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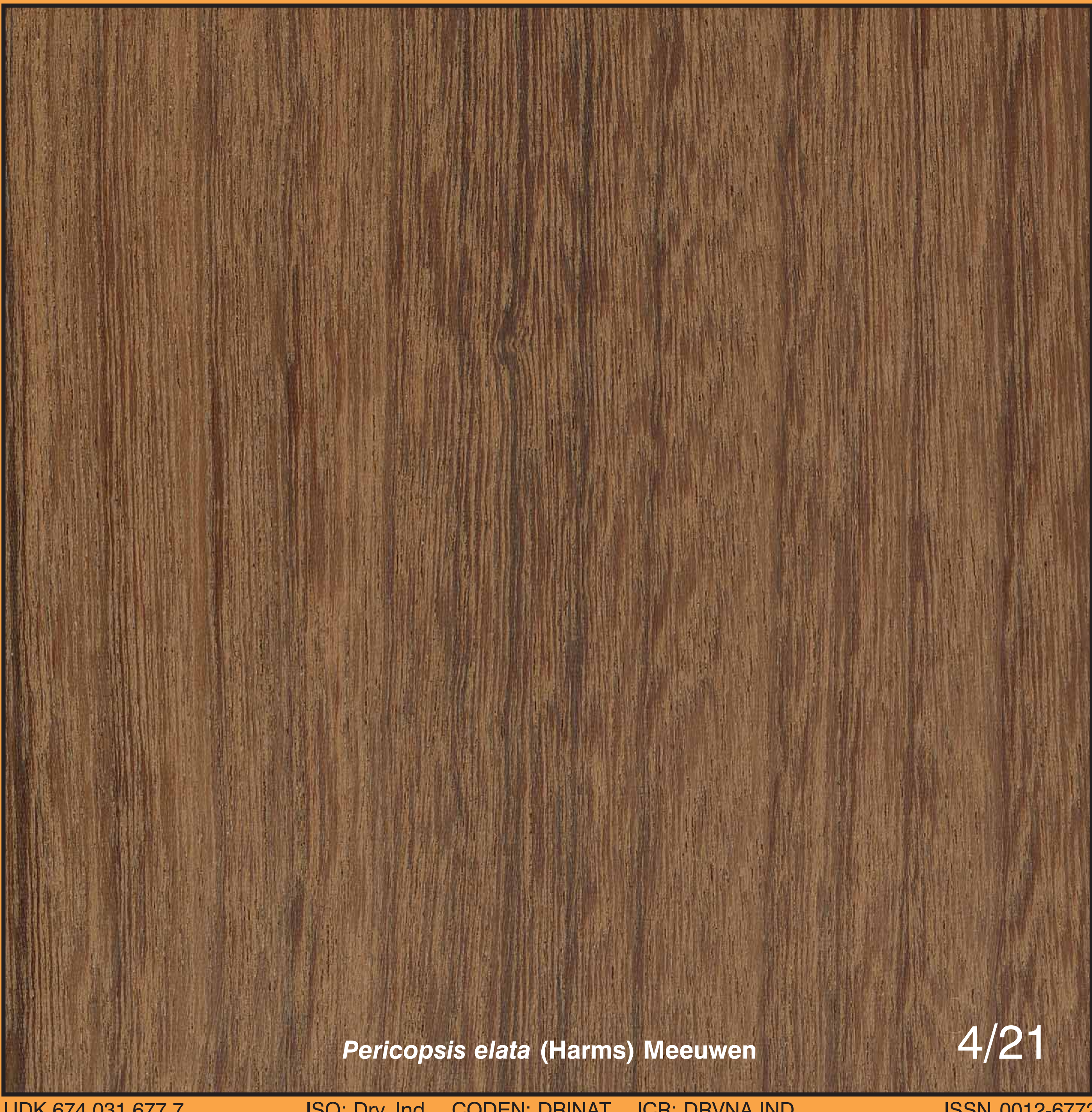

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The Influence of Pulping Process and Energy Consumption on Properties of Nanofibrillated Lignocellulose (NFLC) Films Isolated from Wheat Straw

Utjecaj procesa proizvodnje pulpe i potrošnje energije na svojstva filmova na bazi nanofibrilirane lignoceluloze (NFLC) izolirane iz pšenične slame

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ABSTRACT • *The present research has primarily focused on the production of nanofibrillated lignocellulose (NFLC) instead of nanofibrillated cellulose (NFC), which could be produced with less energy and is expected to have similar uses as NFC, especially in the sectors where the transparency is not important. Furthermore, the effect of energy consumption needed for NFLC production and also the influence of pulping methods on the produced NFLC properties has been surveyed. Through mechanical refining and different passes in microfluidizer, the results showed the average diameter of NFLC declined from around 19000 nm to 36 nm. Soda-NFLC films had higher calliper and lower roughness, compared to those of MEA at given energy consumption in refiner and microfluidizer. For both kinds of pulps, the optimum level of energy consumption to reach the best tensile index of NFLC films was 258 kWh/t, with three passes through microfluidizer. More increase in the number of passes and pressure only resulted in increasing of energy consumption without any positive effect on improving the tensile index. The maximum tensile indices of NFLC films obtained from soda and MEA pulping processes were 113.5 and 119.86 N·m/g, respectively. The burst index of 8.5 kP·m^2/g and the energy consumption of 458 kWh/t were obtained for five passes through microfluidizer. With the increase of the number of passes of soda and MEA samples through microfluidizer, the opacity decreased but transparency increased.*

Keywords: *nanofibrillated lignocellulose; wheat straw; soda and MEA pulping; energy consumption*

SAŽETAK • *Ovo je istraživanje usmjereno na proizvodnju nanofibrilirane lignoceluloze (NFLC) umjesto nanofibrilirane celuloze (NFC). Ta bi se celuloza (NFLC) mogla proizvesti s manje energije i moglo bi se očekivati*

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da će imati sličnu uporabu kao NFC, osobito u područjima gdje transparentnost nije osobito važna. Ispitan je i učinak potrošnje energije potrebne za proizvodnju NFLC-a, kao i utjecaj metode proizvodnje pulpe na svojstva proizvedene lignoceluloze. Rezultati istraživanja pokazali su da je mehaničkim oplemenjivanjem i uz različit broj prolazaka kroz mikrofluidizator prosječni promjer NFLC-a pao s oko 19 000 nm na 36 nm. NFLC filmovi od natronske pulpe pri određenoj su potrošnji energije u rafinatoru i mikrofluidizatoru imali veću debljinu i manju hrapavost u usporedbi s onima od MEA pulpe. Optimalna razina potrošnje energije za postizanje najboljega vlačnog indeksa NFLC filmova za obje vrste pulpe bila je 258 kWh/t, uz tri prolaska kroz mikrofluidizator. Povećanje broja prolazaka i tlaka rezultiralo je samo povećanjem potrošnje energije bez ikakva pozitivnog učinka na poboljšanje indeksa kidanja. Maksimalni indeksi kidanja NFLC filmova od pulpe dobivene natronskim i MEA postupkom bili su 113,5 odnosno 119,86 N·m/g. Indeks prskanja od 8,5 kP·m²/g i potrošnja energije od 458 kWh/t dobiveni su prolaskom pulpe kroz mikrofluidizator pet puta. S porastom broja prolazaka uzoraka natronske i MEA pulpe kroz mikrofluidizator smanjila se neprozirnost, ali se povećala transparentnost uzoraka.

Ključne riječi: *nanofibrilirana lignoceluloza; pšenična slama; priprema pulpe natronskim i MEA postupkom; potrošnja energije*

1 INTRODUCTION
1. UVOD

Nanotechnology can create an entirely new industry, while reducing the demand for some goods and increasing the demand for some other goods (Wegner *et al.,* 2009). With help of nanotechnology, lignocellulosic sector is capable to produce new high-tech products and create markets with a wide variety of applications. One of these new high-tech products is nanofibrillated cellulose (NFC) (Ankerfor, 2012). NFC is composed of nano sized cellulose fibrils with a high aspect ratio (length to width ratio) of more than 100. The development of NFC was pioneered by Turbak *et al.* in 1983.They demonstrated that, by treating wood-based cellulose fiber suspensions with a high-pressure homogenizer, a gel-like material can be produced (Turbak *et al.,*1983; Zimmerman, 2010; Spence *et al.,* 2011_a). The resulting material was denoted as Microfibrillated Cellulose (MFC) or Nanofibrillated Cellulose (NFC), and it showed promising properties and high potential to produce diverse industrial and commercial goods such as cosmetics, health, food, packaging, etc (Abdul Khalil et al., 201; Henrik *et al.,* 2008; Yousefi *et al.,* 2011; Spence, 2011).

Today, NFC is produced from various cellulosic sources among which wood is the most important source of cellulose and is the main raw material for NFC production (Iwamoto *et al.,* 2005; Spence, 2010; Habibi *et al.,* 2010; Afra *et al.,* 2013). Due to the increasing demand for wood in other industrial sectors and also the need to protect forests as one of most important factors in the fight against global warming and climate changes, appropriate alternatives to wood should be sought. Lignocellulosic agricultural residues are a major source as they are renewable, environmentally beneficial and also they need less energy consumption in the production of NFC and thus they could be a good substitute for wood as a raw material (Alemdra, 2008).

To liberate the cellulose from lignocellulosic matrix and produce NFCs, the lignocellulosic raw material must be fractionated. For this purpose, there are some delignification processes in industrial scale such as kraft, sulphite and soda pulping processes. Soda pulping is the dominant process for lignocellulosic agricultural residues. Strong alkaline cooking liquors in this process dissolve carbohydrates to a great extent. In addition, the capability of sodium hydroxides in dissolving lignin is restricted to some specific lignin structures. These conditions result in a pulp with low yield, low hemicelluloses content and high condensed lignin. In return, MEA pulping can dissolve lignin in a good selective way. It means that in comparison with soda pulp, MEA pulp has significantly higher yield, higher hemicelluloses and lower lignin contents (Hedjazi *et al.,* 2009; Salehi *et al.,* 2014).

To produce pure NFC, it was necessary to remove hemicelluloses and lignin by pre-extraction treatments and diverse bleaching sequences, because in principle, NFCs are produced with bleached cellulose fibers (dissolving pulp, white pulp). Bleaching process consumes energy and chemicals and imposes a lot of environmental pollution (Spence *et al.,* 2011). Recently, unbleached lignocellulose fibers (brown pulp) have been converted to nanostructures (Spence, 2011). The resulting materials can be referred to as nanofibrillated lignocellulose (NFLCs). From an environmental and economic point of view, because of less chemicals and energy needed to produce unbleached pulps, NFLCs are superior to NFCs (Yousefi *et al.,* 2018; Djafari Petroudy, 2014).

For NFC production, normally the pulp fibers had to be run several times through an apparatus like homogenizer and microfluidizer, which resulted in high energy consumption. Nowadays, the development of disintegration methods, which are less energy consuming, became a priority in NFC production. Thus, various mechanical pre-treatments have been suggested in order to obtain NFCs at lower energy input. Mechanical pre-treatments are arried out on the pulp to reduce fiber size and/or to pre-defibrillate the fibers, thus reducing the frequency of equipment clogging and the energy consumption during conversion of pulps to NFC (Spence *et al.,* 2011). Alternatives for mechanical reduction of fiber size include disk refiners, PFI mills and valley beaters. Gradual peeling off of the external cell wall layers (P and S1 layers) is carried out by a refining treatment prior to homogenization or microfluidization. Thereby, the S2 layer is better exposed to mechanical actions and causes internal fibrillation during homogenization and micro-fluidization (Andresen *et al.,* 2006; Henriksson *et al.,* 2008; Iwamoto *et al.,* 2005; Nakagaito and Yano, 2004; Spence *et al.,* 2011; Turbak *et al.,* 1983).

Unfortunately, the most important challenging issue of NFC production at industrial scale is its high energy consumption (Yousefi, 2011; Ankerfors, 2012; Spence, 2011b; Zimmermann, 2010; Eriksen, 2008). In general, energy consumption in NFC production differs significantly due to parameters such as source of lignocellulose, fractionation method of lignocellulose, number of passes and flow rate and cellulose fibers.

Nowadays many researchers are trying to find new ways to reduce production costs of NFC to obtain nanomaterials and nanocomposites at a lower price, as well as to increase industrial production capability (Ankerfors, 2012; Spence *et al.,* 2010; Spence *et al.,* 2011; Zimmermann, 2010; Paakko *et al.,* 2008; Syverud *et al.,* 2011; Iwamoto, 2009). Therefore, this research has focused on the study of the influence of simultaneous use of wheat straw as an inexpensive agriculture lignocellulosic residue, different pulping processes without bleaching as downsizing methods, and industrial simulated refiner treatment as mechanical pre-treatment on energy consumption and properties of produced NFLCs.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

Wheat straws were collected from cultivation areas in the north of Germany. Cleaned straw was chopped into small pieces by a cutter in length of 3-5 cm. The prepared samples were conditioned in a laboratory and stored in plastic bags. Before experiments, the moisture content of the material was determined by Tappi T 264-0m 88 standard. Analytical grade monoethanolamine (MEA) and NaOH, purchased from Baden Aniline Soda Factory (BASF, Germany) were used.

2.2 Methods
2.2. Metode

2.2.1 Chemical compositions
2.2.1. Kemijski sastav

The chemical composition of wheat straw was determined according to relevant Tappi test methods as follows: sample preparation (T267-om 85), ash content (T211-om 93), extractives soluble in alcohol-acetone (T207-om 97), cellulose content (T264-om 88), lignin content (T222-om 97) and holocellulose method; Useful method 249- um 75.

2.2.2 Pulp production
2.2.2. Proizvodnja pulpe

Soda and MEA processes were used for pulping wheat straw. In soda process, alkalinity of 16 %, cooking time of 30 min, temperature of 160 °C and liquor to straw (L/S) ratio of 3 to 1 were selected. In MEA pulping, cooking time of 45 minutes, cooking temperature of 145 °C and (L/S) ratio of 4 to 1 was employed. Pulping was performed in a15 l rotating digester equipped with indirect heating. At the end of cooking, the digester was cooled down and the pulp discharged into a sieve, thoroughly washed with tap water, disintegrated in a laboratory pulper for 5 minute and then screened on a vibrating 0.15 mm slot screen. The screened pulp was dewatered and stored in polyethylene bags at 4 °C until further processing. The pulp was drained and after measuring the moisture content of the pulp, the total yield, screened yield and screen reject were determined. After complete hydrolysis of the pulps, carbohydrates and simple sugars were characterized by 72 % sulfuric acid using HPLC. The following pulp properties were also determined: kappa number, (Tappi T 236-om 06), viscosity (Zellcheming-Vorschift IV 61/36) and brightness, (T 425-om 98).

2.2.3 Mechanical pre-treatment
2.2.3. Mehanički predtretman

For refining of pulp, the pilot refiner of Voith Company (LR 40) was employed. For refining purposes, 1500 gr pulp based on dry weight was added to the refiner at energy consumption of 170 kWh/t. This level of energy consumption in the refiner comes from preliminary test (Ahmadi, 2014).

2.2.4 Manufacture of NFLC
2.2.4. Proizvodnja NFLC-a

The refined soda and MEA unbleached pulp suspensions were mechanically disintegrated at 0.24 % solids content using a high-pressure fluidizer (Microfluidizer M-110EH-30; Microfluidics Corp., USA). In the standard procedure, the samples were passed two times through the large chambers and four times through the small chambers. Due to the narrowness of the chambers, operating pressures of approximately 69 MPa (10 kpsi), and 138 MPa (20 kpsi) are normally needed to push the material through the large and small chambers. After passing the chambers, the samples were cooled in a heat exchanger before they were collected in a jar (Ankerfors, 2012). Table 1 presents the energy consumption of microfluidizer in each pass. The microfluidizer energy measurements were 79 and 100 kWh/t per pass for processing pressures of 69 MPa (10 kpsi) and 138 MPa (20 kpsi), respectively.

Table 2 shows sample identification codes. Control sample refers to mechanically un-treated soda and MEA pulps.

2.2.5 Preparation of nanofilms
2.2.5. Priprema nanofilmova

The microfluidizer treated NFLC suspensions at 0.24 % consistency were first degassed for 10 min with a water vacuum pump. The degassed suspensions were then used to form nanofilms using a semiautomatic sheet former (Rapid-Köthen) under vacuum. The apparatus and procedure for film formation was adapted as described in a previous study (Glasenapp, 2014).

Table 1 Energy consumption of microfluidizer in each pass
Tablica 1. Potrošnja energije mikrofluidizatora pri svakom prolasku

Pass number *Broj prolaska*	**Microfluidizer chamber** *Komora mikrofluidizatora*	**Pressure, MPa** *Tlak,* MPa	**Energy consumption, kWh/ton** *Potrošnja energije,* kWh/ton
1	small	69	79
2	small	69	79
3	large	138	100
4	large	138	100
5	large	138	100
6	large	138	100

Table 2 Sample identification codes
Tablica 2. Oznake uzoraka

Code / *Oznaka*	**Sample** / *Uzorak*
Ctrl	Control / *kontrolni*
Pre	Mechanical pre-treatment by refiner *mehanički predtretman rafiniranjem*
1 up to 6	Microfluidizer passes 1 up to 6 *prolazak kroz mikrofluidizator od 1 do 6 puta*

The films with the base weight of 30 g/m^2 were conditioned in a standardized environment at 22 °C and 50 % relative humidity for 24 hours.

2.2.6 Nanofilm testing
2.2.6. Ispitivanje nanofilmova

The characterization of NFLC films were carried out according to the following standards: density (ISO 534), thickness (Tappi 411 om 97), roughness (Tappi 538 om 01), tensile strength (ISO 1924-2), burst strength (TAPPI 403om 02), opacity and transparency (ISO 2471). The selected NFLC films were further analyzed by field emission scanning electron microscopy (FE-SEM; FEI Company). The fiber diameter of NFLC films from soda and MEA pulps was measured by image-J software and the average dimension of 100 fibers was reported.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Chemical composition of wheat straw
3.1. Kemijski sastav pšenične slame

The typical distribution of cellulose, hemicellulose, lignin, and extractives for softwood is 42 %, 27 %, 28 %, and 3 %, respectively, while hardwood distribution is 45 %, 30 %, 20 %, and 5 %, respectively (Smook, 2002). Table 3 presents the results of the chemical composition of wheat straw. Compared to softwoods and hardwoods, wheat straw has higher content of cellulose and lower content of lignin, and these properties can be positive in regard to energy and chemical consumption as well as production yield of pulping processes. However, higher ash content can cause some difficulties in recovery of black liquors in wheat straw based pulp and paper mills.

Table 3 Chemical composition of wheat straw
Tablica 3. Kemijski sastav pšenične slame

Component / *Komponenta*	**Value** / *Vrijednost,* %
Cellulose / *celuloza*	49.78
Lignin / *lignin*	19.64
Hemicelluloses / *hemiceluloza*	20.37
Extractives soluble in alcohol - acetone / *ekstraktivi topljivi u alkohol-acetonu*	4.93
Ash content *sadržaj pepela*	5.28

3.2 Pulp properties
3.2. Svojstva pulpe

Table 4 shows the properties of soda and MEA pulps. As it can be seen, MEA pulp has higher overall yield (60 %) than soda pulp (48 %). One of the remarkable properties of MEA is the conservation of hemicelluloses of the lignocellulosic materials in the pulping process that results in higher pulp yield (Hedjazi *et al.,* 2009; Hedjazi *et al.,* 2011). MEA process resulted also in pulps with lower kappa number than that of soda process. In this study, we have tried to keep the kappa numbers of soda and MEA pulps the same as much as possible to have better comparison of refining effect on

Table 4 Soda and MEA pulp properties
Tablica 4. Svojstva natronske i MEA pulpe

Pulping *Priprema pulpe*	**Properties** / *Svojstva*									
	Screen pulp yield, % *Prinos prosijavanja, %*	**Screen reject, %** *Otpad prosijavanja, %*	**Total pulp yield, %** *Ukupan prinos vlakana, %*	**Kappa no.** *Kappa-broj*	**ISO Brightness, %** *ISO svjetlina, %*	**Viscosity ml/g** *Viskoznost, ml/g*	**Beating degree, SR** *Stupanj mljevenja,* SR	**Extractive, %** *Ekstraktivi, %*	SiO_2, %	**Ash, %** *Pepeo, %*
Soda	43	5	48	18	24.1	805	21	0.5	0.54	1.82
MEA	57.2	2.8	60	16.5	36.3	915	30	1.45	1	2.95

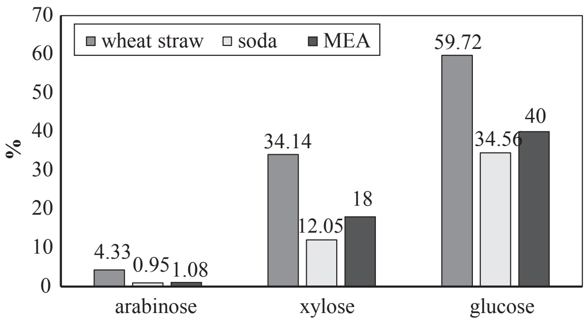

Figure 1 Monosaccharaides composition of wheat straw, soda and MEA pulps
Slika 1. Sastav monosaharida od pšenične slame te od natronske i MEA pulpe

the pulps. The higher beating degree of MEA pulp is attributed to its higher hemicelluloses content, which in turn facilitates the refining process and thus reduces the energy consumption in refiner to achieve certain freeness or beating degree.

