacturing, which includes printing of specific parts, also allows the production of a single piece, which does not affect the economic aspects of the remaining production.

Virtual furniture design is another activity developed from the additive manufacturing and it includes 3D modelling of furniture for architecture and interior design. This allows useful interactive participation of the customers in the selection process when buying furniture (Lin and Hsu, 2004; Chua *et al.*, 1999).

Due to the commercialization of the 3D printers, many smaller manufacturers decide to implement it in accordance with their financial and spatial possibilities. Advances in information science enable them to use services in the data cloud, which solves several limitations, including the problem of lack of professional staff and the implementation of personalized individual production (Xu *et al.*, 2019; Krsnik, 2019). This is good because 3D printing is a way of production that occasionally needs prototyping or parts manufacturing services. That would enable them to avoid the cost of implementing new technology and training of workers. Services in the cloud connect three types of users: manufactures, designers, and consumers. The advantages of this possibility include reduced disruption to the daily flow of production. Rayna *et al.* (2015) explain the market developments for design, 3D printing, service, and group collaboration with users on digital platforms.

6 ADVANTAGES AND DISADVANTAGES OF ADDITIVE MANUFACTURING 6. PREDNOSTI I NEDOSTATCI ADITIVNE PROIZVODNJE

The need of fast prototypes, new design elements and innovative furniture is growing rapidly, and competition is becoming fierce. Shehata* wrote about the advantages of 3D printing. In recent years, design has become a widespread activity that launches many new solutions around the world daily. In order to be competitive, it is necessary to react quickly to launch a new product on the market and to be faster than the competition. This includes prototyping, testing, finishing and market presentation. This is where this 3printing comes forward. Top et al. (2019) point out that 3D printing of parts, assemblies, and segments helps to achieve this goal. This method allows the creation of very complex shapes with high precision and maximum material savings, and an additional plus is that 3D models and print data once prepared can be easily and quickly modified when needed. Ngoa (2018) describes the flexibility of this technology, Tofail et al. (2018) its benefits and Grujović et al. (2016b) write about its cost-effectiveness.

Designing new solutions and products using fast prototypes is now much simpler. In short time, it is possible to change dimensions, materials, etc. on the basic 3D model, depending on the requirements, and print a new sample, if necessary. With rapid prototyping, it is much easier to control the ergonomics, dimensions, product stability, usability and other characteristics that are difficult to assess on-screen. According to Pascucci et al. (2018), this manufacturing gives freedom of design expression, reduces the need for special tools and the cost of production of individual specific parts and components of complex geometric shapes. Murmura and Bravi (2018) quote advantages for creative manufacturing industries and those that develop furniture. This includes reducing the time of product launch on the market, main limitations of the inadequacy of manufacturing and the need for more educated employees, increasing at the same time the freedom of design expression. Opposite of that, Schniederjans (2017) and Ford et al. (2016) claim that, despite all the advantages of this technology, it has not yet reached high levels of adoption mainly because of regulatory and legal issues, high initial investment costs, increased electricity consumption compared to traditional methods and needs for new skills and competencies.

Holzmann *et al.* (2017) outline the positive aspects of 3D printing as opportunity from the perspective of small producers. Opposite of this, Bogers *et al.* 2016 conclude that 3D print can have a devastating impact on business models because of the changes in the production processes that negatively affect the processes of supply and transport, which will no longer be needed because of possibilities of self-printing (Öberg, 2018). Jumaah (2018) also writes about the consequences of localizing AM on supply, transport and transportation activities and states the positive effects on the environment and savings in financing urban infrastructures. Holzmann *et al.* (2020) explore new business models and explain how companies currently underutilize the key benefits of 3D manufacturing.