As figure 1 shows, MEA pulp had higher xylose and glucose contents than soda pulp. This means that the carbohydrates are protected better in MEA pulping. The mechanism of conservation of carbohydrates by MEA was not completely known and there are contradictory theories regarding this matter. Some researchers believe MEA acts as a reducing agent and with converting the reducing ends of cellulose chains to alditols stops the peeling reaction. Some others have reported that monoethanolamine stabilizes polysaccharides by condensation reaction of aldehyde and formation of imines. Based on this theory, the stability of carbohydrates is due to radical reduction in presence of MEA ((Hedjazi *et al.,* 2009; Hedjazi *et al.,* 2011; Salehi *et al.,* 2014; Ghahremani *et al.,* 2014).

3.3 Physical properties of NFLC films
3.3. Fizička svojstva NFLC filmova

Table 5 presents the physical properties of soda and MEA pulps and nanofilms including film thickness, density and roughness. An increase in energy consumption, caused by production of smaller nanofibers with higher compressibility, resulted in sheet texture with less thickness and roughness as well as higher density. Soda pulps and nanofilms had higher calliper and lower roughness compared to those prepared with MEA.

3.4 Mechanical properties of NFLC films
3.4. Mehanička svojstva NFLC filmova

Figure 2 shows the results of tensile indices of sheets produced with soda and MEA pulps (Ctrl) and those of mechanically pre-treated ones (Pre) together with the tensile index of nanofilms prepared from NFLC passed through microfluidizer up to six times (1 up to 6). As shown in Fig. 4, sheets prepared with control samples had the lowest tensile index of 34 and 37 N·m/g for soda and MEA pulps, respectively (47 % and 43 %). When a mechanical pre-treatment was applied, the tensile index significantly increased. In the case of NFLC nanopapers, the increase in number of passes resulted in increased energy consumption and significant improvement of tensile index. The maximum tensile index was observed after the third pass through microfluidizer for NFLC film prepared with soda and MEA (113.5 and 119.86 Nm/g, respectively). Energy consumption by microfluidizer for the current passage was 258 kwh/t. The present results showed the high efficiency of MEA in terms of saving energy to reach the highest tensile index. The tensile strength of nanofilms showed the level or power of hydrogen bonding formed between nanofibers in paper network (Ahmadi *et al*., 2018). The tensile strength of nanofilms showed the level or power of hydrogen bonding formed between nanofibers in paper network. The tensile strength of nanofilms increased with the increase of bonding number or strength. This strength is also dependent on bonding number on one side and individual nanofibers strength on the other. In general, increasing the fibrillation induced by addition of energy consumption in microfluidizer (increased number of passes) as well as applying pre-treatment resulted in an increased tensile strength. Increase in the number of passes (over 3 passes) only caused the increase of energy consumption without effect on improving the tensile index so fiber damage caused the decrease of the tensile index. Other researchers have reported the same trend in production of NFCs by microfluidizer (Spence *et al.,* 2011; Ahmadi *et al*., 2018).

Table 5 Physical properties of pulp sheets and NFLC films from soda & MEA pulping
Tablica 5. Fizička svojstva listova pulpe i NFLC filmova dobivenih od natronske i MEA pulpe

Pulping process *Proces dobivanja pulpe*	**Properties** *Svojstva*	**Ctrl**	**Pre**	**Microfluidizer pass number** *Broj prolaska kroz mikrofluidizator*					
				1	**2**	**3**	**4**	**5**	**6**
Soda	Thickness / *debljina,* μm	53[e]	49.4[d]	44[c]	41.4[b]	41[b]	37.2[b]	36[b]	29.8[a]
	Density / *gustoća,* g/cm^3	0.49[f]	0.51[e]	0.65[d]	0.66[d]	0.78[c]	0.76[b]	0.79[a]	0.81[a]
	Roughness / *hrapavost,* ml/min	3410[f]	3228[e]	2636[d]	2598[c]	2476[c]	2263[b]	2182[b]	1974[a]
MEA	Thickness / *debljina,* μm	51[f]	46.2[e]	37.2[d]	36.2[d]	33.8[c]	32[b]	28.8[a]	28.7[a]
	Density / *gustoća,* g/cm^3	0.51[g]	0.53[f]	0.63[e]	0.66[d]	0.73[c]	0.77[b]	0.78[b]	0.84[a]
	Roughness / *hrapavost,* ml/min	3230[e]	3120[e]	3037[de]	2863[d]	2664[c]	2284[b]	2270[b]	2027[a]

*Lower case letters on the bars (a-f) show Duncan multiple range grouping of means. / *Mala slova (a – f) označuju grupiranje srednjih vrijednosti prema Duncanovu višestrukom rasponu.*

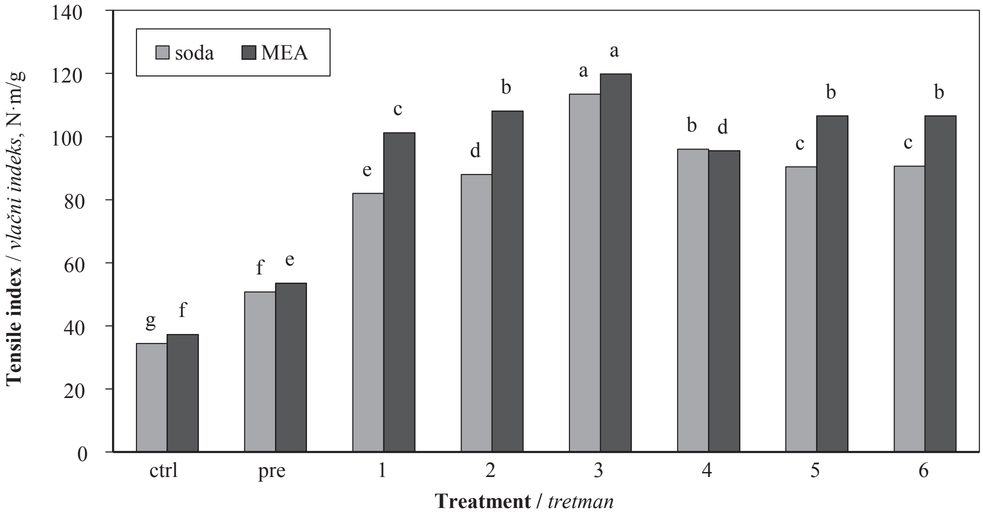

Figure 2 Tensile index of pulp sheets and NFLC films from soda and MEA pulping
(Lower case letters on the bars show Duncan multiple range grouping of means)
Slika 2. Vlačni indeks ploča pulpe i NFLC filmova dobivenih od natronske i MEA pulpe
(mala slova označuju grupiranje srednjih vrijednosti prema Duncanovu višestrukom rasponu)

Figure 3 shows the burst index of sheets made from Ctrl and Pre soda and MEA pulps together with the burst indices of nanofilms prepared with NFLC passed through microfluidizer up to six times. Burst index of NFLC films from soda and MEA pulping increased with the addition of energy consumption in microfluidizer, and the maximum value of 8.5 kP·m^2/g and energy consumption of 458 kwh/t were obtained after five passes through microfluidizer. Also, burst indices increased by 500 % and 466 %, for soda and MEA prepared nanofilms, respectively, at given total energy consumption of 628 kwh/t (refiner and 5 passes through microfluidizer). Burst index of sheets significantly increased with the increase of energy consumption due to the increase in fiber flexibility and the formation of more hydrogen bonds between fibers.

3.5 Optical properties of NFLC films
3.5. Optička svojstva NFLC filmova

Figures 4 and 5 depict opacity and transparency of pulp sheet produced with soda and MEA pulps (control samples) and those of mechanically pre-treated ones together with the opacity and transparency of nanofilms prepared with NFLC passed through microfluidizer up to six times. Soda and MEA control samples had higher opacity and lower transparency values. With the increase of the number of passes through microfluidizer, the opacity values of NFLC films decreased, but their transparency values increased. Generally, nanofilm opacity decreases with increasing the number of linkages between nanofibers along with denser NFLC texture. Thus, the opacity of nanofilms increased with increasing the fibrillation by chemical

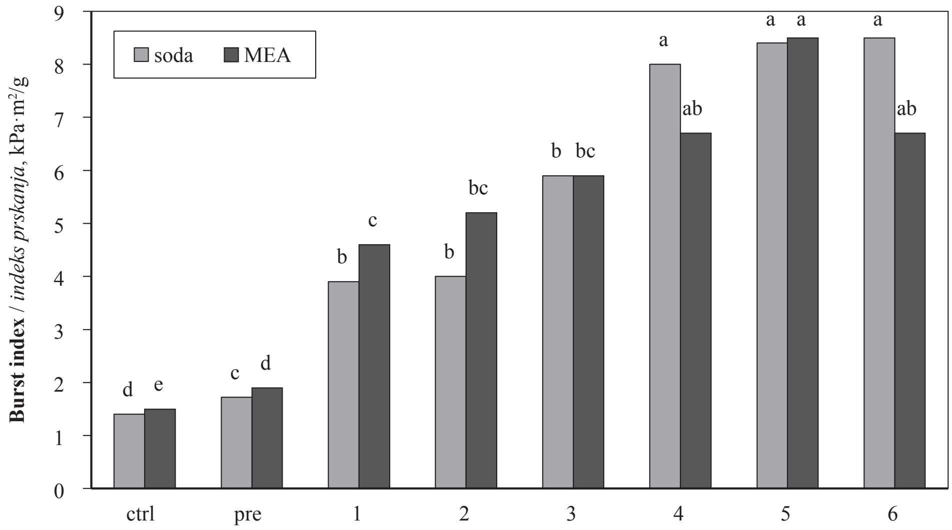

Figure 3 Burst index of pulp sheets and NFLC films from soda and MEA pulping
(Lower case letters on the bars show Duncan multiple range grouping of means)
Slika 3. Indeks prskanja ploča pulpe i NFLC filmova dobivenih od natronske i MEA pulpe
(mala slova označuju grupiranje srednjih vrijednosti prema Duncanovu višestrukom rasponu)

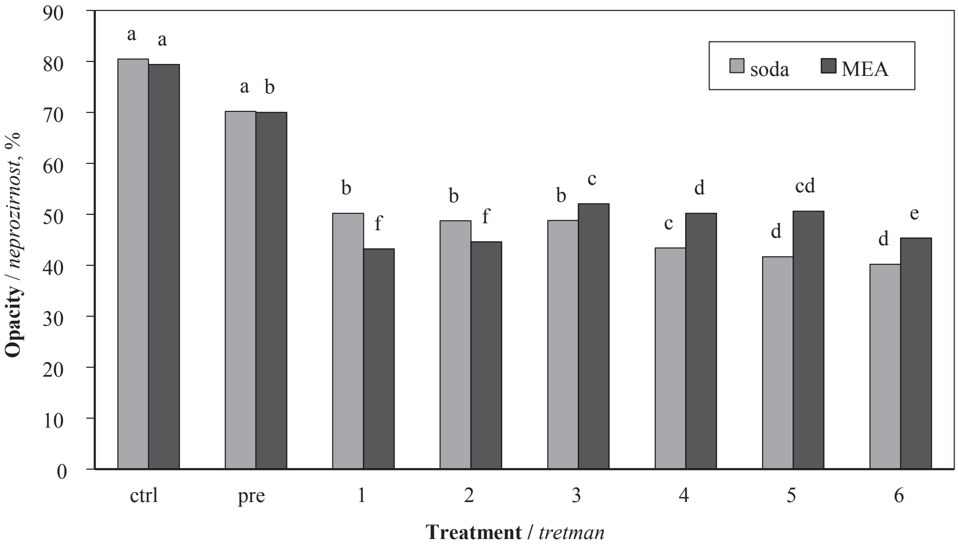

Figure 4 Opacity of pulp sheets and NFLC films from soda and MEA pulping
(Lower case letters on the bars show Duncan multiple range grouping of means)
Slika 4. Neprozirnost ploča pulpe i NFLC filmova dobivenih od natronske i MEA pulpe
(mala slova označuju grupiranje srednjih vrijednosti prema Duncanovu višestrukom rasponu)

or mechanical pre-treatments. In this case, the highest opacity was recorded in the control sample (around 80 %). The opacity of sheets decreased after refining pre-treatment due to fiber shortening, which decreases light scattering. The loss in opacity is continued because of the presence of fibers in nano scales. (Hassan *et al.,* 2011; Ankerfors, 2012; Ahmadi *et al*., 2018).

Among the nanofilms made from soda and MEA NFLC under different energy consumption, the highest transparency was obtained as 76.11 and 72.48 %, respectively. Total energy consumption was 728 kWh/t for soda NFLC and 628 kWh/t for MEA NFLC. In general, the transparency of samples increased after mechanical pre-treatment compared to the control one. Also, there was an increase in the transparency of samples after fibrillation by microfluidizer. Pre-treatment and nanofibrillation caused to change the fiber dimensions from micro scale to nano scale. As the dimension of nanoparticles is lower versus UV-visible wavelength (200-1000 nm), there is no obstacle for the increase of light transmittance and consequently transparency values of samples. Transparency can also be increased when surface roughness decreases (Nishino *et al.,* 2005; Yousefi *et al.,* 2011).

3.6 FE-SEM analysis
3.6. FE-SEM analiza

Figure 6 shows the FE-SEM micrographs of soda pulp sheet (a), MEA pulp sheet (b), soda-NFLC films (c) and MEA-NFLC films (d). The surface of papers from pre-treated fibers was non-uniform with somewhat intact fibers and a large density of inter-fiber

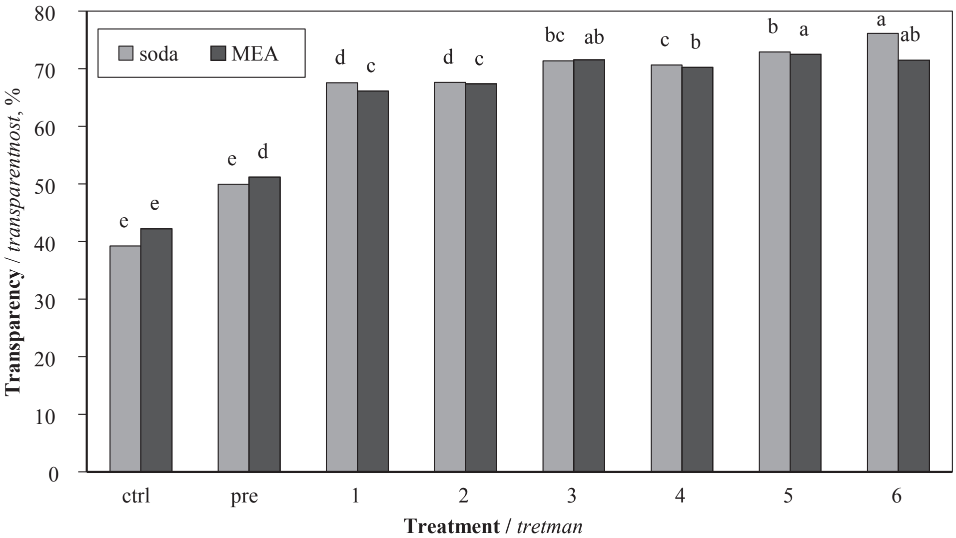

Figure 5 Transparency of pulp sheets and NFLC films from soda and MEA pulping
(Lower case letters on the bars show Duncan multiple range grouping of means)
Slika 5. Transparentnost ploča pulpe i NFLC filmova dobivenih od natronske i MEA pulpe
(mala slova označuju grupiranje srednjih vrijednosti prema Duncanovu višestrukom rasponu)

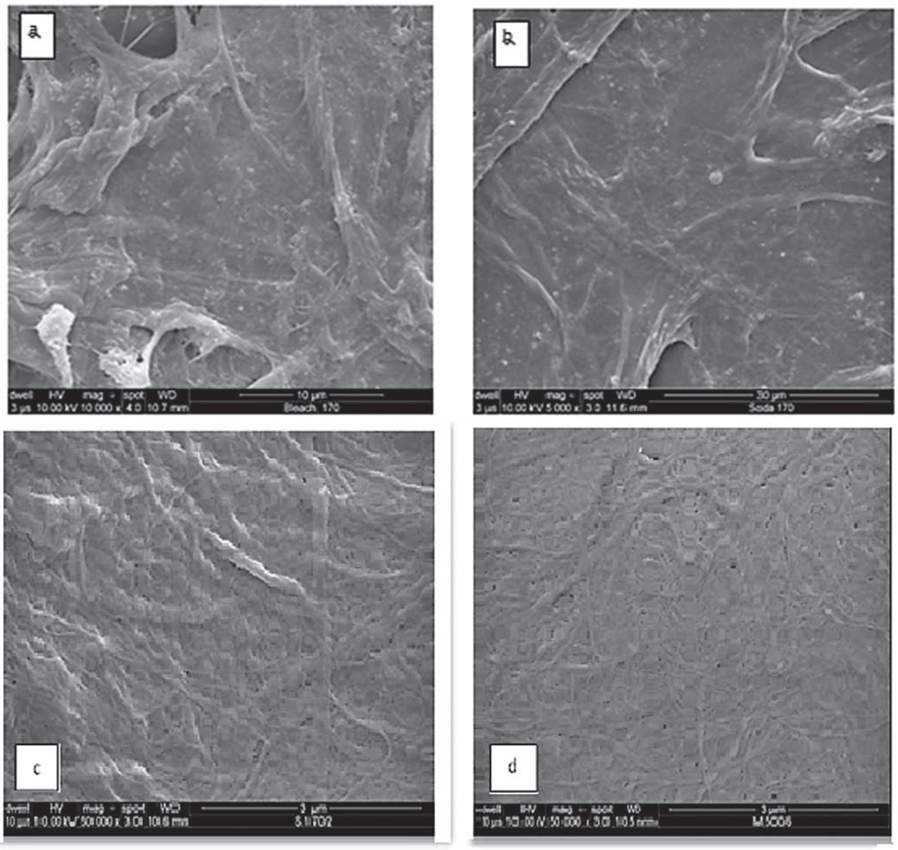

Figure 6 FE-SEM micrographs of a) soda pulp sheet, b) MEA pulp sheet, c) soda-NFLC films and d) MEA-NFLC films
Slika 6. FE-SEM mikrografije: a) listovi natronske pulpe, b) listovi MEA pulpe, c) soda-NFLC filmovi, d) MEA-NFLC filmovi

pores. However, after the sample was passed through microfluidizer, the NFLC films displayed a more uniform surface structure, smaller fibrils and fibril bundles.

Figure 7 depicts the average diameter for 100 fibers of each sample. The average diameter of soda and MEA control samples obtained was 18500 and 19100 nm, respectively. By refining pre-treatment, the corresponding values reached 12000 and 14000 nm, respectively. After downsizing with 2 passes (158 kWh/t) thorough microfluidizer, the corresponding values dramatically decreased to 52 and 45 nm, respectively. Also, the corresponding diameter values decreased to 38 and 36 nm, respectively, after 6 passes (558 kWh/t).