Ryan *et al.* (2017), Oettmeier and Hofmann (2016), Rogers *et al.* (2016), Durach *et al.* (2017) are convinced that, by the implementation of additive manufacturing, the systems of transport and supply ac-

tivities will be excluded from the whole process precisely because then manufacturers will be oriented to reduce unnecessary costs by introducing new machines that could improve production and products and make something they ordered. Improving production and products, in that case, would mean added value to the factory and a better product for the market.

Reducing costs and time, reducing environmental pollution, and even reducing injuries at work are listed by Sakin and Kiroglu Caner (2017) as the main advantages of the implementation of this technology in construction companies. This is applicable to the wood industry as well because the machines can do jobs instead of workers. Implementation of this technology in furniture production processes would contribute to significant savings in some areas, including the usage of only the amount of material that is needed for their design. This means to use less resources and thus reduce waste, to create more innovative designs because 3D

 Table 2 Advantages and disadvantages of implementations of additive manufacturing

 Tablica 2. Prednosti i nedostatci implementacije aditivne proizvodnje

Implementation of additive manufacturing / Implementacija aditivne proizvodnje		
Advantages / Prednosti	Disadvantages / Nedostatci	
Early error detection during design process prepoznavanje i uklanjanje pogrešaka u procesu oblikovanja	Lower product strength, delamination may occur during loading / manja čvrstoća proizvoda, mogućnost pojave raslojavanja	
Check of product assemblies / provjera sastavljanja sklopova proizvoda	Possible lower mechanical properties of prototypes depending on filament quality and technology / moguća lošija mehanička svojstva prototipova, ovisno o kvaliteti filamenta i o tehnologiji	
Making individual or unique products izrada pojedinačnih proizvoda ili unikata	Vanishing of existing traditional mass production plants zatvaranje postojećih tradicionalnih proizvodnih pogona za serijsku proizvodnju	
Making of 3D models and visualizations izrada 3D modela i vizualizacije	Lack of professional staff and educated training nedostatak stručnog osoblja i adekvatne obuke	
Digital storage of the model, quick modification and adaptation to a new product / <i>digitalna pohrana modela i brza izmjena i</i> <i>prilagodba novom proizvodu</i>	Sustainability of production processes / održivost proizvod- nih procesa	
The user / customer personally participates in the process of creation / <i>osobno sudjelovanje korisnika/kupca u procesu kreiranja</i>	Lower 3D printer speed compared to conventional productions / sporost 3D pisača u usporedbi s konvencion- alnim proizvodnjama	
Rapid prototyping brza izrada prototipa	Lack of knowledge of 3D technology industrial printing and application / <i>nedovoljno poznavanje industrije 3D</i> <i>tehnologije ispisa i primjene</i>	
Quick production of devices or parts shortens delivery time brza izrada priručnih alata ili dijelova, što skraćuje vrijeme isporuke	Limited number of materials and their availability ograničen broj materijala i njihova otežana dobavljivost	
Reduced production costs (instead of ordering from other factory) and thus reduce cost of transport, storage and subcontracting services / smanjeni troškovi proizvodnje (umjesto naručivanja), čime se smanjuje i trošak prijevoza, skladištenja te kooperantskih usluga	More expensive than conventional construction due to high cost of 3D printers / proizvodnja skuplja od konvencion- alne gradnje zbog visokih troškova 3D pisača	
Use of direct production - reducing the number of operations primjena izravne proizvodnje – smanjenje broja operacija	3D printers can be too big or they are difficult and expensive to install in place / 3D pisači mogu biti veliki i stoga ih je teško i skupo instalirati na određeno mjesto	
The possibility of making products or parts in the "house" privately or in own factory / mogućnost izrade proizvoda ili dijelova "u kući", privatno ili u vlastitoj tvornici		
24/7 technology management / upravljanje tehnologijom 24/7		

printing can achieve shapes that conventional technologies cannot achieve with lower labour and transport costs and thus enable on-site application. The negatives could be the lack of technical staff, professional staff and workers on printing machines (because some machines require an extra worker). The offer of 3D manufacturing on the market cannot yet replace traditional production of higher capacity but has an impact because the traditional as well as AM face depreciation of machine and labour cost (Steenhuis and Pretorius, 2016; Attaran, 2017). Achillas *et al.* (2017) consider it as an excellent complement to small and micro productions that achieve competitiveness through their application. Table 2 lists the mentioned advantages and disadvantages of additive manufacturing.

Table 2 presents advantages and disadvantages of wide use of additive manufacturing in different production sectors. It should be emphasized that the disadvantages show daily a decreasing trend. Lots of them have already been improved and some problems will be corrected in favour of AM in the near future. The mentioned disadvantages and possibilities can be divided into several groups. They involve the development of the materials, development of printers, reducing costs (of hardware, printers, software, materials, work, or defined properties of the future product) and better education of employees. Development of printers will reduce printing costs and secure better and cheaper hardware on the market. Development of materials includes improved properties of the printed model and the materials themselves, possibilities of using more colours and their combinations, more available materials, and filaments, even combinations of different materials. Education means more education from primary to higher education, also the implementation of education in regular schooling or additional lifelong learning courses, the so-called lifelong learning. Those properties will enable the wide use, which is the goal of this technology.

7 CROATIAN FURNITURE MANUFACTURER NEEDS 7. POTREBE PROIZVOĐAČA NAMJEŠTAJA NA HRVATSKOM

TRŽIŠTU

In Croatia, the wood industry and furniture production do not keep up with new technologies. It can be said that at this time the implementation of additive manufacturing is still slowly progressing, but there are possibilities for the development. 3D technology implementation in solid wood furniture factories depends on the primary interest of the factories, their placement on world markets and their technological capabilities.

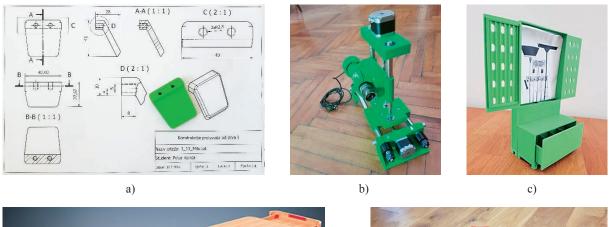




Figure 5 a) Engineering education (Prekrat, S., photography, 2022); b) Devices for research projects (Prekrat, S. photography, 2022); c) Product presentation (Prekrat, S., photography, 2022); d) Creating innovations (Prekrat, S., photography, 2022) Slika 5. a) Inženjersko obrazovanje (fotografija: Prekrat, S., 2022.), b) uređaji za istraživačke projekte (fotografija: Prekrat, S., 2022.); c) prezentacija proizvoda (fotografija: Prekrat, S., 2022.); d) stvaranje inovacija (fotografija: Prekrat, S., 2022.)

The need for rapid prototyping and new design solutions stems from the wishes to conquer the market with novelties. Only a few such factories show good examples and results, and the examples are shown in Figure 4. The other barriers include the procurement of new technologies and software that would be compatible with the existing machines. For some factories, this means a complete reconstruction and renewal of production and the design of new production processes.

For a greater degree of use of 3D printing in the design and manufacture of furniture, the problem is insufficient knowledge of 3D modelling as a basis for 3D printing. Still, there is progress. As part of the study program at the Faculty of Forestry and Wood Technology, University of Zagreb, students make models of furniture or its parts. Within this program, students use 3D print in product development and innovation, and they manufacture parts for devices in research projects (Figure 5).

Thus, the 3D printing is for now used just for prototyping and product development and not for big series and large furniture (Figure 6). While turning to ecological materials, wooden furniture is a trend on the domestic market, and ecological awareness when buying wooden furniture is still moving in small steps. At present, the companies that are already aiming at the foreign markets could benefit most from this technology.