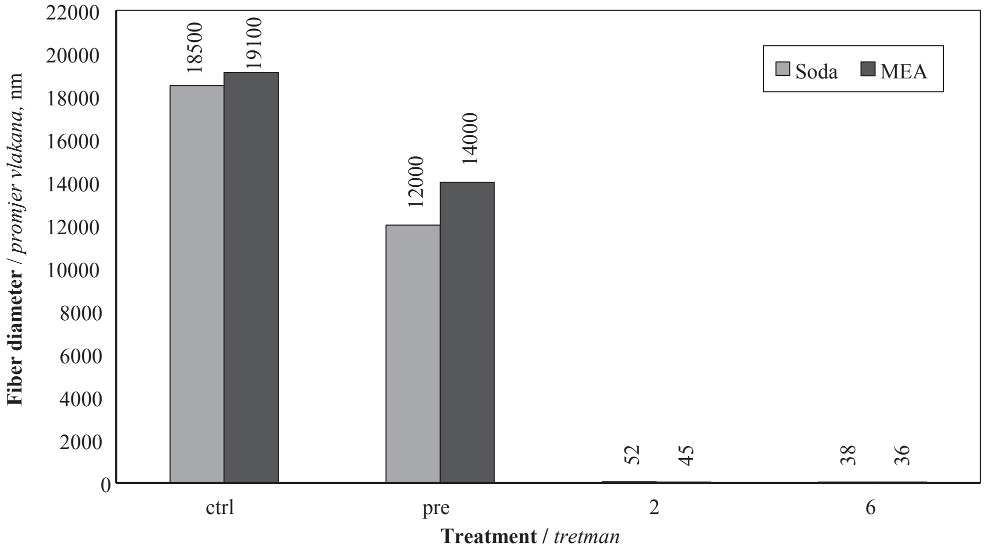

Figure 7 Fibers diameter of NFLC films from soda and MEA pulps
Slika 7. Promjer vlakana NFLC filmova dobivenih od natronske i MEA pulpe

Based on these results, it can be concluded that the average diameters of fibers decreased with the increase of energy consumption. Also, the average diameter of soda-NFLC and MEA-NFLC decreased 486 and 530 fold compared to the average diameters of corresponding control samples.

4 CONCLUSIONS
4. ZAKLJUČAK

In this study, two different processes using MEA and soda were used to produce unbleached pulp from wheat straw. The unbleached pulps were then downsized using microfluidizer with different passes and energy consumptions. The results showed that the production of NFLC from pulp obtained by MEA method leads to better properties with less energy consumption compared to the soda pulp production method. MEA pulping led to higher yield than soda pulping at given delignification rate. Also, MEA pulping had more refine-ability versus soda pulping, so that the cellulosic nanofibers with higher strength properties might be produced by microfluidizer of wheat straw with lower energy consumption versus soda pulping process. The results showed that using energy led to improve the strength properties of lignocellulosic nanofibers to some extent, while overuse of energy had no positive effect on improving these properties and only caused to increase the production costs. It was concluded that the MEA pulping process was more efficient than the soda pulping process in similar energy consumption.

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Regional Clusters, Similarities, and Changes in Turkey's Wood Production: A Comparative Analysis Using K-Means and Ward's Clustering Methods

Regionalni klasteri, sličnosti i promjene u preradi drva u Turskoj: usporedna analiza uz pomoć algoritma K-prosjeka i Wardove metode klasteriranja

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ABSTRACT • *This study aimed to separate the wood production in regions and provinces of Turkey into homogeneous groups based on similarities by using the country's wood production figures for 2013 and 2018. Within this context, the hierarchical Ward's and non-hierarchical K-means clustering methods were used comparatively. Clustering analyses of 2 to 6 in number were performed via both methods, and the same regions mostly fell into the same cluster groups, although in different cluster combinations. The results showed that some provinces with rich forest areas did not produce enough wood. It was observed that these provinces were in the same clusters with provinces having a low amount of forest areas and low wood production. Over the five-year period, very few provinces and regions differed in line with the previous development plans. The creation of a spatial database for wood raw material production using the findings obtained in this study will contribute to the development of operational inventory methods that can be included in long- and medium-term forestry plans.*

Keywords: *wood production; clustering; K-means, Ward's method*

SAŽETAK • *Cilj ovog istraživanja bio je prema sličnosti grupirati preradu drva u regijama i pokrajinama Turske u homogene skupine na temelju podataka o drvnoj industriji u 2013. i 2018. U tom kontekstu primijenjene su hijerarhijska Wardova metoda klasteriranja i nehijerarhijski algoritam K-prosjeka. Analize klasteriranja regija s 2 – 6 klastera provedene su uz pomoć obiju metoda, a iste su regije uglavnom pripadale istim skupinama klastera, iako s različitim kombinacijama klastera. Rezultati su pokazali da neke pokrajine s bogatim šumskim površinama ne*

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prerađuju dovoljno drva. Uočeno je da su te pokrajine svrstane u iste skupine kao i pokrajine s malom količinom šumskih površina i niskom preradom drva. Tijekom petogodišnjeg razdoblja vrlo se malo pokrajina i regija razlikovalo od prethodnih razvojnih planova. Stvaranje sveobuhvatne baze podataka za proizvodnju drvne sirovine uz pomoć nalaza dobivenih u ovoj studiji pridonijet će razvoju operativnih metoda upravljanja zalihama koje se mogu uključiti u dugoročne i srednjoročne planove šumarstva.

Ključne riječi: *prerada drva; klasteriranje; algoritam K-prosjeka; Wardova metoda*

1 INTRODUCTION
1. UVOD

Forests are important examples of sustainable natural resources. A great variety of products can be put on the market by cutting down trees, and the earned income provides good capital to produce more products for the coming years (Tietenberg, 1996; Koulelis, 2009).

Turkey is prominent among the countries with a high utilization rate in regard to rich forest resources and forest products (Istek *et al*., 2017). The latest report on Turkey's forest cover was published in 2015. According to this report, the forest area that amounted to 21.5 million ha in 2010 had reached 22.3 million ha in 2015. This number comprises 29 % of the country's surface area of approximately 78 million ha. According to the data of 2015, 57 % of the forest area (12.7 million ha) with canopy closure of above 10 % is classified, in terms of wood raw material production, as fertile forest, whereas the rest (43 % - 9.6 million ha) with a canopy cover of less than 10 % is considered as infertile forest land, also referred to as unproductive or degraded forest (TAF, 2019). The official database of the General Directorate of Forestry (GDF) reported that by 2020 the fertile forest area reached 13 million ha, and the total forest cover was 22.7 million ha (GDF, 2020).

The wood production and marketing policies of Turkey are based on developments in the market and the raw material expectations of the forest industry. The GDF has increased production substantially when the developments in the economy, the growth potential of the construction sector, and the capacity of the industrial sector to expand are taken into consideration (GDF, 2016). About 75 % of Turkey's wood production is supplied by State forests, both legally (60 %) and "off the books" (15 %), 19 % by private sector production, and 6% by import (Ministry of Development, 2014; TAF, 2019). Figure 1 presents the changes in Turkey's wood production for the years 2013 and 2018. An examination of the figure draws attention to the increase in all production types except for thin poles and fuelwood. Overall, over the five-year period, log and fiber-chip wood display the highest amount of production, whereas telephone poles (118 %) and logs (54.5 %) have the highest rate of change.

Long- and medium-term forestry plans are prepared according to the principle of efficient use of the forest, and the production of wood raw materials is also regulated in accordance with this principle. When preparing these plans, attention must be given to the density and clustering of industrial plantations. It is important that density groups be technically and economically appropriate, environmentally tolerable, and socio-economically and institutionally acceptable. With these features in mind, this study separated Turkey's geography into homogeneous groups according to the similarities in wood production for the years 2013 and 2018. By analyzing the changes of homogeneous groups over the specified years, the aim was to determine to what extent the plans and arrangements made for the sustainability of wood production had been effective. The said years

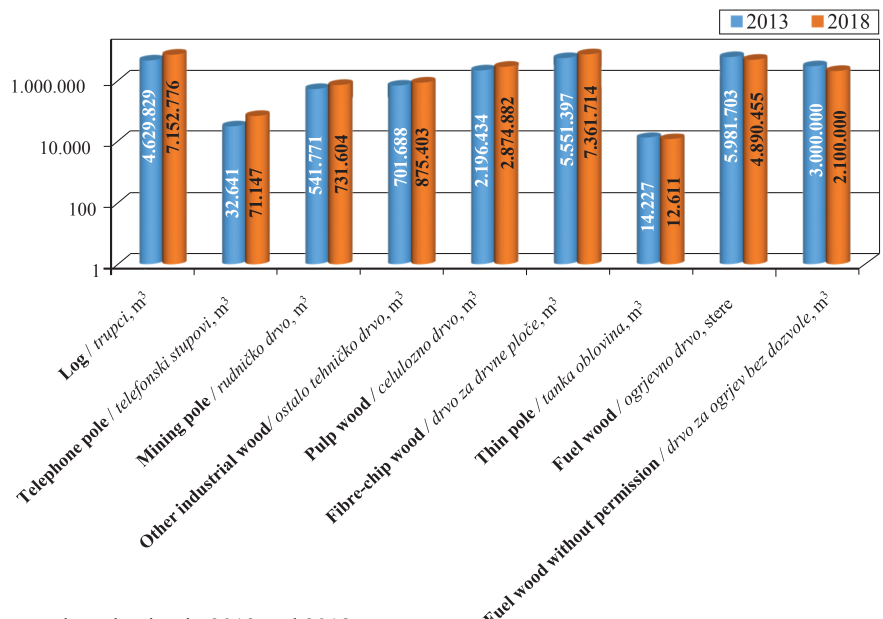

Figure 1 Turkey's wood production in 2013 and 2018.
Slika 1. Prerada drva u Turskoj 2013. i 2018.

cover the last year of Turkey's five-year 9th Development Plan (2013) together with the last year of the 10th Development Plan (2018). In this way, the change in the forestry policies of the country could be followed more easily. Therefore, in order to promote the efficient use of forest resources, it would be possible to determine the necessary changes to be made in the distribution of industrial plantations according to regions. Thus, an effective forest products database could be established to render forestry activities more efficient and to facilitate the follow-up of these activities. This study presents the cluster analyses carried out using K-means and Ward's clustering methods, and the regional and provincial changes observed over the two different periods.

1.1 Literature review
1.1. Pregled literature

A literature view of studies related to clustering analysis methods was carried out. It was observed that Yildirim *et al.* (2008) used hierarchical clustering and discriminant analysis methods in order to identify the status of some wooden panel product groups within the forest products industry in Turkey and the European Union (EU) countries. According to the results, the wooden panel industry in Turkey is capable of competing with the EU countries. Koulelis (2009) also used hierarchical clustering analysis to classify 25 EU member countries depending on logging production between 1992 and 2002. The results indicated that 10 new member countries having forest-covered areas had significantly contributed to Europe's production. Moreover, Michinaka *et al.* (2011) used some indicators to categorize 180 countries in the Global Forest Products Model (GFPM) based on forest products, including plywood, particleboard, paperboard, newsprint, printing and writing paper, and other paper and cardboard. Within this context, they used K-methods and silhouette clustering methods. Furthermore, Caridi *et al.* (2012) investigated the Italian furniture industry's supply chain preferences depending on product modularity and innovativeness. They compared supply chains of firms offering products with modularity and innovativeness at different levels using the K-means method and Pearson distance with factor analysis for clustering. The results revealed that both product features should be taken into account when designing a supply chain. In addition, Hitka *et al*. (2017) developed motivation programs for management and employee groups at a medium-sized wood-processing enterprise in Slovakia using hierarchical clustering analysis. In the study, in which three motivation-oriented clusters were determined for both groups, they indicated that the existing program of the enterprise was incorrectly designed and would have negative effects on personnel. They asserted that their own program would meet most personnel needs and increase the performance of the firm. Akyuz *et al*. (2019) also used hierarchical clustering and discriminant analysis to research the amount of industrial wood production in regional forest directorates in terms of similarities. According to the clustering analysis results, regional forest directorates could be divided into a maximum of six and a minimum of two groups. In another study, Fang *et al*. (2021), using hierarchical cluster analysis, classified poplar clones into different categories according to their growth performance, crown structure, and wood properties.

In addition, Keskin and Demirgil (2009) carried out a work clustering analysis of the Isparta forest products industry via Porter's diamond model. This model was also used in the studies of Karayilmazlar and Uzcan (2016) and Uzcan and Karayilmazlar (2018), who performed clustering and competition analyses of the TR81 Nuts 2 Region Forest Products Industry. Perić *et al*. (2019) applied the two-step cluster method to determine the information technology level of business operations in the Croatian woodworking industry and to measure business performance. On the other hand, with the tests they applied to help improve the wood quality of loblolly pine grown in Brazil, Schimleck *et al*. (2020) grouped trees with similar wood properties using hierarchical complete linkage with square Euclidean distance cluster analysis.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

The dataset of this study consisted of 81 wood production values in seven different geographical regions of Turkey. These data were obtained from the GDF and cover the years 2013 and 2018. Figure 1 gives Turkey's total output value for the specified years. In order to perform clustering analysis on the basis of regions and provinces, the production values of the provinces for logs, telephone poles, mine poles, industrial wood, pulpwood, fiber-chip wood, and thin poles were used. In the analysis phase, the years in question were evaluated independently from one another. Thus, the similarities and differences between 2013 and 2018 were observed. Within this context, first, it had to be determined whether or not the data showed normal distribution. The data regarding variables in the study did not comply with normal distribution, and a high level of positive skewness was found. Therefore, logarithmic transformation, which is used in cases of positive skewness, was applied to the data. As for fuelwood data, it was normalized by converting it to a range of 0-1. After logarithmic transformation, the skewness and kurtosis values of the data for wood production were between +1.5 and -1.5, which is considered normal distribution in the literature (Tabachnick *et al*., 2007; Eryilmaz and Kara, 2018) (Table 1).

The two clustering methods used in the study were Ward's hierarchical method and the non-hierarchical K-means method. In the K-means method, the Silhouette Index was used in order to determine the number of clusters. The Silhouette Index values for wood production are presented in Table 2. The literature indicates that a Silhouette Index of more than 0.5 reveals that the clustering was successful within reason, and a value exceeding 0.7 indicates highly strong clustering (Ng and Han, 1994). Regarding the index values, two is the optimal

Table 1 Skewness and kurtosis values before and after logarithmic transformation
Tablica 1. Asimetričnost i kurtozija prije i nakon logaritamske transformacije

Industrial wood *Tehničko drvo*	**Before logarithmic transformation** *Prije logaritamske transformacije*				**After logarithmic transformation** *Nakon logaritamske transformacije*			
	Skewness *Asimetričnost*		**Kurtosis** *Kurtozija*		**Skewness** *Asimetričnost*		**Kurtosis** *Kurtozija*	
	2013	**2018**	**2013**	**2018**	**2013**	**2018**	**2013**	**2018**
Logs / *trupci*, m^3	3.381	7.725	16.238	62.473	-0.765	0.918	-1.185	-0.891
Telephone poles / *telefonski stupovi*, m^3	3.484	4.383	13.772	21.661	0.855	0.867	-1.098	-1.055
Mine poles / *rudničko drvo*, m^3	2.086	5.413	4.046	37.027	-0.622	-0.776	-1.264	-1.009
Other industrial wood / *ostalo tehničko drvo*, m^3	3.200	7.601	12.256	63.181	-0.567	-0.630	-1.332	-1.159
Pulpwood / *celulozno drvo*, m^3	2.595	8.587	6.876	75.421	-0.609	-0.795	-1.405	-1.088
Fiber-chip wood / *drvo za drvne ploče*, m^3	2.093	6.600	7.435	44.091	-0.941	-1.068	-0.675	-0.401
Thin poles / *tanka oblovina*, m^3	3.024	6.322	9.048	46.036	0.798	0.935	-0.944	-0.632
Fuelwood / *ogrjevno drvo*, stere	1.479	1.819	1.462	3.398	-1.479*	-1.505*	1.462*	1.511*

*After normalization / *nakon normalizacije*

number of clusters, with significant clustering achievable for up to six clusters. Consequently, a higher number of clusters would be better for observing and comparing regional changes. Therefore, the number of clusters in the study was selected as six.

2.1 Clustering analysis
2.1. Analiza klastera

Clustering analysis provides the categorization of units investigated in a study by grouping them based on their similarities, presenting their common features, and determining general definitions related to these categories (Kaufman and Rousseuw, 2009; Dinler, 2014). In parallel with discriminative analysis, it puts similar individuals in the same groups, and similar to factor analysis, it gathers similar variables in the same groups (Cakmak, 1999; Kizgin, 2009).

Clustering analysis methods are divided into two main categories: hierarchical and non-hierarchical clustering analysis. The hierarchical Ward's method is frequently used in clustering analysis and is considered to be a method that gives the best results (Ferreira and Hitchcock, 2009; Everitt *et al*., 2011; Cetinturk and Gencturk, 2020), whereas K-means is identified as the most popular of the non-hierarchical clustering methods (Evans *et al.,* 2005; Yedla *et al*., 2010; Dhanachandra *et al*., 2015).

2.2 K-Means method
2.2. Metoda K-prosjeka

The K-means clustering method (MacQueen, 1967) is widely used to divide a data cluster into k groups automatically (Wagstaff *et al*., 2001). The K-means method can be briefly described as creating various sections from a series of data and evaluating these sections via a specific standard (Tekin and Temelli, 2020). In this method, the k value is identified beforehand, and random points are then selected as cluster centers. All the samples are assigned to the closest cluster center based on the normal Euclidian distance metric. After that, the center of samples in each cluster is calculated. Those centers are accepted as new center values for their own clusters. Finally, the whole process is repeated with the new cluster centers. Repetition continues until points are assigned to each cluster in successive clusters/tours, after which the cluster centers are fixed and remain the same forever (Kilic *et al*., 2020). The K-means assignment mechanism allows each data item to be assigned to only one cluster. Therefore, it is a strict clustering algorithm (Evans *et al*., 2005; Şen and Varürer, 2019).

In the K-means method, the objective function f is minimized using Eq 1 given below (Tucker *et al*., 2010; Kilic *et al*., 2020).

$$f = \sum_{j=1}^{k} \sum_{x_i \epsilon s_j} x_i - c_j^{\ 2} \tag{1}$$

Here, S_j is the data point cluster, c_j is the center of the S_j cluster, x_i is a data point belonging to the cluster, and k represents the number of clusters indicated by the user beforehand.

Although the K-means method has a great advantage in its ease of implementation, it also has some disadvantages. The quality of the final clustering outcomes depends on the arbitrary selection of the cluster centers at the beginning. Consequently, random selection of the centers at the beginning would give different results for different initial centers. Therefore, the first center should be selected meticulously and thus, the desired clustering should be provided. Moreover, computational complexity depending on the amount of data, the number of clusters, and the number of repetitions is another factor that must be taken into account when designing with K-means clustering (Yedla *et al*., 2010; Dhanachandra *et al*., 2015).

Table 2 Silhouette index values for 2013 and 2018
Tablica 2. Vrijednosti indeksa siluete za 2013. i 2018.

Year / *Godina*	**Number of clusters** / *Broj klastera*				
	2	**3**	**4**	**5**	**6**
2013	0.685	0.671	0.590	0.594	0.615
2018	0.682	0.644	0.630	0.624	0.622

2.3 Ward's method
2.3. Wardova metoda

This method, also called the minimum variance method, was proposed by Joe Henry Ward (1963). In

Ward's clustering method, the aim is to minimize the intra-cluster sum of squares (Ozdamar, 2004). This minimizes the variance in clusters and maximizes the distance between clusters (Dardac and Giba, 2011; Atalay, 2019). In this method, the equation for the Error Sum of Squares (*ESS*) is used (Eq 2):

$$ESS = \sum_{i=1}^{n} x_i^2 - \frac{\left(\sum_{i=1}^{n} x_i\right)^2}{n} \quad (2)$$

Here, x_i is the score of the i^{th} observation and n is the amount of data (Aldenderfer and Blashfield, 1984; Celik, 2013). As a result of the analysis via Ward's method, clusters are presented in a diagram called a "dendrogram" in which they come together successfully at different levels (Dibb, 1998; Ozturk, 2012). This method is quite effective and responsive to cross points; however, it tends to create small-scaled clusters (Sekerler, 2008).