Rapid prototyping also includes technological process with simple manufacturing (example is small printers and machines for fast prototyping) that most producers could afford and that would greatly support the development process of production or marketing. Positive factors of faster work process, reducing the cost of raw materials spent on the production of test prototype elements, and use of technology only (noninterference of regular production process, where workers would continue to work on their usual positions in production) would certainly show results.

8 CONCLUSIONS

8. ZAKLJUČAK

Today, additive manufacturing could speed up production (in case of rapid prototyping, one piece of unique furniture, alternate pieces of machines, etc.), reduce the cost of the design process and increase product quality. The number of disadvantages is much smaller than advantages. With such manufacturing, it is possible to make small production series, cheaper products, complex geometry, and bionic structures. It allows design freedom of making any shapes or furniture parts and make personalized products for a variety of customers. Additive manufacturing allows optimization of product performances. 3D printing has increased implementation in different fields of furniture production starting with making fast prototypes to final functional products or furniture parts. Figure 7 shows good examples of the mentioned advantages. Also, accelerated development of applied materials, improvement of technologies and reduction of hardware cost in additive manufacturing results in greater application in the design process and furniture manufacturing. Using natural polymers allows green production and possibility of recycling. All this gives the product new added value and lots of possibilities in product development.

Acknowledgements – Zahvala

The article was published as part of the project "Development of innovative products from modified Slavonian oak" by Spin Valis d.d. and partner Faculty of Forestry and Wood Technology, University of Zagreb. The total value of the project is HRK



Figure 6 Example of backrest product development (rapid prototyping) in Croatian furniture manufacturing; (Jarža, L., photography, 2022)

Slika 6. Razvoj proizvoda (brzog prototipa) na primjeru elementa naslona u proizvodnji namještaja (fotografija: Jarža, L., 2022.)



Figure 7 Good examples of 3D printing in furniture manufacturing (https://www.3dnatives.com/en/3d-printed-furniture-130220194/)

Slika 7. Dobri primjeri 3D ispisa u proizvodnji namještaja (https://www.3dnatives.com/en/3d-printed-furniture-130220194/)

55,064,343.84, while the amount co-financed by the EU is HRK 23,941,527.32. The project was co-financed by the European Union from the Operational Program Competitiveness and Cohesion 2014-2020, European Fund for Regional Development.

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Drvo topole *Populus* spp.

OPĆENITO O VRSTI

Topole su članice botaničkog roda Populus spp. i porodice Salicaceae. Istoj porodici pripadaju i vrbe (rod Salix). Rod Populus obuhvaća između 22 i 75 vrsta, ovisno o primijenjenoj taksonomskoj klasifikaciji, ali najčešće se govori o približno 30 različitih vrsta. Nadalje, rod topola dijeli se na šest sekcija: Abaso Ecken (meksičke topole), Aigeiros Duby (crne topole), Leucoides Spach. (močvarne topole), Leuce Duby (jasike i bijele topole), Tacamahaca Spach. (balzamne topole) i Turanca Bunge (topole aridnih područja i tropske topole). U Europi su tim imenom uglavnom obuhvaćene ove vrste: bijela topola (Populus alba L.) (njem. Weisspappel, engl. white poplar, franc. peuplier blanc, tal. pioppo bianco); crna topola (Populus nigra L.) (njem. Schwarzpappel, engl. black poplar, franc. peuplier noir, tal. pioppo nero) i trepetljika (Populus tremula L.) (njem. Aspe (Zitterpapel), engl. aspen, franc. peuplier tremble, tal. pioppo tremolo). Topole su široko rasprostranjene diljem sjeverne hemisfere, u borealnim i suptropskim zonama te u planinskim i poplavnim područjima.

Osim velikoga uzgojnog potencijala topola, drvo te vrste ima široku primjenu. Raspon proizvoda od topolovine vrlo je širok, osobito u području proizvodnje papira, pulpe i kartonske ambalaže, ali i u proizvodnji drvnih ploča i furnira, drvene ambalaže, šibica, bioenergije, ogrjeva i dr. Aktivnosti na podučju konzervacije genetskih izvora topole u svijetu, poglavito bijele i crne topole, provode se u sklopu mreže EUFORGEN. Topola nije na popisu ugroženih vrsta međunarodne organizacija CITES, s tim da se *P. alba* i *P. tremula*, prema IUCN-u, smatraju vrstama najmanje zabrinjavajućeg opstanka, dok za *P. nigra* podatci nedostaju. Samo se *P. caspica* navodi kao ugrožena vrsta.

VAŽNOST UZGOJA TOPOLA

U današnje vrijeme u svijetu je pojačano zanimanje za uzgoj različitih vrsta i križanaca topole. Neki od razloga jesu organska svojstva i ekonomska vrijednost te vrste: brz rast i sposobnost proizvodnje drva prikladnoga za tehničku uporabu već nakon rotacije od 20 godina; izvor sirovine zadovoljavajuće kvalitete za

GENERAL INFORMATION ON SPECIES

Poplars are members of the botanical genus Populus spp. and the Salicaceae family. Willows (genus Salix) also belong to the same family. The genus Populus includes between 22 and 75 species, depending on the taxonomic classification used, while the most common are about 30 different species. Furthermore, the genus Populus is divided into six sections: Abaso Ecken (Mexican poplars), Aigeiros Duby (black poplars), Leucoides Spach. (swamp poplars), Leuce Duby (aspens and white poplars), Tacamahaca Spach. (balsam poplars) and Turanca Bunge (arid and tropical poplars). In Europe, the following species mainly come under this name: white poplar (Populus alba L.) (Weisspappel (GER), white poplar (EN), peuplier blanc (F), pioppo bianco (I)); black poplar (Populus nigra L.)(Schwarzpappel (GER), black poplar (EN), peuplier noir (F), pioppo nero (I)) and aspen (Populus tremula L.) (Aspe (Zitterpapel)(GER), aspen (EN), peuplier tremble (F), pioppo tremolo (I)). They are widely distributed throughout the northern hemisphere, in boreal and subtropical zones and in mountainous and floodplain areas.

In addition to the great breeding potential of poplars, poplar wood has a wide range of applications. The options for products from poplar wood are numerous, especially in the production of paper, pulp and cardboard packaging, but also in the production of wooden boards and veneers, wooden packaging, matches, bioenergy, for heating purposes, etc. Activities in the field of conservation of poplar genetic resources in the world, especially white and black poplar, are carried out within the EUFORGEN network. These species are not listed in the international organization CITES Appendices of endangered species. According to the IUCN, *P. alba* and *P. tremula* are reported as species of the least concern, while no data are available for *P. nigra*. Only *P. caspica* stands out as an endangered species.

IMPORTANCE OF POPLAR BREEDING

Nowadays, there is an increased global interest in the cultivation of different poplar species and hybrids. Some of the reasons are: the organic properties and široku industrijsku upotrebu; sposobnost rasta na marginalnim tlima; sposobnost većine popularnih vrsta i križanaca da se vegetativno razmnožavaju (Sarsekova, 2015.). Dobivanje genotipova šumskog drveća čija su svojstva bolja od postojećih vrsta glavni je cilj primjene oplemenjivanja (Kajba i Ballian, 2007.). Oplemenjivanje se vrlo često provodi radi povećanja proizvodnosti drvne mase. Usto, među važnim ciljevima oplemenjivanja topola jest i povećanje kvalitete drva.