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 K-Means cluster results
3.1. Rezultati klasteriranja metodom K-prosjeka

Table 3 gives the means for the final cluster centers at the end of the clustering analysis. High mean values here indicate the clusters where the wood production in question is intense, whereas low values represent the clusters where production is lower compared to other clusters. In addition, the data in the table give information about the reasons for the cluster differences of provinces in groups. For example, even though Clusters-1 and -2 had similar production means in 2013, the fact that the means for telephone pole and thin pole production in the provinces of Cluster-2 was close to 0 indicated that the specified products had not been produced in those provinces and thus, a different clustering was created. Similarly, the differences in Clusters-5 and -6 indicate that almost no production had been carried out in Cluster-6; however, fuelwood and fiber-chip wood were produced in Cluster-5.

Table 4 presents the groups formed as a result of the clustering analysis related to wood production in 2013 and 2018. Clustering is observed to intensify in clusters-1 and -2 that include the provinces with high wood production. When the provinces showing cluster changes in the 5-year process are examined, the provinces of Hakkari, Van, and Mus are identified as producing only fuelwood in 2013 and producing nothing in 2018; in Mardin and Nevsehir, on the other hand, no wood was produced in 2013, but fuelwood and fiber-chip wood production began in 2018. Moreover, Bayburt, Nigde, and Elazig fell within Cluster-4 in 2018. They had produced only fiber-chip wood five years earlier, but in 2018 started to produce all wood products except for telephone and thin poles. In the provinces showing changes in Clusters -1, -2, and -3, in addition to the increase/decrease in their wood production, some product groups such as telephone and thin poles had never been produced or begun to be produced.

Furthermore, it can be stated that the aforementioned clusters also ranked in total wood production; however, the fact that the number of clusters was kept high and there were eight different types of products resulted in some provinces relinquishing one cluster based on their similarities.

The clusters are also presented as colored maps in order to demonstrate more clearly the regional changes in 2013 and 2018 (Figure 2). An overall examination of the figure shows that Clusters-1 and -2 constitute some districts of the Black Sea, Marmara, Aegean, Mediterranean, and Central Anatolian regions where the forest areas are dense, and the provinces in the clusters apart from these two extend out to other regions of Turkey, with changes in the five-year process seen to occur more intensely in these provinces.

3.2 Ward's clustering results
3.2. Rezultati klasteriranja Wardovom metodom

The dendrogram of clustering results obtained via the Ward's hierarchical clustering analysis is given in Table 5. When the dendrogram is examined, Turkey's wood production is shown divided into a maximum of four clusters in 2013 and five clusters in 2018. In the most general categorization, wood production in both 2013 and 2018 is divided into two clusters. Almost all the provinces in the clusters obtained via the

Table 3 Final cluster centers for 2013 and 2018
Tablica 3. Središta finalnih klastera za 2013. i 2018.

Wood product type *Vrsta proizvoda od drva*	**Cluster** / *Klaster*											
	1		2		3		4		5		6	
	2013	2018	2013	2018	2013	2018	2013	2018	2013	2018	2013	2018
Logs / *trupci*	11.42	11.79	10.38	10.97	8.97	9.77	7.60	7.05	0.16	0.12	0.00	0.00
Telephone poles / *telefonski stupovi*	6.50	7.29	0.39	0.15	4.47	2.61	0.00	0.00	0.00	0.00	0.00	0.00
Mine poles / *rudničko drvo*	9.28	9.32	7.65	8.23	6.89	7.69	6.21	6.40	0.44	0.00	0.00	0.00
Other industrial wood *ostalo tehničko drvo*	8.73	9.06	8.54	8.70	3.12	3.38	7.13	5.77	0.35	0.27	0.00	0.00
Pulpwood / *celulozno drvo*	10.64	10.56	9.50	10.15	8.52	9.02	4.35	4.14	0.13	0.48	0.00	0.00
Fiber-chip wood / *drvo za drvne ploče*	11.59	11.90	10.82	11.17	1.36	2.23	8.23	7.53	2.92	3.03	0.00	0.00
Thin poles / *tanka oblovina*	4.40	4.07	1.22	1.06	6.42	4.30	1.59	1.01	0.20	0.00	0.00	0.00
Fuelwood / *ogrjevno drvo*	11.67	11.40	11.05	10.91	9.45	9.04	8.62	8.15	8.59	8.04	1.34	0.00

Table 4 K-Means clustering analysis results
Tablica 4. Rezultati klasteriranja metodom K-prosjeka

Cluster No. *Br. klastera*	**2013**			**2018**		
Cluster-1 *klaster-1*	Balıkesir Çanakkale Aydın Denizli Muğla Manisa Kütahya Uşak	Bursa Eskişehir Bilecik Bolu Ankara Antalya Burdur Adana	Sivas Yozgat Karabük Kastamonu Sinop Samsun Çorum Ordu	Balıkesir Çanakkale Aydın Denizli Muğla Manisa Kütahya Uşak	Bursa Eskişehir Bilecik Bolu Ankara Antalya Burdur Adana	Sivas Yozgat Kastamonu Sinop Samsun Çorum Ordu ***Kahramanmaraş*** **(↑)** ***Osmaniye*** **(↑)**
Cluster-2 *klaster-2*	İstanbul Tekirdağ Edirne Kırklareli İzmir Kocaeli Sakarya Düzce	Yalova Konya Isparta Mersin Hatay Osmaniye Zonguldak Bartın	Çankırı Tokat Amasya Trabzon Giresun Artvin Gümüşhane Afyonkarahisar Kahramanmaraş	İstanbul Tekirdağ Edirne Kırklareli İzmir Kocaeli Sakarya Düzce	Yalova Konya Isparta Mersin Hatay Zonguldak Bartın Çankırı	Tokat Amasya Trabzon Giresun Artvin Gümüşhane Afyonkarahisar ***Rize*** **(↑)** ***Karabük*** **(↓)** ***Karaman*** **(↑)**
Cluster-3 *klaster-3*	Kars	Ardahan		Kars	Ardahan	***Erzurum*** **(↑)**
Cluster-4 *klaster-4*	Karaman Kayseri	Rize Erzincan	Gaziantep Kilis	Kayseri Erzincan	Gaziantep Kilis	***Elâzığ*** (↑) ***Bayburt*** (↑) ***Niğde*** (↑)
Cluster-5 *klaster-5*	Kırıkkale Aksaray Niğde Kırşehir Bayburt Malatya	Elâzığ Bingöl Tunceli Van Muş Bitlis	Hakkâri Adıyaman Şanlıurfa Diyarbakır Batman Şırnak Siirt	Kırıkkale Aksaray Kırşehir Malatya Bingöl	Tunceli Bitlis Adıyaman Şanlıurfa Diyarbakır	Batman Şırnak Siirt ***Mardin*** (↑) ***Nevşehir*** (↑)
Cluster-6 *klaster-6*	Nevşehir Ağrı	Iğdır Mardin	Erzurum	Ağrı Iğdır	***Van*** (↓) ***Muş*** **(↓)**	***Hakkâri*** (↓)

*Provinces in bold and italics showed a change in 5 years; provinces with (↑) sign reached a higher number of clusters; provinces with (↓) sign were relinquished at the end of 5 years / *Pokrajine napisane zadebljanim i kosim slovima pokazale su promjenu unutar pet godina; pokrajine sa znakom (↑) dosegnule su veći broj klastera; pokrajine sa znakom (↓) otpale su na kraju 5-godišnjeg razdoblja*

Ward's method are in the same cluster as the provinces in the clusters created via the K-means method. These similarities confirmed that Turkey's wood production had been categorized properly and successfully. As distinct from the K-means, the Ward's method reduced the number of clusters only by uniting some clusters. Indeed, the situation was indicated at the beginning of the study with the Silhouette Index values. It was also emphasized that clusters of two to six in number could be successfully categorized. According to Caglar (1990), the fact that the same regions remained within almost the same clusters at different cluster combinations can be considered as an important sign indicating the significance of the findings.

4 CONCLUSIONS
4. ZAKLJUČAK

This study utilized hierarchical and non-hierarchical clustering analysis methods to separate Turkey's forestry sector into homogenous clusters and investigated regional and provincial changes and similarities within a 5-year process.

In the study, the analyses made with both K-means and Ward's methods showed similarities. In other words, an increase or decrease in the number of homogenous clusters did not cause any provinces to be placed into different clusters. The provinces divided into six clusters via the K-means method were allocated to fewer clusters in the Ward's method solely to enable the change to be observed more clearly.

The evaluation revealed that during the five-year process, in some provinces such as Nigde, Elazig, Bayburt, Mardin, and Nevsehir, despite having less forest area, wood production had begun, whereas in Hakkari, Van, and Mus, wood production had completely stopped. Some provinces showed changes in their clusters from 2013 to 2018.

The aforementioned clustering results also give information about the effective use of the forest areas. The clustering results determined that some provinces with rich forest areas did not produce enough wood. It

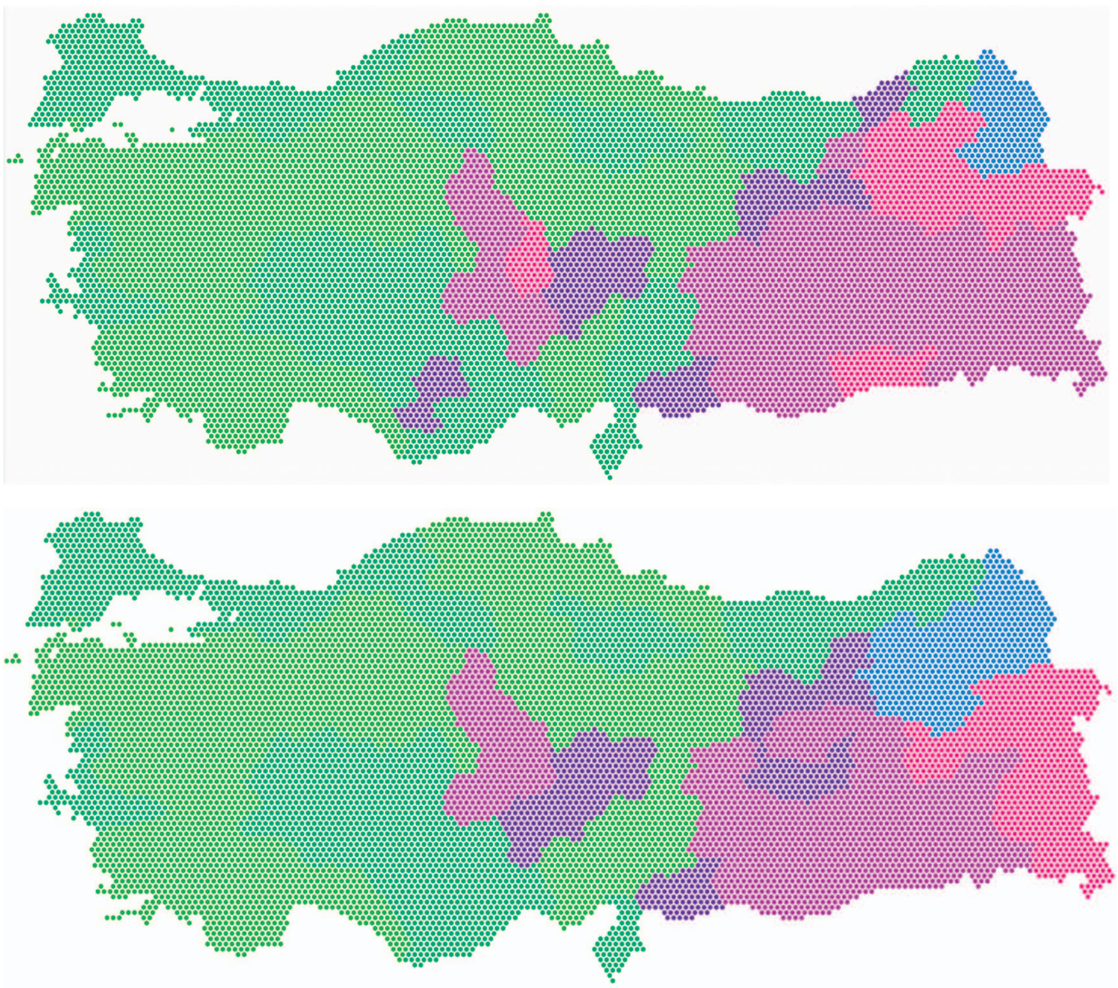

Figure 2 Regional changes in 2013 and 2018 based on K-means analysis
Slika 2. Regionalne promjene u 2013. i 2018. na temelju analize metodom K-prosjeka

was also observed that these provinces were located in the same clusters with provinces having a low amount of forest area and low wood production. This situation leads to inefficient use of forest resources and consequently, needs to be rectified by taking into account the factors included in the development plans, thus ensuring the sustainability of forest resources.

When the clustering results for 2013 and 2018 were compared, the systems and training in wood production did not show sufficient development and therefore, the necessary professionalization could not be achieved. In addition, wood production not only varied according to the type of wood, but also varied according to the climatic differences among regions. In regions where different climatic circumstances are experienced, production activities are losing pace under aggravated working conditions. In order to meet the needs of the wood raw material market, it is important to establish regeneration and maintenance areas within the scope of economic management, taking into account production costs and silvicultural principles. This situation has been partially resolved in provinces where temporal and spatial arrangements have been made and is seen in the change between clusters.

The results of the study can contribute to the development of operational inventory methods by creating a spatial database for wood raw material production. Therefore, it can provide economic and technical integration in terms of making annual applications and monitoring of long-term national forestry or development plans and medium-term forestry plans for management and silviculture.

Because Turkey is in the position of an importer with regard to forest products, it is essential to develop production in a more systematic and projectized way. Thus, imports will decrease, and production will increase in provinces that are rich in forest areas, with those that are unproductive in terms of wood products remaining in low-level clusters. Moreover, in the regions belonging to Clusters-5 and -6 where wood production is either very low or non-existent, building industrial plantations and planting the appropriate species and clones demanded by the country would be an effective way to end the deficit.

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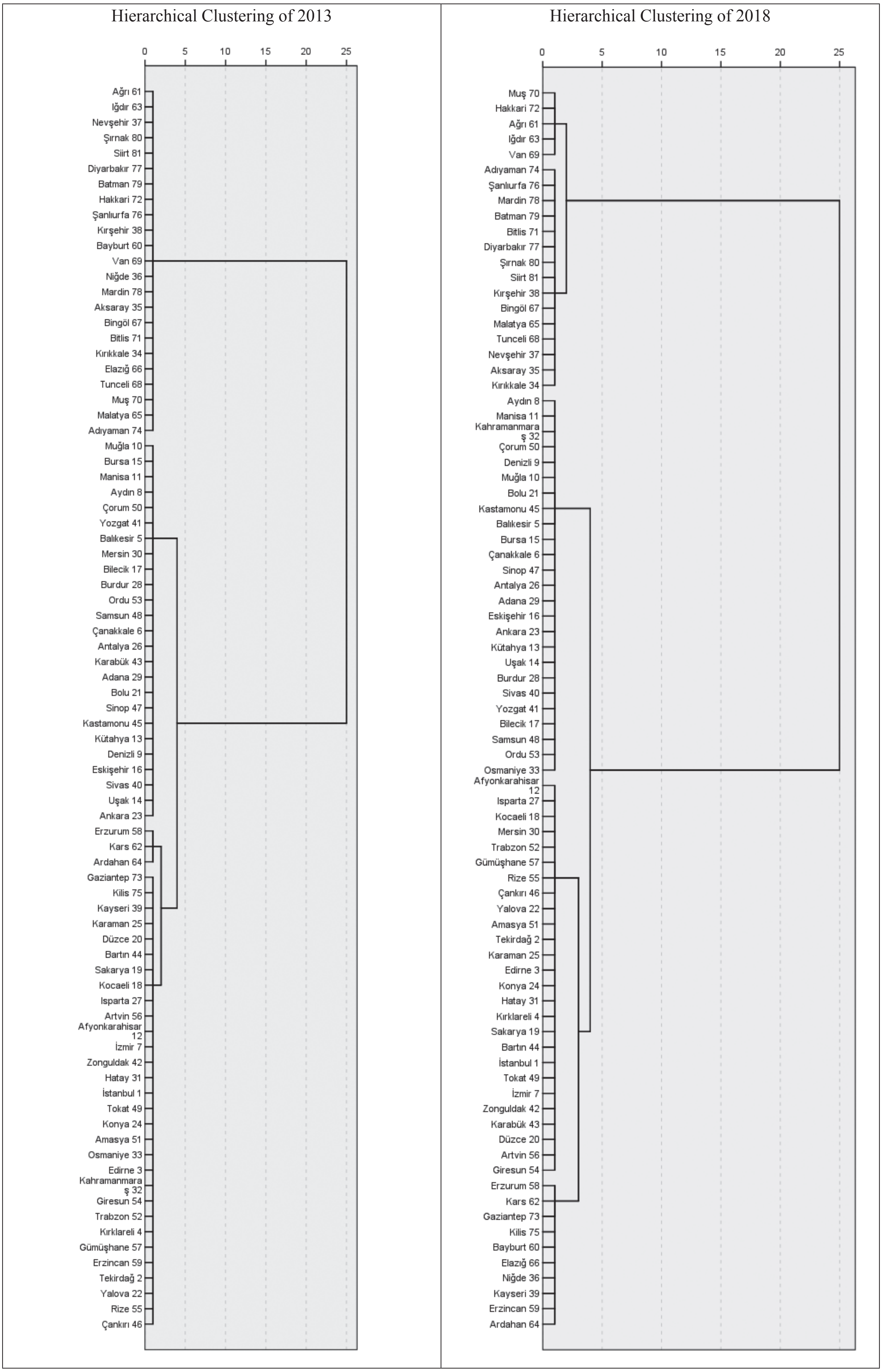
Table 5 Ward's method clustering analysis results
Tablica 5. Rezultati klasteriranja Wardovom metodom



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Optimization of Production Parameters in Oriented Strand Board (OSB) Manufacturing by Using Taguchi Method

Optimizacija parametara u proizvodnji OSB ploča primjenom Taguchijeve metode

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ABSTRACT • *Optimization of process conditions in oriented strand board (OSB) manufacturing is a very important issue for both reducing cost and improving the quality of panels. Taguchi experimental design technique was applied to determination and optimization of the most influential controlling parameters of OSB panels such as press condition (pressure-time-temperature) and the ratio of adhesive parameters on modulus of elasticity (MOE). The value of the MOE is one of the very important mechanical properties of OSB panels. For this purpose, several experiments were conducted according to Taguchi L27 orthogonal design. The signal-to-noise (S/N) and the analysis of variance (ANOVA) were used to find the optimum levels and to indicate the impact of the controlling parameters on MOE. A verification test was also performed to prove the effectiveness of Taguchi technique. Since the predicted and the measured values were very close to each other, it was concluded that the Taguchi method was very successful in the optimization of effective parameters in OSB's manufacturing.*

Keywords: *oriented strand board; production parameters; optimization; Taguchi method*

SAŽETAK • *Optimizacija parametara u proizvodnji ploča s orijentiranim iverjem (OSB ploča) vrlo je važna kako za smanjenje troškova proizvodnje, tako i za poboljšanje kvalitete ploča. Za određivanje najutjecajnijih kontrolnih parametara tijekom proizvodnje OSB ploča i za njihovu optimizaciju primijenjena je Taguchijeva metoda projektiranja eksperimenta. Ispitan je utjecaj uvjeta prešanja (tlaka, vremena i temperature) i omjera parametara lijepljenja na modul elastičnosti (MOE) ploča. Vrijednost modula elastičnosti vrlo je važno mehaničko svojstvo OSB ploča. Stoga je provedeno nekoliko pokusa prema ortogonalnom dizajnu Taguchi L27. Za pronalaženje optimalnih veličina i određivanje utjecaja kontrolnih parametra na modul elastičnosti primijenjeni su omjer signala i šuma (S/N) i analiza varijance (ANOVA). Također je provedena verifikacija kako bi se dokazala učinkovitost Taguchijeve metode. Budući da su predviđene i izmjerene vrijednosti bile vrlo blizu jedne drugima, zaključeno je da je Taguchijeva metoda bila vrlo uspješna u optimizaciji utjecajnih parametara u proizvodnji OSB ploča.*

Ključne riječi: *OSB ploča; proizvodni parametri; optimizacija; Taguchijeva metoda*

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1 INTRODUCTION
1. UVOD

Oriented strand boards (OSB) are a relatively new kind of wood-based panels that are defined in the European Standard. Particle boards are classified depending on the size and orientation of their components (Rebollar *et al*., 2007). OSB is a multilayer structural panel made from wood strands by drying, sizing, oriented formation and hot pressing, in which the strands comprise uniformly thick and long wood shavings (Sumardi *et al*., 2007). OSBs are made of a three-layer mat called strand with highly densified core. While the strands in the surface layers are aligned in parallel to the board length or width, the strands in the core layers can be randomly oriented or perpendicular to the surface layers for enhancing product strength and stiffness (Semple *et al*., 2015). OSBs, wood-based panel products are classified into four types ranging from general purpose boards to high load-bearing boards in the humid conditions according to the European Standard (EN 300: 2006). Oriented strand board has been produced as a structural panel material, replacing softwood plywood in North America since the early 1980s (Spelter *et al.,*1997). OSB market is also growing worldwide, and it is expected a growth rate of nearly 28 % until 2022 (Ferro *et al*., 2018).