Mnoge vrste topole prilagodile su se različitim klimatskim uvjetima i tipovima staništa, uključujući visoke temperature kineskih pustinja, kao i hladne, vjetrovite uvjete južnoameričkih Anda. Velika genetska varijabilnost karakteristika je upravo topola, a križanje među vrstama i sekcijama rasprostranjeno je u prirodi, kao i unutar kultiviranih topola. Topole su uzgojno zanimljive jer ih, osim brzog rasta, obilježavaju i brojne mogućnosti međusobnog križanja. Plantažno se sade diljem svijeta, uključujući i južnu hemisferu. Procijenjeno je da se topole uzgajaju u 70 zemalja svijeta, kako u mješovitim sastojinama, tako i u plantažnim nasadima te kao pojedinačna stabla u krajobrazima. Glavnina topola u svijetu, tj. 91 %, raste u prirodnim šumama, 6 % ih uspijeva na plantažama, a 3 % u mješovitim poljoprivredno-šumskim sustavima. Ukupna površina prirodnih šuma topole iznosi više od 70 milijuna hektara, od čega 97 % otpada na Kanadu, Rusku Federaciju i SAD, gdje se najčešće iskorištavaju za proizvodnju drvne sirovine. U ostalim većim zemljama u kojima topole prirodno rastu (Kina, Njemačka, Finska, Francuska, Indija i Italija) primarno se koriste za unaprjeđenje okoliša, uključujući konzervaciju in situ, zaštitu voda i tla te za obnavljanje krajolika. Kad je riječ o posađenim sastojinama topole u svijetu (plantaže, zaštita šuma i poljoprivredno-šumski sustavi), njihova površina iznosi 6,7 milijuna hektara, od čega je 3,8 milijuna hektara zasađeno topolom ponajprije radi proizvodnje drvne sirovine, a ostatak od 2,9 milijuna hektara radi zaštite okoliša.

RELEVANTNE SPOZNAJE O DRVU TOPOLE

Drvo topole je rastresito porozno, s uočljivim ili slabo uočljivim godovima te je fine i jednolične teksture. U pojedinih vrsta topole postoje određene razlike u makroskopskim karakteristikama drva. Drvo trepetljike je bakuljavo, prljavo bijele do žućkastobijele boje; drvo crne topole je jedričavo, sa svjetlosmeđom do svijetlo zelenkastosmeđom srži, dok drvo bijele topole ima crvenkastožutu do žutosmeđu srž.

Pregled ranih istraživanja drva topole prema Bendtsenu (1978.), Zobelu i van Buijtenenu (1989.), Tsoumisu (1991.) te Zobelu i Spragueu (1998.) donosi ove spoznaje: stabla koja se uzgajaju u kratkim ophodeconomic value that poplar trees provide; rapid growth and the ability to produce wood suitable for technical use already after a rotation of 20 years; the source of raw material of satisfactory wood quality for wide industrial use; the ability to grow on marginal soils; the ability of most poplar species and hybrids to reproduce asexually (Sarsekova, 2015). Obtaining genotypes of forest trees with properties better than the existing ones is the main goal of the improvement (Kajba and Ballian, 2007). Very often the improvement is carried out in order to increase the productivity of wood mass. Also, one of the important goals of poplar improvement is to increase the quality of the wood.

Many poplar species have adapted to different climates and habitat types, including the high temperatures of the Chinese deserts and cold, windy conditions of the South American Andes. High genetic variability is a characteristic of poplars, while crossing between species and sections is widespread in nature and in poplar cultivation. Poplars are interesting for cultivation because, in addition to their rapid growth, they are also characterized by the wide range of cross-breeding possibilities. Plantations are distributed all over the world, including the southern hemisphere. It is estimated that poplars are grown in 70 world countries, both in mixed stands and plantations, and as individual trees in landscapes. The majority of poplars in the world grow in natural forests (91 %), 6 % in plantations and 3 % in mixed agroforestry systems. The total area of native poplar forests is over 70 million hectares, of which 97 % is in Canada, the Russian Federation and the United States of America, where they are most often used for the production of wood raw material. In other major countries where poplars occur naturally (China, Germany, Finland, France, India and Italy), they are primarily used for environmental improvement, including in situ conservation, water and soil protection and landscape restoration. As for planted poplar stands in the world (plantations, forest protection and agro-forestry systems), their area is 6.7 million hectares, of which 3.8 million hectares are planted primarily for the production of wood raw material, and the remaining 2.9 million hectares for the purpose of environmental protection.