OSB has been mostly used in building, construction, transportation, furniture and packaging industries. In the field of civil engineering, as a structural or non-structural panel, OSB is gradually accepted on the construction market. Furthermore, OSB is commonly used as shear wall material of buildings and in the construction of furniture skeleton (Jin *et al.*, 2016). The application of OSB panels is very significant in construction and furniture industry. With the general decrease of forest resources in the world, the use of OSB in many areas, especially in furniture manufacturing and civil construction, as well as wall coverings and roof panels, becomes essential (Kasal, 2008; Benetto *et al*., 2009).

Mechanical and physical properties of OSB panels can be affected by many factors such as raw materials, pressing parameters (pressure-temperature and time), type of adhesive, adhesive ratio, and density profile of the panel (Ciobanu *et al*., 2014). Physical and mechanical properties of OSB are in agreement with the changes in chemical components caused by the treatments (Fatrawana *et al*., 2019). As a result of drying and hot-pressing processes in the manufacturing of OSBs, volatile organic compounds (VOC) are emitted into the atmosphere.

The majority of wood-based panels rely on thermosetting polymers. Urea formaldehyde adhesive can be especially chosen due to its suitable combination, including low cost, fast reaction time in hot press, low cure temperatures, and excellent thermal properties (Aydın *et al*., 2006). The OSB manufacturer uses water resistant adhesive such as isocyanates (polymeric diphenyl methane diisocyanate (PMDI)), phenol-formaldehyde (PF), melamine-urea-phenol formaldehyde (MUPF) or UF-melamine resins. OSB is produced to meet specific requirements related to size and thickness of strand, density, and texture. These products are widely used for indoor and outdoor structural applications (Dumitrascu *et al*., 2020).

There are a lot of factors like the type of raw material, size of the particle, adhesive ratio and press conditions (time-pressure-temperature) that affect the properties of OSBs. It is stated that a good performance of the OSB product can be achieved by using optimum process parameters (Barnes, 2000). Experimentation is a frequent task to measure and analyze the output, and for this purpose, engineers/researchers use many tools like statistics, analytical models, etc., regardless of their background (Bisgaard, 1991). In today's era, the purpose of experiments in industries is essentially optimization and robust design analysis (Davis and John, 2018). Design of Experiments (DoE) is one of the most important tools used in determining optimum conditions. DoE techniques enable designers to determine simultaneously the individual and interactive effects of many factors that could affect the output in any design. DoE also provides a full insight of interaction between design elements. Smardzewski (2019) examined experimental and numerical analysis of wooden sandwich panels with an auxetic core and oval cells. Velosa *et al*. (2013) applied Taguchi method to design the experiment and investigate the influence of various parameters of compression molding process on the mechanical properties of composite panels. Auchet *et al*. (2018) designed and conducted the drilling experiments according to Taguchi's method to predict the influence of cutting parameters on thrust force in drilling of particleboard panels. Response surface methodology has been adopted by *Davim et al*. (2008) to investigate the relationships and parametric interaction between two controllable variables, namely, feed rate and cutting speed on the delamination factor at entry and exit of the holes in drilling of MDF.

The factors that affect the mechanical properties of OSB panels can be expressed as adhesive type and amount, type of raw material, moisture content, chip or strand size, and press conditions (time-pressure-temperature, etc.). On the other hand, the *MOE* value is a mechanical property that should be considered in applications such as the building construction sector and interior decoration. Therefore, it is necessary to create optimal production conditions and determine the effect of the factors affecting the amount of *MOE*. This study aims to determine and optimize the most influential controlling parameters of OSB panels such as press condition (pressure-time-temperature) and the ratio of adhesive parameters on the modulus of elasticity (*MOE*). For this purpose, the Taguchi experimental design technique is applied and the results are analyzed in detail.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Manufacturing of OSB panels
2.1. Proizvodnja OSB ploča

Scots pine (*Pinus sylvestris* L.) was used in the production of the oriented strand boards (OSB). The strands dimension was approximately 80 mm long, 20 mm wide, and 0.7 mm thick. First, the wood strands were dried to 2 or 3 % moisture content before adhesive was sprayed on them. Then, phenol formaldehyde adhesive was applied in 6, 9 and 12 percent ratios based on the weight of oven dry wood strands. The OSB panels were produced under the fallowing conditions: press time (3, 6 and 9 minutes), temperature of press plates was (175, 185 and 195 °C) and press pressure (30, 40 and 50 kg/cm^2). The shelling ratio was 40 % for core layer and 60 % for face layer, and density of the boards was aimed at 0.65 g/cm^3. Mats having dimensions of 56 cm × 56 cm × 1.2 cm were formed. OSB panels were totally produced in this study and labeled from 1 to 27. All mats were pressed under automatically controlled conditions. After pressing, the boards were conditioned to constant weight at (65±5) % relative humidity and at a temperature of (20±2) °C until they reached stable weight.

2.2 Determination of modulus of elasticity
2.2. Određivanje modula elastičnosti

The modulus of elasticity in bending strength is measured according to EN 310: 1996 standard. The values of modulus of elasticity of test panels produced in this study were determined by using Zwick/Roell Z050 universal test device with the capacity of 5000 kg. This machine is able to record the loading wedge movement (with a precision of 0.01 mm) and load (with a precision of 0.01 N) every 1 s. In testing, the loading mechanism was operated with a velocity of 5 mm/min. The calculation of *MOE* was based on the forces measured at 15 % and 45 % of the maximum loading force (force of destruction), and the corresponding deflections of the bent beam were measured by extensometer. The *MOE* was calculated using Eq 1:

$$MOE = \frac{l^3 \cdot (F_2 - F_1)}{4 \cdot b \cdot h^3 \cdot (u_1 - u_2)} \quad (1)$$

where l is the span of supports (360 mm), F_2 and F_1 are forces at 5 % and 15 % level of the maximum force F_{max}, b is the width of the cross-section of sample and h is the thickness of sample (height of cross-section), $u_{45\%}$ and $u_{15\%}$ are deflections at forces $F_{45\%}$ and $F_{15\%}$. The values of *MOE* calculated from the five measured specimens from each board were averaged.

2.3 Taguchi method
2.3. Taguchijeva metoda

The technique of defining and investigating some conditions in an experiment involving multiple factors is known as the experimental design. In the literature, this technique is also referred to as factorial design. Experimental design methods were developed originally by Fisher (1992). However, the classical experimental design method is complex, time-consuming and is not efficient under today's industrial competitive conditions. Furthermore, a large number of experiments have to be carried out when the number of the process parameters increases. To reduce the number of experiments, Taguchi used a special design of orthogonal arrays to study the entire parameter space (matrix of experiments) with a smaller number of experiments. With the help of this matrix, the maximum information from a minimum number of trials and the best level of each parameter can be obtained for an objective function. The steps to be followed to design the experiment according to the Taguchi method are as follows:

1. Identify the signal and noise factor. Signal factors are the input parameters of the problem that change during the experiment to obtain the optimal conditions. Noise factors are also called the all factors that cause changes but are assumed constant during the tests.
2. Determine the number of levels for the parameters and possible interactions between the parameters.
3. Select the appropriate orthogonal array and assign the parameters to the orthogonal array.
4. Conduct the experiments based on the arrangement of the orthogonal array.
5. Calculate the response of the experimental trails using the signal-to noise (*S*/*N*) ratio and performing a statistical analysis of variance (ANOVA) to see which parameters are statistically significant. There are three categories of quality characteristic in the analysis of the *S*/*N* ratio. Lower is better, nominal is better, higher is better, and these can be expressed by the following equations (Agrawal *et al*., 2018).
   a. The lower is better characteristics are expressed by the following equation:
   $$S/N = -10 \log_{10}\left(\frac{1}{n}\sum h^2\right) \quad (2)$$
   b. The nominal is better characteristics are expressed by the following equations:
   $$S/N = -10 \log_{10}\left(s^2\right) \quad (3)$$
   $$S/N = 10 \log_{10}\left(\frac{y^2}{s^2}\right) \quad (4)$$
   c. The larger is better characteristics are expressed by the following equation:
   $$S/N = -10 \log_{10}\left(\frac{1}{n}\sum \frac{1}{h^2}\right) \quad (5)$$

In the above equations, n – number of cases / test runs; – experimental results/data; where Y – mean of responses for a combination of selected factor level; s = standard deviation of the responses for given factor-level combination.

6. Select the optimal levels of parameters.
7. Verify the optimal parameters through the confirmation experiment.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

The density and moisture content values of OSBs were determined according to the applicable standards (TS EN 323, 1999; TS EN 322, 1999). It was observed that the obtained density and moisture content values of the test samples changed between 0.60 - 0.72 g/cm^3 and 5 - 7 %, respectively. In the study, it was seen that the values obtained from experimental studies were in harmony with the targeted moisture content and den-

Table 1 Selected experimental parameters and their assigned levels
Tablica 1. Odabrani eksperimentalni parametri i njihove dodijeljene razine

Symbol *Simbol*	**Parameters** *Parametri*	**Levels of parameters** *Razina parametara*		
		1	**2**	**3**
A	Pressure / *tlak,* kg/cm^2	30	40	50
B	Time / *vrijeme,* min	3	6	9
C	Temperature *temperatura,* °C	175	185	195
D	Adhesion rate *stupanj adhezije,* %	6	9	12

sity values. In this study, the parameters of press pressure, press time, press temperature and adhesion ratio were considered as controllable (signal) factors that have three levels each (Table 1). Other parameters were considered as non-controllable (noise) factors and were considered constant during the tests. The effects of four signal factors, each at three levels, on the value of MOE were studied.

Considering the input parameters and their levels, L27 orthogonal arrays were selected as the experimental matrix. To analyze the experimental result, the interactions among the main factors were not considered and the *S/N* ratio was computed for each experiment.

Table 2 presents the experimental plan and results as an L27 orthogonal array and the corresponding *S/N* ratio. The largest value of *MOE* is very important for quality improvement of the product. For this reason, the ‘‘larger-the-better’’ concept has been applied for the calculation of *S/N* ratios. The influence of control parameters such as press pressure, press time, temperature of the press plate, and the adhesive ratio on the *MOE* was analyzed using *S/N* response table (Table 3). In Table 3, the delta value is equal to the difference between maximum and minimum *S/N* ratios values for levels of each parameter, and the influence order of parameter was determined by comparison of delta values.

The level values of control factors for *MOE* given in Table 3 are shown in graph forms in Figure 1. The optimum set of parameters for obtaining maximum *MOE* can be determined by selecting the largest value

Table 2 Orthogonal array of L27 for modulus of elasticity
Tablica 2. Ortogonalni niz L27 za modul elastičnosti

Exp. No *Broj eksperimenta*	**Main parameters** *Glavni parametri*				***MOE***, N/mm^2	***S/N***
	A	B	C	D		
1	30	3	175	6	5946	75.48
2	30	3	175	9	3034	69.64
3	30	3	175	12	2396	67.59
4	30	6	185	6	6817	76.67
5	30	6	185	9	8067	78.13
6	30	6	185	12	3655	71.26
7	30	9	195	6	5204	74.33
8	30	9	195	9	7537	77.54
9	30	9	195	12	5404	74.65
10	40	6	195	6	6796	76.65
11	40	6	195	9	7876	77.93
12	40	6	195	12	6722	76.55
13	40	9	175	6	6769	76.61
14	40	9	175	9	7490	77.49
15	40	9	175	12	6131	75.75
16	40	3	185	6	6542	76.31
17	40	3	185	9	5839	75.33
18	40	3	185	12	2472	67.86
19	50	9	185	6	5641	75.03
20	50	9	185	9	6227	75.89
21	50	9	185	12	5238	74.38
22	50	3	195	6	4779	73.59
23	50	3	195	9	2998	69.54
24	50	3	195	12	2308	67.26
25	50	6	175	6	6223	75.88
26	50	6	175	9	8222	78.30
27	50	6	175	12	5406	74.66

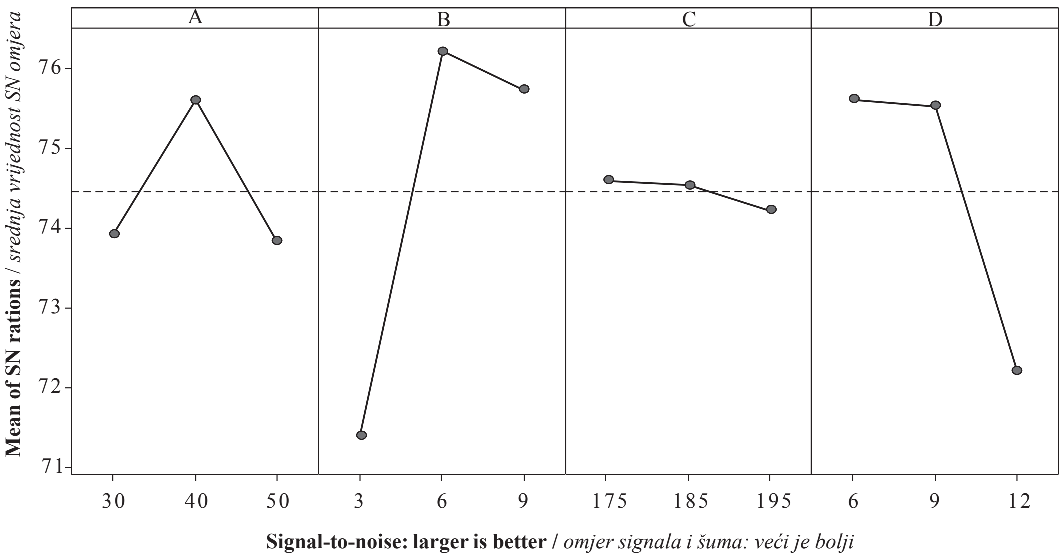

Figure 1 Effects of control parameters on *MOE* (SNR data)
Slika 1. Utjecaj kontrolnih parametara na modul elastičnosti (SNR podatci)

Table 3 Response table for signal to noise ratios
Tablica 3. Tablica odgovora za omjere signala i šuma

Level	A	B	C	D
1	73.92	71.40	74.60	75.62
2	**75.61**	**76.22**	**74.54**	**75.53**
3	73.84	75.74	74.23	72.22
Delta	1.77	4.82	0.37	3.40
Rank	3	1	4	2

of *S*/*N* ratio for each factor. Hence, the optimum control parameter level is A2 (factor A at level 2, *S*/*N* = 75.61), B2 (factor B at level 2, S/N = 76.22), C1 (factor C at level 1, *S*/*N* = 74.60), and D1 (factor D at level 1, *S*/*N* = 75.62). In other words, an optimum *MOE* value was obtained at a press pressure of 40 kg/cm^2, press time of 6 min, temperature press plate of 175 °C, and at the ratio of adhesive of 6 %.

ANOVA is a statistical method used to determine the individual interactions of all of the control factors in the test design. In this study, analysis of variance was used to determine the percent contribution of each parameter on output (*MOE*). However, in order to determine whether the parameters used in ANOVA analysis have a statistically significant effect on the result, an analysis called *F*-test is required. The results of ANOVA analysis of *S*/*N* ratios are shown in Table 4. This analysis was carried out at a 5 % significance level and a 95 % confidence level.

With *F*-distribution table for alpha = 0.05, with numerator of degrees of freedom 2 and denominator degrees of freedom 18 (df for Error), the *F* critical value was found to be 3.5546. Therefore, according to the values obtained in Table 4, it was concluded that just parameters B and D have a significant effect on the result.

As shown in Table 4, the percentage contributions of the A, B, C and D factors on the *MOE* were found to be 7.51 %, 42.43 %, 0.27 % and 24.31 %, respectively. Thus, the most important factor affecting the *MOE* is "Time" (factor B, 42.43 %).

3.1 Test of confirmation
3.1. Potvrdni test

The final step in the design of experiments with the Taguchi method is to perform a validation test. The purpose of the validation test is to confirm the results and discussions in the analysis section. The confirmation test is a crucial step and is highly recommended by Taguchi to verify the experimental results as stated by Ross (1996). The results of confirmation experiments in Table 5 show the comparison of the predicted by using design of experiment and experimental values.

The predicted and calculated results have very close values with the experimental results. Therefore, the results obtained from the confirmation tests reflect successful optimization.

4 CONCLUSIONS
4. ZAKLJUČAK

In this study, four main parameters in the production of oriented strand boards including temperature, pressure, time and adhesive ratio at three different levels were investigated using Taguchi's experimental design. The purpose of the design of the experiment is to achieve the optimal setting of control parameters which would result in maximum modulus of elasticity. In addition to these analyzes, analysis of variance was used to determine the percentage contribution of each parameter on output (modulus of elasticity). Furthermore, the validity of the approach was tested. Accordingly:

- The optimum levels of the control parameter for maximizing the modulus of elasticity using S/N rates were determined. The optimal conditions for this output are at A2B2C1D1 (i.e., Pressure = 40 kg/cm^2, Time = 6 min., Temperature=175 °C and adhesive ratio = 6 %)
- According to the results of statistical analyses, it was found that the "time" and "adhesive ratio" were the most significant parameters for MOE value with a percentage contribution of 42.43 % and 24.31 %, respectively.

Table 4 Result of ANOVA analysis
Tablica 4. Rezultati ANOVA analize

Source	*DF*	Adj SS	Adj MS	*F*-value	*P*-value	Contribution, %
A	2	6172788	3086394	2.65	0.098	7.51
B	2	34871788	17435894	14.99	0.000	42.43
C	2	221781	110890	0.10	0.910	0.27
D	2	19979775	9989887	8.59	0.002	24.31
Error	18	20937379	1163188			25.48
Total	26	6172788	3086394	2.65	0.098	100.00

Table 5 Comparative results for confirmation tests
Tablica 5. Usporedni rezultati za potvrdne testove

	Optimal parameter condition *Optimalni uvjeti parametara*				The value of *MOE* *Vrijednost MOE*		Error *Pogreška,* %
	Pressure *Tlak,* kg/cm^2	Time *Vrijeme*, min	Temperature *Temperatura,* °C	Adhesive ratio *Omjer adhezije,* %	Predicted *Predviđena*	Experimental *Eksperimentalna*	
Optimum	40	6	175	6	7468	7437	0.41
Random 1	50	9	185	6	5593	5641	0.85
Random 2	30	6	195	12	5061	4932	2.58

- According to the confirmation test, the results and discussions in the analysis section were verified.

Researchers studying this subject can optimize other mechanical properties (withdraw screw or nail, shock strength, internal bond, etc.) and physical properties (water absorption, thickness swelling, surface roughness, etc.) of OSBs by Taguchi or other experimental design methods according to the type of OSB panels.