RELEVANT KNOWLEDGE ABOUT POPLAR WOOD

Poplar wood is diffuse porous, with noticeable or barely noticeable annual growth rings and a fine and uniform texture. Regarding the macroscopic characteristics of the wood, there are certain differences among different poplar species. The heartwood of aspen wood is uncolored, dirty white to yellowish white in color; the heartwood of black poplar wood is colored, light njama poput topola proizvode drvo s velikim udjelom juvenilnog drva; gustoća drva u blizini srčike je velika, smanjuje se, a zatim se u zrelom drvu, nakon otprilike 15. goda, ponovo povećava; s povećanjem starosti stabala nema varijacija u omjeru volumena različitih elemenata strukture drva; u rastresito poroznom drvu kao što je topolovina kasno drvo nije jasno vidljivo i ne postoji praktičan način proučavanja odnosa između gustoće drva i širine godova.

Novija istraživanja drva topole govore o sljedećemu: a) juvenilno drvo obuhvaća prvih deset godova počevši od srčike (Senft, 1986.; Barcik i dr., 2008.; Efhami i Saraeyan, 2009.); b) zabilježene su varijacije između i unutar križanaca i prirodnih vrsta topola, i to u smislu anatomskih i mehaničkih svojstava drva (Telewski i dr., 1996.); c) gustoća drva topole ima širok raspon između taksonomskih sekcija i između klonova, rangirane su prema gustoći vrste topole male gustoće (Balatinecz i dr., 2014.); d) topole veće gustoće također pokazuju sličan potencijal poput, primjerice, breze ili bukve (Balatinecz i dr., 2014.); e) u nekih novih kultivara godovi mogu biti prilično široki, često širi od 2 cm, katkad čak široki tri ili više centimetara (Balatinecz i dr., 2014.); f) glavne greške povezane s rastom jesu kvrge i tenzijsko drvo (Koman i dr., 2013.); g) oborine su navedene kao čimbenik koji određuje širinu godova topola (Ziemiańska i Kalbarczyk, 2018.); h) brzina prirasta negativno se odražava na gustoću drva određenih klonova topola (Ištok i dr., 2016.).

Uzgojem topola dobiva se sirovina različite kvalitete, ovisno o krajnjoj namjeni drva. Napomena: podatci o tehničkim i tehnološkim svojstvima drva topole dostupni su na web stranicama i u priručnicima navedenim u literaturi na kraju teksta. brown to light greenish brown, while the heartwood of white poplar wood is reddish yellow to yellow-brown.

A review of early research on poplar wood according to Bendtsen (1978), Zobel and van Buijten (1989), Tsoumis (1991) and Zobel and Sprague (1998) provides the following knowledge: trees grown in short rotations, such as poplars, produce wood with a large proportion of juvenile wood; wood density is high near the pith, decreases, then increases again in the mature wood after about the 15th annual growth ring; there is no variation in the proportion of different wood structure elements by volume with increasing age of trees; in diffuse porous hardwoods, such as poplars, latewood is not clearly discernible and there is no practical way to study the relationship between wood density and ring width.

Later research on poplar wood leads to the following conclusions: (a) juvenile wood includes first ten annual growth rings from the pith (Senft, 1986; Barcik et al., 2008; Efhami and Saraeyan, 2009); (b) there are considerable variations between and within hybrid and natural poplar species for anatomical and mechanical properties (Telewski et al., 1996); (c) density of poplar wood has a wide range between taxonomic section and between clones, ranking the species with low density (Balatinecz et al. 2014); (d) the higher-density poplars also show similar potential to, for example, birch or beech (Balatinecz et al. 2014); (e) in some new cultivars, growth rings may be quite broad, often exceeding 2 cm and sometimes even 3 cm or even more (Balatinecz et al. 2014); (f) the main growthrelated defects include knots and tension wood (Koman et al. 2013); (g) rainfall is indicated as a factor determining the tree-ring width in poplars (Ziemiańska and Kalbarczyk, 2018); (h) increased growth rate has a negative effect on wood density of certain poplar clones (Ištok et al. 2016).