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Decay Resistance and Color Change of Pine and Beech Wood Impregnated with *R. Luteum* and *R. Ponticum* Plant Extracts

Otpornost borovine i bukovine impregnirane ekstraktima biljaka *R. luteum* i *R. ponticum* na propadanje i promjenu boje

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ABSTRACT • *In this study, the effect of impregnation with natural extracts on decay resistance and color change of pine and beech wood was analyzed. Flowers of Rhododendron luteum and Rhododendron ponticum plants were extracted according to the decoction method and aqueous solutions were prepared at different concentration levels (2 %, 4 % and 7 %). In addition, ferrous sulfate, copper sulfate and aluminum sulfate mordants were added to the solution to improve the properties of the extracts. Then the wood specimens were impregnated with the prepared solutions. The results indicated that the effect of plant species on the mass loss of specimens exposed to T. versicolor (white-rot fungus) was insignificant. Non-mordant extracts had a slight effect on the mass loss of the specimens. However, in pine and beech specimens impregnated with mordant-added (especially ferrous sulfate-added) extracts, mass loss was significantly reduced and resistance to fungal rot was almost completely achieved. The concentration level did not have a significant effect on the mass loss of specimens treated with mordant-added extracts. After impregnation, the L* value of all specimens (especially those treated with ferrous sulfate-added extracts) decreased and the specimens darkened. The a* and b* values increased in specimens treated with non-mordant and aluminum sulfate-added extracts and these specimens tend to have a red-yellow color. The a* value decreased and the b* value increased in wood specimens treated with copper sulfate-added extracts. The green-yellow color trend of these specimens increased. Both the a* and b* values of the specimens treated with ferrous sulfate-added extracts decreased and the green-blue color tendency increased in these specimens. The increase in the concentration level positively affected the determined color changes. The total color change (**Δ**E*) was higher in wood specimens (especially pine) treated with ferrous sulfate-added R. ponticum extracts.*

Keywords: *color change; decay resistance; impregnation; natural extract; wood material*

SAŽETAK • *U istraživanju je analiziran utjecaj impregnacije borovine i bukovine biljnim ekstraktima na otpornost na propadanje. Cvjetovi biljaka Rhododendron luteum i Rhododendron ponticum ekstrahirani su metodom dekocije te su pripremljene vodene otopine različitih koncentracija (2 %, 4 % i 7 %). Osim toga, otopini su dodani fiksatori željezov sulfat, bakrov sulfat i aluminijev sulfat radi poboljšanja svojstava ekstrakata. Zatim su uzorci*

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drva impregnirani pripremljenim otopinama. Rezultati su pokazali da je utjecaj vrste biljke na gubitak mase uzoraka izloženih djelovanju T. versicolor (gljivi bijele truleži) bio neznatan. Ekstrakti bez fiksatora imali su mali učinak na gubitak mase uzoraka. Međutim, znatno je smanjen gubitak mase uzoraka borovine i bukovine impregniranih ekstraktima s dodatkom fiksatora (osobito željezova sulfata), a postignuta je gotovo potpuna otpornost na gljive truležnice. Koncentracija otopine nije imala znatniji utjecaj na gubitak mase uzoraka zaštićenih ekstraktima s dodatkom fiksatora. Nakon impregnacije smanjila se L vrijednost svih uzoraka (posebno onih zaštićenih ekstraktima s dodatkom željezova sulfata) i uzorci su potamnjeli. Povećale su se vrijednosti a* i b* uzoraka impregniranih ekstraktima bez dodatka fiksatora i ekstraktima s dodatkom aluminijeva sulfata i ti su uzorci poprimili crvenožutu boju. Vrijednost a* uzoraka drva premazanih ekstraktima s dodatkom bakrova sulfata smanjila se, a vrijednost b* se povećala. Udio žutozelene komponente na tim se uzorcima povećao. Vrijednosti a* i b* uzoraka premazanih ekstraktima s dodatkom željezova sulfata smanjile su se i povećao se udio zelenoplave komponente. Povećanje koncentracije otopine kojom su uzorci impregnirani pozitivno je utjecalo na promjenu boje. Ukupna promjena boje (ΔE*) bila je veća na uzorcima drva (osobito borovim) impregniranim ekstraktima R. ponticum s dodatkom željezova sulfata.*

Ključne riječi: *promjena boje; otpornost na propadanje; impregnacija; prirodni ekstrakt; drvni materijal*

1 INTRODUCTION
1. UVOD

Wood materials are structural materials that can generally degrade under the influence of biotic and abiotic factors. Therefore, this material, which has a certain service life, can be made resistant to biological, physical and chemical factors, and its technical and economic losses can be reduced or eliminated completely. For this purpose, methods such as impregnation, upper surface treatments and various modification processes are used for the protection of wood. Among these methods, deep impregnation of organic or inorganic based biocides into wood is highly preferred from past to present.

With the increasing environmental awareness in recent years, the restriction or complete prohibition of the use of inorganic based preservative salts has increased the interest in the development of environmentally friendly preservatives in the field of wood protection. In this context, one of the subjects that are considered as a scientific alternative and studied scientifically is herbal extracts and tannins (Goktas *et al.,* 2008; Tascioglu *et al.,* 2013; Broda, 2020). These extractives are compounds that naturally protect wood from biological degradation (Toshiaki, 2001; Windeisen *et al.,* 2002; Kartal *et al.,* 2006; Lin *et al.,* 2007; Goktas *et al.,* 2010; Ozen *et al.,* 2014; Colak, 2016). In addition, extracts can affect the color, odor, drying, adhesion, water absorption and acoustic properties of wood (Tanaka *et al.,* 2008). Extracts can be found in various parts of plants such as bark, wood, fruit, flower, leaf, and root (Onal, 2000). Extractives can be obtained from plants through cold water, hot water, and soxhlet extraction and with the aid of different solvents such as hexane, dichloromethane, toluene/ethanol mixture or water (Toshiaki, 2001).

Wood materials need to be colored in different tones other than their natural color, both in line with user requests and in order to ensure a homogeneous color harmony (Sonmez, 2005). To meet this need, natural and environmentally friendly extractives have the potential to be an important alternative (Onal, 2000). Synthetic-based dyes can cause some serious health problems such as allergic reactions and carcinogenicity due to their toxicity and non-biodegradable nature (Prabhu and Bhute, 2012). The use of natural dyes has further increased substantially over the current years, with a slow but growing revival phase at present, due to people's concern over reducing environmental pollution and hence to avoid chemically more hazardous synthetic dyes and intermediates (Samanta, 2020).

The most important problem encountered in impregnation of wood with plant extracts is the potential of these polyphenols to be washed with water from the wood. Fixation of extracts to wood is an important issue as extracts usually dissolve when exposed to high humidity or in contact with water. Therefore, the extracts are used together with various inorganic substances (boron, copper, aluminum compounds, etc.) (Yamaguchi, 2001, Thevenon *et al.,* 2009; Tondi *et al.,* 2012). Mordants are used to increase the binding of natural extracts to functional groups in wood material and for different color options. Ferrous sulfate, copper sulfate and aluminum sulfate are metal salts and they are the most widely used mordants for natural extracts derived from plants (Yeniocak *et al.,* 2018). Metal mordants contribute to developing wide gamut of hues after complexing with natural coloring compounds. Most of these are toxic in nature, but their presence in trace quantity has been found to be safe for users (Prabhu and Bhute, 2012).

Rhododendrons are perennial plants and belong to the *Ericaceae* family. More than 850 species of *Rhododendron* L. are distributed in the Northern Hemisphere. The genus *Rhododendron* distributed in North Eastern and West Anatolia is represented by 9 species, two being endemic and altogether 12 taxa, 4 hybrids and 1 form (Stevens, 1978; Terzioglu *et al.,* 2001; Kucuk *et al.,* 2018). *R. ponticum* and *R. luteum* among these species are widely spread in Turkey (Kucuk, 2005; Birinci *et al.,* 2020). The flowers of *R. ponticum*, which bloom between March and May, are purplish pink and have plenty of nectar. The flowers of *R. luteum*, which bloom between April and September, are yellow and have plenty of nectar (Stevens, 1978; Ceter and Guney, 2011). *Rhododendron* species contain

Grayanotoxin (Andromedotoxin, Acebotoxin, Acetylandromedol, Rhodotoxin) in diterpene structure as a poisonous compound in leaves, flowers, nectar and pollens (Kucuk, 2005). Honey from the flowers of *Rhododendron* is locally known as ''deli bal, acı bal, tutar bal'' (mad honey). *Rhododendron* is used for therapeutic purposes as a medicinal herb due to its positive effects on stomach-bowel ailments, skin diseases, asthma, pain and colds. *Rhododendron* species is also used as an ornamental plant due to its showy flowers (Kucuk *et al.,* 2018; Akgun, 2019).

The objective of this study is to investigate the wood material protection efficiency and coloring potential of impregnation solutions prepared from *R. luteum* and *R. ponticum* plant extracts at different concentration levels. In addition, the effects of ferrous sulfate ($Fe_2(SO_4)_3{\times}7H_2O$), copper sulfate ($CuSO_2{\times}5H_2O$) and aluminum sulfate ($KAl_2(SO_4)_3{\times}18H_2O$) mordants added to impregnation solutions on selected properties were analyzed.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Wood material
2.1. Drvni materijal

Scotch pine (*Pinus sylvestris* L.) and Eastern beech (*Fagus orientalis* Lipsky) wood, which are widely used in the woodwork industry and decoration applications, were used as wood material. Wood materials, which have a moisture content of approximately 12 %, were selected randomly from a timber company in Düzce, Turkey. Wood specimens were cut in rough sizes from sapwood, in accordance with the study methodology. The air-dry density values of specimens were 534 kg/m^3 for pine and 698 kg/m^3 for beech. The surfaces of the specimens to be used in the color tests were sanded with 80 and 100 number sandpaper, respectively, using a caliber sanding machine. The specimens were cut to the dimensions of 40 mm × 30 mm × 5 mm (L × R × T) for color tests and 40 mm × 15 mm × 5 mm (L × R × T) for fungal decay tests. The test specimens were prepared in a number sufficient to accommodate six repetitions (n = 6) for each variable in the study.

2.2 Preparation of plant solutions
2.2. Priprema biljnih otopina

Flowers of *Rhododendron luteum* and *Rhododendron ponticum* L. plants were used to test the coloring potential of wood and their protection against fungal rot. Both plant flowers were obtained in the season from Gürgentepe district of Ordu province in Turkey and dried in the shade for three months. Then the flowers were ground and powdered. The extraction of both plant flowers was carried out according to the hot water method under the conditions shown in Table 1.

At the end of the process, the extracts were separated from their solid parts by filtering with a filter paper. Pure water was added to the solution in the amount evaporated during boiling and was brought to the initially determined concentration ratios. Mordant substances were added to the solutions in order to ensure the adhesion of dyestuffs to wood samples and to obtain different color tones. Ferrous sulfate ($Fe_2(SO_4)_3{\times}7H_2O$), copper sulfate ($CuSO_2{\times}5H_2O$) and aluminum sulfate ($KAl_2(SO_4)_3{\times}18H_2O$) were used as mordant in the study. Addition rates of mordant materials into the solution by weight were 3 %, 5 % and 5 %, respectively. Each mordant substance was added separately to the plant extracts prepared in different concentrations.

2.3 Impregnation
2.3. Impregnacija

Before impregnation, wood specimens prepared for decay testing were dried at 60 °C for 48 h and weighed. The wood specimens were dried at a low temperature (60 °C) in order to prevent the closure of the cell passages due to sudden drying and to ensure deep impregnation. A cylindrical tank assembly with a vacuum holder was used in the impregnation of the wood specimens. A pre-vacuum equivalent pressure of 760 mm/Hg was applied to the specimens for 30 minutes and then the pressure of 8 bar was applied for 30 min (ASTM D 1413-99). The treated specimens were immediately removed and weighed to determine gross solution uptake. The retentions of wood specimens impregnated with plant solutions were determined using Eq. 1:

$$\text{Retention (kg/m}^3\text{)} = (G \times C) / V \times 10 \qquad (1)$$

Where G is the amount (g) of solution absorbed by the specimens, C is the concentration of the solution, and V is the volume (cm^3) of the wood specimens.

Classical immersion method, which is a more economical method, was used in the impregnation process of the color test specimens. The samples were completely immersed in a container filled with solution under atmospheric pressure and at (20±2) ºC temperature and kept for 2 hours. After the impregnation processes, all specimens remained in a conditioning cabin (RH 65 ± 3 % and 20 ± 2 °C) until they reached a stable weight.

Table 1 Extraction conditions
Tablica 1. Uvjeti ekstrakcije

Plant species *Vrsta biljke*	**Concentration, % (plant/pure water)** *Koncentracija, % (biljka / čista voda)*	**Extraction method** *Metoda ekstrakcije*	**Temperature, °C** *Temperatura,* °C	**Duration, min** *Trajanje,* min
R. luteum or *R. ponticum*	2	Hot water (decoction) *vruća voda (dekocija)*	100	60
	4			
	7			

2.4 Determination of decay resistance
2.4. Određivanje otpornosti na propadanje

Decay test was carried out in the Wood Preservation Laboratory of Düzce University according to TS 5563 EN 113:1996. Impregnated and control specimens were then exposed to the white rot fungus *Trametes versicolor* (L: Fr.) Pilat. Test fungi were grown on a malt extract agar medium, which was autoclaved at 121 ºC for 20 min and poured into petri dishes (15 ml for each petri dish). The petri dishes were incubated at (24±2) ºC and (75±2) % relative humidity until the mycelium covered the whole of the agar surface. The wood specimens were dried at 60 ºC for 48 h, weighed (M_1) to determine the initial weight before fungi exposure and then sterilized at (121±2) ºC for 20 min. Control and treated specimens were placed in the petri dishes, with a feeder strip laid. Following fungal exposure of 12 weeks at 24 ºC and 75 % relative humidity, the wood specimens were cleaned, dried (at 60 ºC for 48 h), and weighed (M_2). The mass losses were calculated using Eq. 2:

$$\textit{Mass loss}\ (\%) = (M_1 - M_2) / M_1 \times 100 \tag{2}$$

2.5 Determination of color changes
2.5. Određivanje promjene boje

Color measurements were performed according to ASTM D2244-16 using the *BYK-Gardner Spectrophotometer* instrument. The three-dimensional *CIEL*a*b** color system was used for measuring the color values of specimens. In this system, L^* (lightness) is positioned on the black-white axis (L^* = 0 for black, L^* = 100 for white), a^* on the red-green axis (red for positive values and green for negative values), and b^* on the yellow-blue axis (yellow for positive values and blue for negative values). L^*, a^* and b^* values of the wood specimens were analyzed separately. In addition, by using the differences (ΔL^*, Δa^* and Δb^*) between the values of L^*, a^* and b^* values before and after the plant extract applications, the total color change (ΔE^*) in the specimens was determined according to Equation (3),

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \tag{3}$$

Where ΔL^* is the difference in lightness ($L^*_{treated} - L^*_{control}$), Δa^* is the difference in a^* value ($a^*_{treated} - a^*_{control}$), Δb^* is the difference in b^* value ($b^*_{treated} - b^*_{control}$). Measurements were taken from two different regions of each specimen and their averages were recorded as a single value.

2.6 Statistical analyses
2.6. Statistička analiza

Analysis of variance (ANOVA) tests were performed to determine the effect of plant solutions on the mass loss of pine and beech specimens exposed to *T. versicolor* at the 0.05 significance level. Significant differences between the average values of the groups were compared using Duncan's test.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Retention
3.1. Retencija

The retention values of pine and beech specimens impregnated with plant solutions at different concentration levels are given in Table 2. Higher retention values were found in *R. luteum* solutions compared to *R. ponticum* solutions for both wood species. In addition, retention values increased due to the increase in concentration of the solutions.

3.2 Decay resistance
3.2. Otpornost na propadanje

The mass loss values of wood specimens treated with *R. luteum* and *R. ponticum* extracts were similar. The effect of plant species on mass loss in both wood species was statistically insignificant (Table 3). Additionally, the mass loss from *T. versicolor* was higher in beech specimens than in pine specimens. Bozkurt *et al.* (1993) stated that white rot fungus has a greater effect on hardwood species compared to softwood species. In ad-

Table 2 Retention values
Tablica 2. Vrijednosti retencije

Concentration, % *Koncentracija, %*	Mordant substance *Fiksator*	Retention / *Retencija,* kg/m³			
		Pine / *Borovina*		**Beech** / *Bukovina*	
		R. luteum	*R. ponticum*	*R. luteum*	*R. ponticum*
2	Non-mordant / *bez fiksatora*	10.5 (1.0)	8.6 (1.0)	11.4 (0.4)	10.1 (0.3)
	Ferrous sulfate / *željezov sulfat*	10.4 (0.6)	10.5 (0.9)	10.2 (2.2)	8.6 (2.4)
	Copper sulfate / *bakrov sulfat*	10.0 (0.7)	9.8 (0.4)	9.9 (0.4)	9.6 (0.4)
	Aluminum sulfate / *aluminijev sulfat*	9.5 (1.2)	8.5 (0.8)	9.1 (0.5)	8.7 (0.4)
4	Non-mordant / *bez fiksatora*	21.3 (2.2)	17.3 (1.3)	22.2 (0.4)	20.5 (0.5)
	Ferrous sulfate / *željezov sulfat*	21.2 (0.9)	20.6 (0.7)	20.4 (0.5)	20.4 (0.7)
	Copper sulfate / *bakrov sulfat*	19.2 (2.1)	19.9 (1.1)	19.9 (0.8)	20.2 (0.5)
	Aluminum sulfate / *aluminijev sulfat*	17.5 (1.8)	18.2 (1.5)	17.8 (1.4)	18.2 (0.8)
7	Non-mordant / *bez fiksatora*	37.5 (1.9)	34.9 (2.7)	38.2 (0.5)	36.9 (1.0)
	Ferrous sulfate / *željezov sulfat*	37.0 (2.5)	34.9 (2.6)	36.4 (0.8)	34.6 (2.2)
	Copper sulfate / *bakrov sulfat*	33.2 (2.7)	34.1 (3.5)	34.9 (0.9)	34.8 (1.3)
	Aluminum sulfate / *aluminijev sulfat*	35.6 (4.4)	32.9 (2.8)	34.8 (0.6)	31.4 (2.3)

Table 3 Duncan's one-way test results for mass losses in pine and beech specimens
Tablica 3. Rezultati Duncanova jednosmjernog testa za gubitak mase uzoraka borovine i bukovine

Factor / *Čimbenik*	**Mass loss** / *Gubitak mase,* %			
	Pine / *Borovina*		**Beech** / *Bukovina*	
	Mean	SG	Mean	SG
Plant species / *Vrsta biljke*				
R. luteum	9.44	a	16.75	a
R. ponticum	9.06	a	16.73	a
Concentration / *Koncentracija*				
2 %	9.64	a	16.96	a
4 %	9.50	a	16.61	a
7 %	8.62	b	16.65	a
Mordant substance /*Fiksator*				
Control (untreated) / *kontrolni uzorci (netretirani)*	21.35	a	39.87	a
Non-mordant / *bez fiksatora*	18.58	b	37.55	b
Ferrous sulfate / *željezov sulfat*	1.27	d	1.29	d
Copper sulfate / *bakrov sulfa*t	2.36	c	2.55	c
Aluminum sulfate / *aluminijev sulfat*	2.71	c	2.42	c

SG: Statistical group (different letters denote a significant difference) / *statistička grupa (različita slova označavaju značajnu razliku)*

dition, the decay resistance varies between wood species and within the same species, depending on the tree age, growing region conditions and seasonal differences. It was also found in different study results that mass loss due to *T. versicolor* (white rot fungus) was higher in beech wood than in pine wood (Goktas *et al.,* 2008; Yalcin, 2012; Ozen *et al.,* 2014; Colak, 2016).

For pine specimens, the highest mass loss regarding concentration level was found at 2 % concentration and the lowest was determined at 7 % concentration. However, the effect of the concentration level on the mass loss of beech specimens was insignificant (Table 3). Mass loss tended to decrease with increasing concentration level in specimens treated with non-mordant extracts. However, the effect of the concentration level on wood specimens treated with all mordant added extracts is not clear (Figure 1).

The highest mass loss regarding mordant substance was found to be in the control (untreated) specimens (21.35 % for pine and 39.87 % for beech), while the lowest was obtained in the specimens treated with ferrous sulfate-added extracts (1.27 % for pine and 1.29 % for beech) (Table 3). Mass losses were slightly reduced in specimens impregnated with non-mordant extracts compared to control (untreated) specimens. However, mass loss was significantly reduced in all specimens impregnated with mordant-added extracts. Mordant substances were found to be the main influencing factor on the mass loss of specimens exposed to *T. versicolor* (Figure 1). The mass loss results of specimens treated with copper sulfate and aluminum sulfate-added extracts were similar and the difference between them was statistically insignificant. The most successful results for both wood species were determined in specimens treated with ferrous sulfate-added extracts. For pine wood, the mass loss in the specimens impregnated with non-mordant, ferrous sulfate, copper sulfate and aluminum sulfate-added extracts decreased

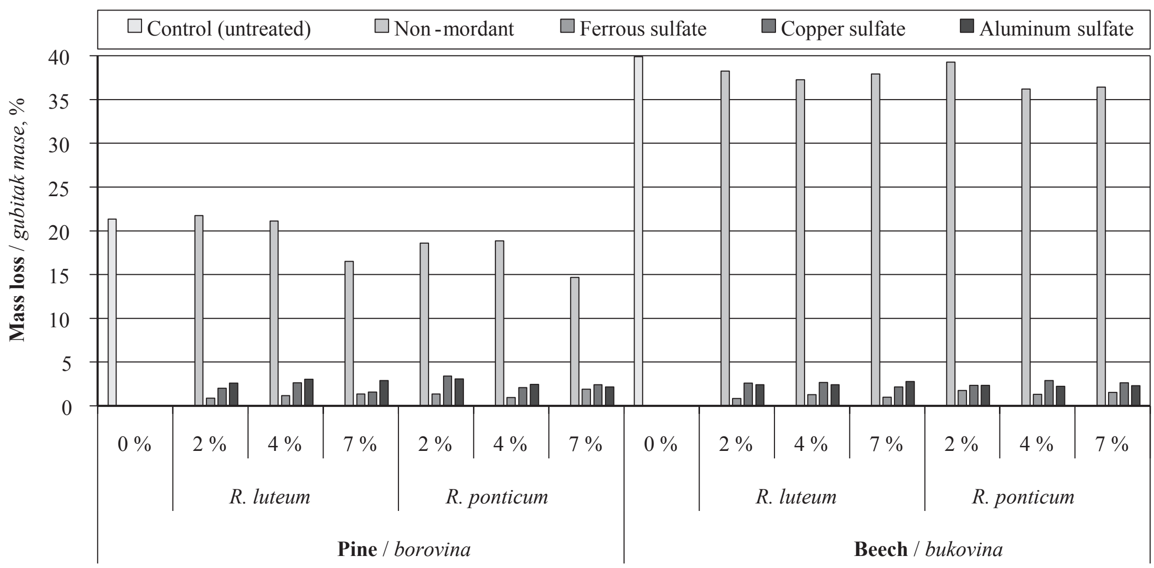

Figure 1 Mass losses for pine and beech wood exposed to *T. versicolor* depending on plant solutions
Slika 1. Gubitci mase uzoraka borovine i bukovine izloženih *T. versicolor* ovisno o biljnim otopinama

13 %, 94 %, 89 % and 87 %, respectively. For beech wood under the same conditions, the mass loss decreased 6 %, 97 %, 94 % and 94 %, respectively. It is known that ferrous, aluminum and especially copper compounds are used in some biocides and these compounds are good fungicides. In a previous study, it was stated that the resistance against brown and white rot fungus in wood materials treated with ferrous sulphate, aluminum sulphate, and copper sulphate mixed madder root extracts was much higher than that of untreated or synthetic dye treated wood materials. In addition, it was reported that mordant agents have the ability to make a complex chemical bond with wood components having -OH groups, which may have an effect on fungal resistance (Ozen *et al.,* 2014). Yamaguchi (2001) determined that chemically modified mimosa tannins and copper are effective against brown and white rot fungi that cause rot in wood material. Shah *et al.,* (2010) stated that the production of lignocellulose degrading enzymes decreased by iron and copper aggregated nanoparticles in the fungus *T. versicolor*. Nano-copper preservatives are able to deliver bioactive components into wood cell walls and thus improve leach resistance as well as bio-durability (Matsunaga *et al.,* 2009). Furthermore, it has been reported by previous studies that treatment with nano-copper significantly improved decay resistance to *T. versicolor* (Kartal *et al.,* 2009; Akhtari and Arefkhani, 2013; Pařil *et al.,* 2017). Moreover, due to the combined effect of natural tannins and metal mordant (copper sulfate) bonding, resistance to microorganisms in cotton and silk fabrics has increased significantly and the rate of fading has been greatly reduced with this eco-friendly process (Prabhu *et al.,* 2011).

3.3 Color change
3.3. Promjena boje

The average *L*, a** and *b** values of pine and beech specimens impregnated with *R. luteum* and *R. ponticum* extracts prepared in different concentrations are given in Table 4. The *L** value of wood specimens impregnated with *R. luteum* extracts was generally higher than that of *R. ponticum*. It can be said that the original colors of the plant flowers used in the study affected the results, because *R. ponticum* flowers have a purplish pink color, while *R. luteum* flowers have a lighter golden yellow color (Stevens, 1978; Kucuk, 2005; Ceter and Guney, 2011).

After treatment with all extracts, the *L** value of pine and beech specimens decreased (Table 4) and the samples darkened to a certain extent (Figure 2 and 3). A decrease in the *L** value indicates a darker color tone, and an increase indicates a lighter color (Sogutlu and Sonmez, 2006). For both wood species, the *L** value was similar for specimens treated with non-mordant, copper sulfate and aluminum sulfate-added extracts. The lowest *L** values were determined in specimens treated with ferrous sulfate-added extracts. The *L** value of these specimens decreased by an average of 27 % in pine and 22 % in beech compared to the control (untreated) specimens. In previous studies using madder root (Goktas *et al.,* 2009), acorn (Peker *et al.,* 2012) and red beetroot (Yeniocak *et al.,* 2015) extracts, the lowest *L** values for pine and beech wood were determined in ferrous sulfate mixed solutions compared to other mordant mixed solutions. Furthermore, wooden materials colored with all metal mordant mixed solutions have darker colors (Yeniocak *et al.,* 2015). Prabhu and Bhute (2012) stated that aluminum sulfate, potassium dichromate and tin (Stannous chloride) falls under the category of brightening mordants, while copper sulphate and ferrous sulphates are dulling mordants. Ferrous sulfate mordant causes significant color differences as well as a color shift towards dark tones, while aluminum sulphate mordant increases the lightness value of the color but slightly affects the color tone (Acar, 2013).

The *L** value of the impregnated specimens tends to decrease due to the increase in the concentration level of the solutions (Table 4). It can be said that with the increase in concentration, the increase in the ratio of extractives in darker hues compared to the natural color of wood materials has an effect on the results.

The highest *a** values for both wood species were determined in specimens (except pine specimens treated with ferrous sulfate-added extracts) impregnated with *R. ponticum* solutions. In a previous study, an *Anthocyanidin* named *Peonidin* causing purple-red coloration in the flowers of the *R. ponticum* plant was determined (Gündüz, 1996). The increase in the concentration level of the solutions caused a general increase in the *a** value of pine specimens. This situation is more pronounced in specimens treated with *R. ponticum* solutions. However, *a** values decreased because of increasing concentration in pine specimens treated with ferrous sulfate-added both plant solutions. On the other hand, the effect of the concentration level was not evident for beech specimens (Table 4). After treatment with extracts, the *a** value of pine and beech specimens differed depending on the mordant substance. For pine wood, in specimens treated with non-mordant and aluminum sulfate-added extracts, the *a** value increased by an average of 55 % and 61 %, respectively, and consequently the red color tendency of the specimens also increased. On the other hand, in specimens treated with ferrous sulfate and copper sulfate-added extracts, the *a** value decreased by an average of 55 % and 56 %, respectively, and consequently the green color tendency of the specimens increased (Table 4 and Figure 2). For beech wood, the *a** value of specimens treated with non-mordant extracts gave similar results to control (untreated) specimens. The *a** value increased by an average of 8 % in specimens treated with aluminum sulfate-added extracts. It was reduced by 18 % in specimens treated with copper sulfate-added extracts. However, the most significant change in *a** value of beech specimens was observed after treatment with ferrous sulfate-added extracts. In these specimens, the *a** value decreased by 76 % compared to the control (untreated) specimens and the green color tendency increased significantly (Table 4 and Figure 3). In the literature, it has been stated that ferrous sulphate mordant

Table 4 Color values in pine and beech wood impregnated with plant solutions
Tablica 4. Vrijednosti boje uzoraka borovine i bukovine impregniranih biljnim otopinama

Plant species *Vrsta biljke*	**Concentration, %** *Koncentracija, %*	**Mordant substance** *Fiksator*	**Color parameters** / *Parametri boje*					
			Pine / *Borovina*			**Beech** / *Bukovina*		
			*L**	*a**	*b**	*L**	*a**	*b**
R. luteum	2	Non-mordant *bez fiksatora*	76.6 (0.1)	7.2 (0.2)	29.8 (0.6)	65.8 (0.8)	11.1 (0.3)	23.6 (0.5)
		Ferrous sulfate *željezov sulfat*	63.4 (0.7)	5.0 (0.6)	24.5 (1.0)	54.2 (2.0)	2.6 (0.3)	14.1 (0.7)
		Copper sulfate *bakrov sulfat*	73.9 (1.3)	0.5 (0.2)	26.9 (0.4)	65.0 (1.2)	9.4 (0.3)	23.2 (0.6)
		Aluminum sulfate *aluminijev sulfat*	77.1 (0.9)	7.9 (0.3)	32.4 (0.8)	66.0 (0.3)	12.3 (0.4)	25.5 (0.4)
	4	Non-mordant *bez fiksatora*	75.9 (1.2)	7.3 (0.4)	29.5 (0.6)	65.1 (1.1)	11.0 (0.5)	23.0 (0.6)
		Ferrous sulfate *željezov sulfat*	62.1 (1.1)	3.8 (0.9)	22.9 (0.8)	53.5 (1.4)	2.7 (0.3)	14.2 (0.8)
		Copper sulfate *bakrov sulfat*	74.0 (1.4)	2.0 (0.4)	29.6 (0.5)	63.9 (0.4)	9.4 (0.4)	24.6 (0.9)
		Aluminum sulfate *aluminijev sulfat*	75.9 (1.4)	8.1 (0.5)	33.1 (0.7)	64.7 (0.5)	12.0 (0.6)	26.0 (0.5)
	7	Non-mordant *bez fiksatora*	71.2 (1.1)	9.5 (0.6)	31.4 (0.8)	64.4 (1.2)	11.5 (0.3)	24.2 (0.5)
		Ferrous sulfate *željezov sulfat*	58.1 (1.7)	2.6 (0.7)	20.3 (1.6)	51.8 (0.7)	2.4 (0.3)	14.7 (0.5)
		Copper sulfate *bakrov sulfat*	73.5 (0.7)	1.7 (1.1)	31.0 (1.0)	64.1 (0.7)	9.7 (0.3)	25.4 (0.9)
		Aluminum sulfate *aluminijev sulfat*	75.3 (1.0)	7.5 (0.5)	35.0 (0.5)	66.0 (0.7)	11.5 (0.4)	26.9 (0.4)
R. ponticum	2	Non-mordant *bez fiksatora*	67.8 (1.1)	8.9 (0.6)	27.9 (1.3)	63.3 (0.5)	12.1 (0.7)	24.0 (0.2)
		Ferrous sulfate *željezov sulfat*	62.1 (1.2)	2.6 (0.3)	19.8 (0.6)	55.7 (1.5)	3.5 (0.3)	14.6 (0.6)
		Copper sulfate *bakrov sulfat*	73.9 (1.3)	1.7 (0.9)	33.2 (1.7)	63.6 (0.8)	9.7 (0.5)	27.2 (1.0)
		Aluminum sulfate *aluminijev sulfat*	74.5 (1.3)	9.0 (0.8)	30.7 (0.9)	65.4 (0.4)	12.7 (0.2)	25.2 (0.3)
	4	Non-mordant *bez fiksatora*	66.1 (1.8)	9.0 (0.6)	27.0 (1.3)	61.7 (0.7)	11.7 (0.5)	23.7 (0.9)
		Ferrous sulfate *željezov sulfat*	58.6 (1.1)	1.1 (0.4)	17.7 (0.6)	54.7 (1.2)	3.0 (0.3)	14.7 (0.3)
		Copper sulfate *bakrov sulfat*	70.4 (1.2)	3.4 (0.8)	38.7 (0.5)	63.1 (0.6)	9.7 (0.6)	29.6 (0.6)
		Aluminum sulfate *aluminijev sulfat*	72.4 (1.1)	10.6 (0.5)	32.9 (0.3)	62.6 (0.8)	13.5 (0.3)	26.5 (0.7)
	7	Non-mordant *bez fiksatora*	65.9 (1.7)	10.7 (1.2)	30.3 (1.5)	61.2 (1.0)	12.6 (0.3)	25.3 (0.6)
		Ferrous sulfate *željezov sulfat*	55.2 (1.7)	0.3 (0.4)	15.8 (1.0)	53.3 (1.0)	2.8 (0.2)	14.1 (0.5)
		Copper sulfate *bakrov sulfat*	67.8 (1.1)	5.7 (0.8)	40.9 (1.1)	61.8 (0.9)	9.7 (0.5)	32.1 (0.9)
		Aluminum sulfate *aluminijev sulfat*	68.9 (1.3)	11.4 (1.0)	33.6 (0.9)	61.8 (0.6)	13.7 (0.3)	27.8 (0.6)
Control / *Kontrolni uzorci*			82.0 (0.1)	5.6 (0.1)	26.3 (0.1)	69.5 (0.1)	11.7 (0.1)	21.5 (0.1)

Values in parentheses are standard deviations. / *Vrijednosti u zagradama standardne su devijacije.*

is used to obtain dark colors and black colors with all dyestuffs, while copper sulphate mordant gives brown-green color with many dyestuffs and gives uninteresting dark colors with some dyestuffs and is used to obtain green colors as mordant (Anonymous, 1991).

The *b** value of pine and beech specimens increased after treatment with all other solutions except ferrous sulfate-added solutions. For pine wood, the *b** value was higher in *R. luteum* compared to *R. ponticum* for non-mordant and ferrous sulfate-added solutions. However, higher *b** values were obtained in *R. ponticum* compared with *R. luteum* for copper sulfate and aluminum sulfate-added solutions. For beech wood, the *b** value of specimens treated with *R. luteum* and *R.*

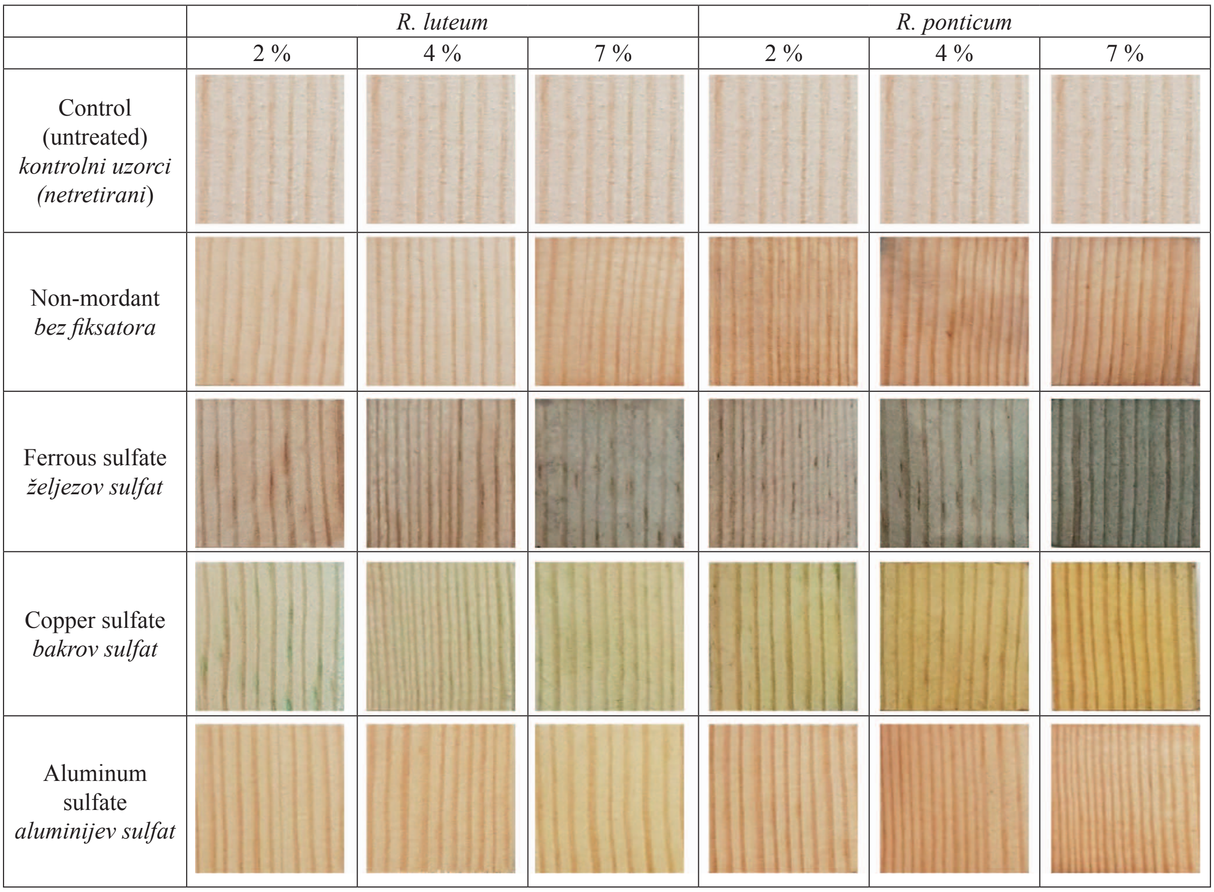

Figure 2 Color change images in pine wood after impregnation with plant solutions
Slika 2. Promjena boje borovine nakon impregnacije biljnom otopinom

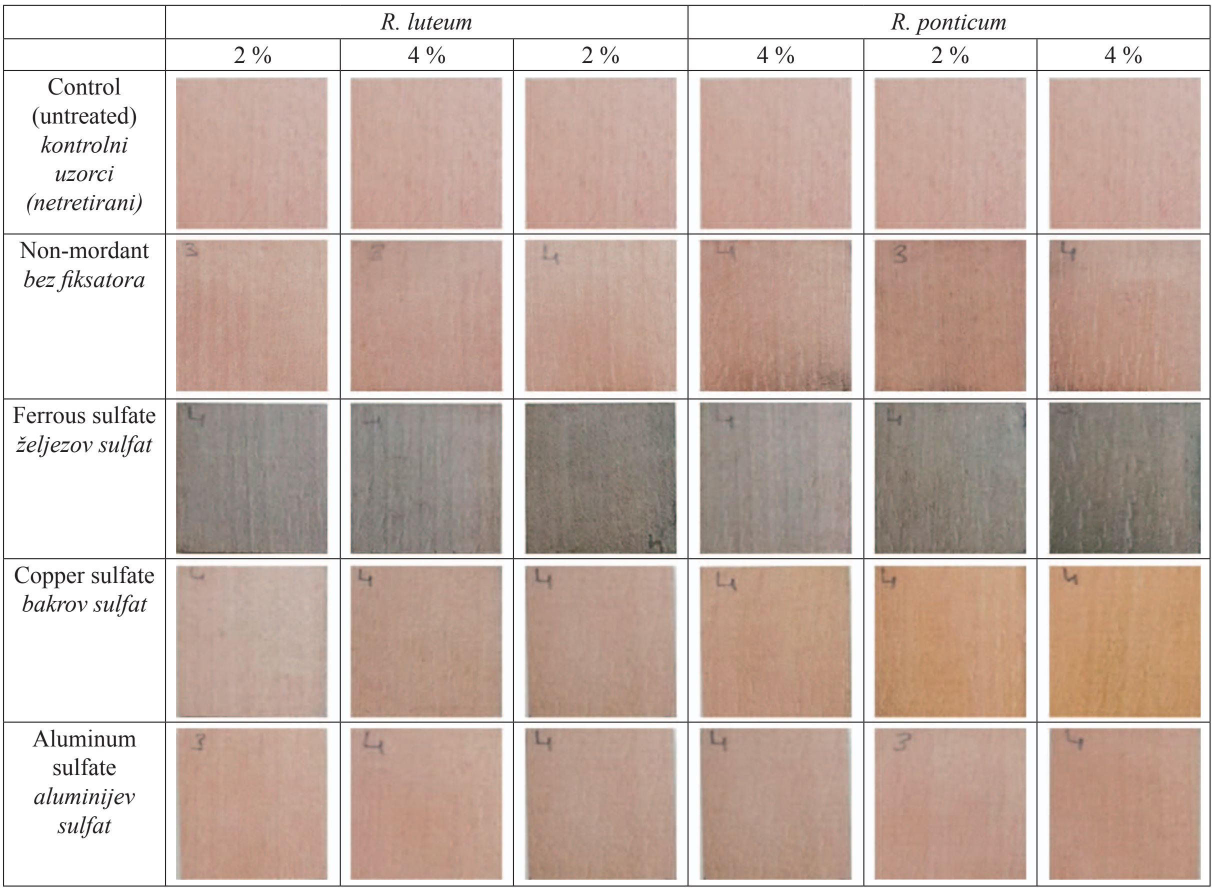

Figure 3 Color change images in beech wood after impregnation with plant solutions
Slika 3. Promjena boje bukovine nakon impregnacije biljnom otopinom