The cultivation of poplar produces raw material of different quality, depending on the end use of wood. Note: data on technical and technological properties of poplar wood are available on web pages and in manuals listed in literature section.

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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.

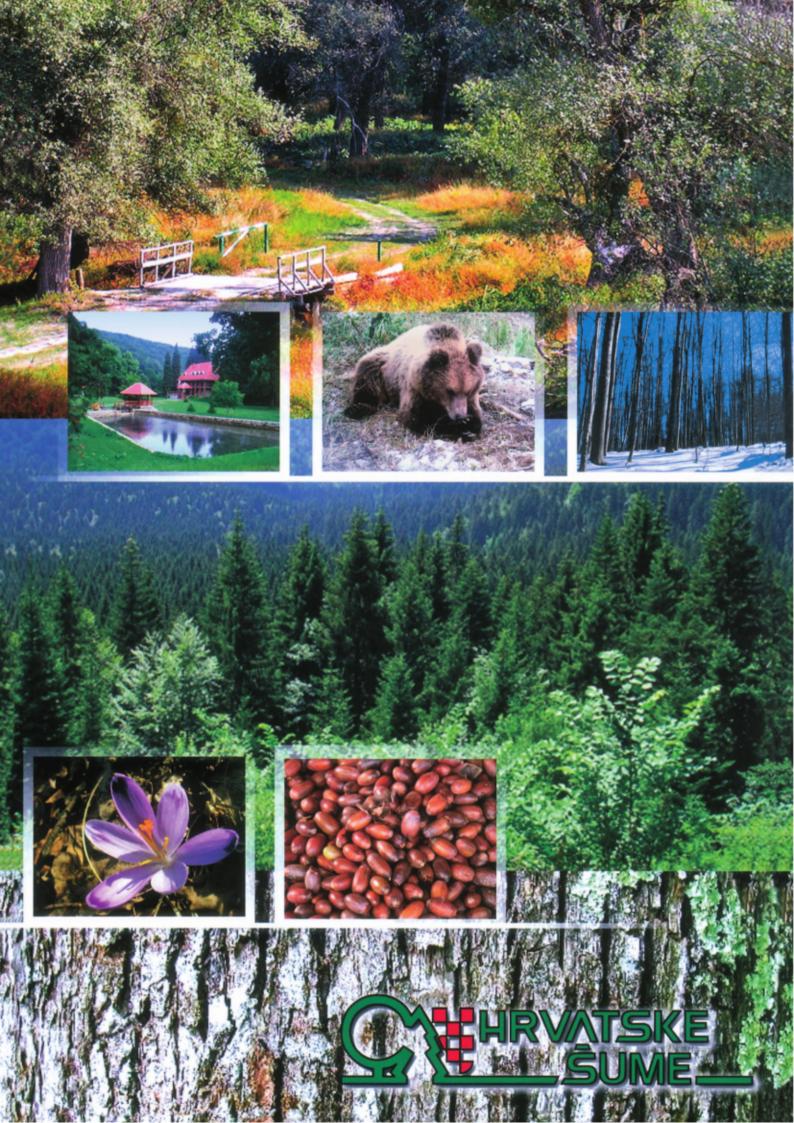
Na temelju provedenih upitnika i intervjua, zaključeno je kako su osobni razvoj i cjeloživotno učenje put za uspješnu karijeru svake žene u svim sektorima, pa tako i šumarskom, a na tom putu potrebno je:

- 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).





povezite se s prirodom

D drzodjelac

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