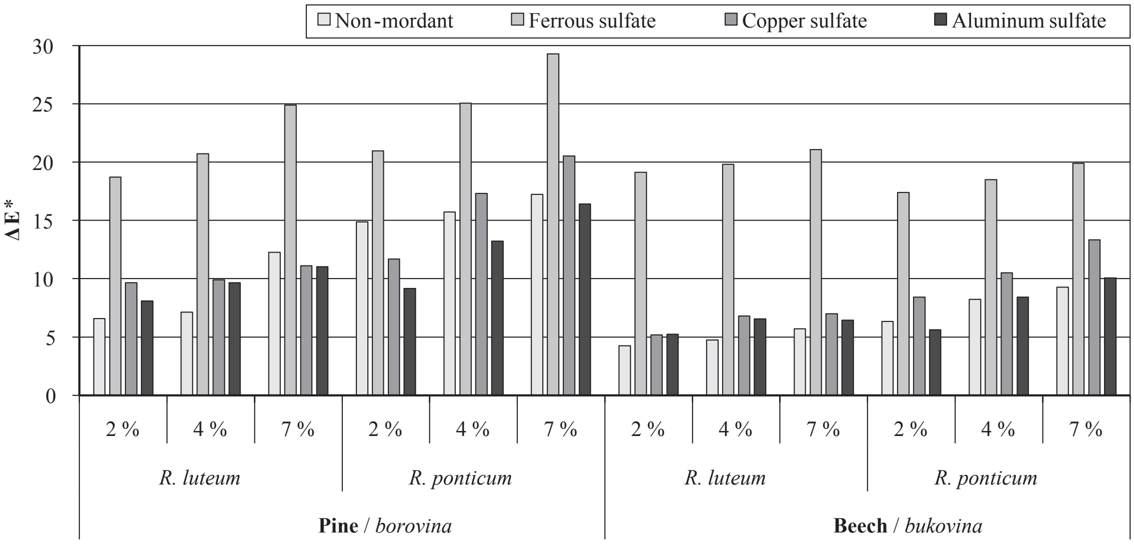

Figure 4 Total color change (*ΔE**) for impregnated pine and beech wood according to control (untreated) samples
Slika 4. Ukupna promjena boje (*ΔE**) impregniranih uzoraka borovine i bukovine u usporedbi s kontrolnim (netretiranim) uzorcima

ponticum solutions (except for copper sulfate-added solutions) was similar. Higher b^* values were determined in *R. ponticum* for copper sulfate-added solutions. The b^* value of pine and beech specimens (except specimens treated with ferrous sulfate-added extracts) increased due to the increase in the concentration ratio of the solutions. For pine specimens treated with ferrous sulfate-added extracts, the b^* value decreased with increasing concentration ratio, while for beech specimens the effect of concentration ratio was insignificant (Table 4). The b^* value of pine specimens treated with non-mordant, copper sulfate and aluminum sulfate-added extracts increased by 3 %, 17 % and 16 %, respectively, compared to the control (untreated) specimens. Whereas, the b^* value of beech specimens increased by 6 %, 18 % and 15 %, respectively. Especially after treatment with copper sulfate and aluminum sulfate-added extracts, the yellow color tendency of the specimens increased. On the other hand, the b^* value of pine and beech specimens treated with ferrous sulfate-added extracts decreased on average by 25 % and 35 %, respectively, compared to the control specimens. In these specimens, the blue color tendency was increased (Figure 2 and 3).

After treatment with *R. luteum* and *R. ponticum* extracts, the total color change (ΔE^*) is higher in pine wood than in beech wood. It can be said that the natural color of pine wood, being more light than beech wood, has an effect on the results. Yeniocak *et al.,* (2017) stated that different interactions of natural extracts and mordant-added extracts with wood species and differences in the chemical composition of wood species may be effective in color change. On the other hand, genetic, environmental and technical factors such as anatomical structure, extractive substance content, heat, light, moisture, alkalis, acids, steaming, drying and finishing affect the color change of wood (Temiz *et al*., 2005; Peker *et al.,* 2012; Bekhta *et al.,* 2014; Pelit, 2017). Regarding the plant species, the ΔE^* was higher in specimens treated with *R. ponticum* solutions compared to *R. luteum*. However, as an exception, the ΔE^* was found to be higher in beech specimens treated with ferrous sulfate-added *R. luteum* solutions. Due to the increase in the concentration level of the solutions, the ΔE^* value increased in all impregnated wood specimens (Figure 4). The ΔE^* value in pine specimens treated with *R. luteum* and *R. ponticum* solutions prepared at 7 % concentration increased by 38 % and 47 %, respectively, compared to 2 % concentration. For beech specimens under the same conditions, the ΔE^* increased by 19 % and 39 %, respectively. Regarding the mordant substance, the highest ΔE^* values for all solutions were determined in specimens impregnated with ferrous sulfate-added extracts. Compared to the specimens impregnated with other mordant added extracts, the ΔE^* value was significantly higher in these specimens. The decrease in a^* values of these specimens considerably affected the results. Although the ΔE^* values of the specimens treated with non-mordant, copper sulfate and aluminum sulfate-added extracts differ depending on the plant species and concentration level, the ΔE^* values in these specimens were generally found to be close to each other (Figure 4).

4 CONCLUSIONS
4. ZAKLJUČAK

Mass losses in wood specimens exposed to white rot fungus after treatment with *R. luteum* and *R. ponticum* plant extracts were similar, and the effect of plant species on mass losses was insignificant. The mass loss of the specimens impregnated with non-mordant plant extracts was slightly reduced compared to the control (untreated) specimens. However, mass losses were significantly reduced in pine and beech specimens impregnated with mordant added extracts. While similar results were obtained for specimens treated with copper sulfate and aluminum sulfate-added extracts, the most successful results were determined in specimens treated with ferrous sulfate-added extracts. After impregnation, the mass loss of pine and beech specimens decreased up to 94 % and 97 %, respectively. In addition, mass losses tend to decrease with increasing concentration level in non-mordant extracts. However, the effect of the concentration level was not pronounced in mordant-added extracts. Mordant substances were the main factor influencing the mass loss of wood specimens.

The *L**, *a** and *b** color values of the impregnated pine and beech specimens varied depending on the plant species, concentration level and mordant substances. After treatment with plant extracts, the *L** value of all specimens (especially those treated with ferrous sulfate-added extracts) decreased and the specimens darkened. In wood specimens (especially pine) treated with non-mordant and aluminum sulfate-added extracts, the *a** and *b** values increased due to the increase in the concentration level and the red-yellow color tendency of these specimens also increased. In wood specimens treated with copper sulfate-added extracts, the *a** value decreased and the *b** value increased. Depending on this situation, the green-yellow color tendency of these specimens increased. In specimens treated with ferrous sulfate-added extracts, both *a** and *b** values decreased due to the increase in the concentration level and the green-blue color tendency of these specimens increased. Total color change (ΔE^*) is higher in pine as wood type, *R. ponticum* as plant species, 7 % as concentration level and ferrous sulfate as mordant substance.

As a result, *R. luteum* and *R. ponticum* extracts did not have sufficient resistance to *T. versicolor* fungus. Therefore, it can be recommended to use natural extracts in combination with mordant substances to significantly improve the decay resistance and increase the color options.

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Biodegradation and Micro-Scale Treatability Pattern of Loblolly Pine Heartwood Bioincised by *Bacillus Subtilis* and *Physisporinus Vitreus*

Biorazgradnja i model impregnacije srži lobodijskog bora na mikrorazini, uz poboljšanje prodora zaštitnog sredstva primjenom *Bacillus subtilis* i *Physisporinus vitreus*

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ABSTRACT • *One strategy for improving the treatability of refractory wood species is biological incising, and its efficiency depends on how the microorganisms modify the porous structure of the wood. Evaluation of the bioincised wood treatability on a micro-scale can thus help to better understand the treatability enhancing mechanisms. In the present study, the biodegradation pattern and micro-scale treatability of Loblolly pine (Pinus taeda L.) heartwood were determined after bioincising with the white-rot fungus Physisporinus vitreus (Pers.: Fr.) P. Karsten isolate 136 and bacterium Bacillus subtilis UTB22. Oven-dried specimens with dimensions of 50 mm × 25 mm × 15 mm (L × T × R) were incubated with the microorganisms at (23±2) °C and (65±5) % relative humidity for six weeks. The control and exposed wood blocks were then pressure treated by 1 % fluorescent dye (fluorescein)-containing water to study the treatability pattern under a fluorescence microscope. The longitudinal and tangential air permeability and compression strength parallel to the grain of the specimens were also determined at the end of the incubation period. Scanning electron microscopic (SEM) studies showed that degradation by B. subtilis UTB22 was limited to the pit membranes, but the cell walls were also degraded to some extent by P. vitreus. The fungus caused a higher mass loss compared to the bacterium, whereas the permeability enhancing ability of the bacterium was more pronounced. The fluorescent dye tracer also showed that higher treatability with more uniformity was obtained by B. subtilis UTB22. The improvement in treatability by both microorganisms was mainly due to the degradation of the earlywood tracheids.*

Keywords: *Bacillus subtilis; bioincising; penetration; Physisporinus vitreus; wood treatability*

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SAŽETAK • *Jedna od metoda poboljšanog impregniranja slabo poroznih vrsta drva jest tretiranje drva mikroorganizmima, a cilj tog postupka jest promjena porozne strukture drva. Evaluacija tretmana drva mikroorganizmima može pridonijeti boljem razumijevanju mehanizama poboljšanja prodora zaštitnog sredstva u drvo. U ovom je istraživanju promatrana biorazgradnja i impregnacija srži lobodijskog bora (Pinus taeda L.) na mikrorazini nakon izloženosti drva gljivi bijele truleži Physisporinus vitreus (Pers.: Fr.) P. Karsten izolat 136 i bakteriji Bacillus subtilis UTB22. Apsolutno suhi uzorci drva dimenzija 50 mm × 25 mm × 15 mm (L × T × R) zaraženi su mikroorganizmima pri 23±2 °C i 65±5 % relativne vlažnosti zraka tijekom šest tjedana. Nakon toga kontrolni i zaraženi blokovi drva tlačnim su postupkom impregnirani fluorescentnim bojilom otopljenim u vodi (1 %-tni fluorescein) kako bi se pod fluorescentnim mikroskopom promotrio učinak tretmana drva mikroorganizmima. Na kraju inkubacije utvrđena je propusnost zraka u uzdužnom i tangentnom smjeru te izmjerena čvrstoća na tlak paralelno s vlakancima. Istraživanje pretražnim elektronskim mikroskopom (SEM) pokazalo je da je razgradnja bakterijom B. subtilis UTB22 ograničena na membrane jažica, dok su stanične stijenke u određenoj mjeri razgrađene djelovanjem gljive P. vitreus. Gljiva je uzrokovala opsežniji gubitak mase nego bakterija, dok je sposobnost bakterije da poveća propusnost bila veća. Fluorescentno bojilo također je pokazalo da je bakterijom B. subtilis UTB22 postignuta bolja propusnost, s većom ujednačenošću. Poboljšanje propusnosti primjenom obaju mikroorganizama uglavnom je posljedica razgradnje traheida ranog drva.*

Ključne riječi: *Bacillus subtilis; tretiranje drva mikroorganizmima; prodor Physisporinus vitreus; impregniranje drva*

1 INTRODUCTION
1. UVOD

Most wood species are not sufficiently durable against wood-destroying organisms and have to be treated with chemical preservatives to ensure the desired service life or to improve other relevant properties like resistance to fire and UV stability. Preservatives Adequate penetration of the wood by the preservatives during the treatment process is necessary to achieve a successful protection against biological degradation. However, the penetration depth of active ingredients alone is not sufficient to judge the wood treatability; the uniformity of the penetration should be evaluated, e.g., by microscopic studies of the treated wood (Tarmian *et al.,* 2020). Wood decay can occur from the non-uniform impregnation of the wood cells (Omidvar and Schneider, 2004). This type of penetration is sometimes called marble penetration.

Various strategies can be used to enhance the penetration of the preservatives into refractory wood species, among which mechanical incising is commonly used in industry for this purpose (Hansmann *et al.,* 2002; Morrell and Morris, 2002; Lehringer *et al.,* 2009 a, b; Evans 2016; Tarmian *et al.,* 2020). Biological incising (i.e. bioincising) using a variety of fungi and bacteria can also be considered as a wood treatability enhancing method which might have the potential for industrial upscaling (Johnson, 1979; Daniel, 2003; Mai *et al.,* 2004; Schwarze *et al.,* 2006; Pánek and Reinpercht, 2008, 2011; Lehringer *et al.,* 2011; Fuhr *et al.,* 2011; Thaler *et al.,* 2012; Yildiz *et al.,* 2012; Dale *et al.,* 2019). For example, it has been known for a long time that some bacteria (e.g., *Bacillus subtilis*) are able to increase the permeability of some refractory coniferous wood species during ponding or water sprinkling by deteriorating the bordered pit membranes (Pánek and Reinprecht, 2008; Yildiz *et al.,* 2012).

The white-rot fungus *Physisporinus vitreus* has been used as a bioincising fungus to enhance the permeability of some refractory softwood species, like Norway spruce (*Picea abies*), without any considerable reduction in the mechanical strength of the wood (Schmidt *et al.,* 1997; Schwarze and Landmesser, 2000; Schwarze *et al.,* 2006, 2008; Lehringer *et al.,* 2010, 2011; Schwarze and Schubert, 2011; Emaminasab *et al.,* 2015, 2016). It is claimed that *P. vitreus* is able to selectively degrade pectin-rich pit membranes under certain incubation conditions (Schmidt *et al.,* 1997; Schwarze and Landmesser, 2000; Lehringer *et al.,* 2010; Fuhr *et al.,* 2011).

Some works have evaluated the treatability of wood after bioincising with *Bacillus sp.* (Pánek and Reinprecht, 2011; Yildiz *et al.,* 2012) and *P. vitreus* (Schwarze *et al.,* 2006; Volkmer *et al.,* 2010; Lehringer *et al.,* 2011; Emaminasab *et al.,* 2015, 2016) with macro-scale analyses. The current study gives some detailed information on the micro-scale treatability of bioincised wood which could be helpful in achieving an in-depth understanding of the treatability-enhancing mechanisms.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Wood sample preparation
2.1. Priprema uzoraka drva

Wood specimens with dimensions of 50 mm × 25 mm × 15 mm (L × T × R) were prepared from the heartwood of air-dried Loblolly pine (*Pinus taeda* L.) boards according to EN 113:2004 for fungal and bacterial incubation. Loblolly pine is native to the southeastern United States and widely grown on plantations as well. Wood samples from the trees planted in northern Iran were used for this study. The treatability class of Loblolly pine heartwood varies from III to IV, indicating that it is difficult or extremely difficult to treat (EN 350: 2016). Compression strength parallel to the grain of the specimens with dimensions of 60 mm × 20 mm × 20 mm (L × T × R) was also determined according to DIN 52185:1976. Five replicates were used for each test.

2.2 Incubation by *B. subtilis* and *P. vitreus*
2.2. Inkubacija pri izloženosti drva *B. subtilis* i *P. vitreus*

The wood samples were oven dried at (103±2) °C for 24 h, steam-sterilized at 121 °C and 124.1 kPa for 20 min, and weighed before fungal and bacterial exposure. *Physisporinus vitreus* (Pers.: Fr.) P. Karsten isolate 136 was obtained from the University of Hamburg (Bari *et al*., 2015). The woodblocks were exposed to fungal mycelia grown in Kolle flasks containing 4.8 % malt extract agar (Merck, Germany).

Bacillus subtilis UTB22 was obtained from the Department of Plant Protection at the University of Tehran. One ml of *B. subtilis* UTB22 was transferred to sterilized Luria Bertani aqua culture medium and put in a shaker incubator for 120 min^{-1} at 28 °C for 24 h. The bacterium was harvested by centrifugation for 6 minutes at 8000 min^{-1}. A suspension of bacterium and distilled water was supplied, and the optimum population of bacteria was determined using a spectrophotometer. Incubation by both microorganisms was carried out at a temperature of (23±2) ºC and (65±5) % relative humidity for 6 weeks. At the end of incubation, the samples were oven dried and weighed to calculate the mass loss. A Scanning Electron Microscope (SEM; Zeiss DSM 960 A, Germany) was used to observe the biodegradation pattern of the incubated specimens.

2.3 Micro-scale treatability test
2.3. Ispitivanje impregnacije na mikrorazini

The control and incubated wood specimens with a moisture content of 12 % were pressure treated with 1 % fluorescent dye (fluorescein)-containing water. Before the impregnation process, the end sections of the specimens were completely coated with epoxy resin to limit solution penetration through the transverse direction. An initial vacuum of 30 kPa was first applied for 20 minutes, and then impregnation was carried out at atmospheric pressure for 30 minutes. Transverse sections of about 10-15 μm thickness were cut from the treated specimens using a sledge microtome (GSL1) and studied by fluorescence microscopy (BEL FLUO-3, Italy). Under the UV light of the microscope (exciting spectrum area: 330-400nm), the treated area gave off a green color.

2.4 Measuring air permeability
2.4. Mjerenje propusnosti zraka

There is a strong correlation between wood treatability and air permeability (Siau, 1984). Thus, air permeability of the specimens was also measured. For this purpose, cylindrical samples 18 mm in diameter and 20 mm in length were cut along the longitudinal and tangential directions of the wood specimens with ten replicates for each treatment. The lateral surfaces of the specimens conditioned to equilibrium moisture content (*EMC*) of about 12 % were first coated by epoxy resin to avoid lateral air leakage. Superficial gas permeability) was then determined under steady-state conditions based on Darcy's law using a well-known falling-water displacement method suggested by Siau (1984) (see Figure 1 for details of the apparatus):

$$k_g = \frac{V_d \cdot C \cdot L \cdot \left(P_{atm} - 0.074 \cdot \bar{h}\right)}{t \cdot A \cdot \left(0.074 \cdot \bar{h}\right) \cdot \left(P_{atm} - 0.037 \cdot \bar{h}\right)} \cdot \frac{0.760\,\text{mHg}}{1.013 \cdot 10^5\,\text{Pa}} \quad (1)$$

Where $V_d = \pi \cdot r^2 \cdot \Delta z$ (m^3), P_{atm} is the atmospheric pressure (mHg), L is the sample length (m), $\bar{h}$ is the average height of water over surface of reservoir during period of measurement (m), A is the cross-sectional area of specimen (m^2), t is the time (s) required for the water drop through Δh and C *is* the correction factor for gas expansion:

$$C = 1 + \frac{V_r \cdot (0.074 \cdot \Delta h)}{V_d \cdot \left(P_{atm} - 0.074 \cdot \bar{h}\right)} \quad (2)$$

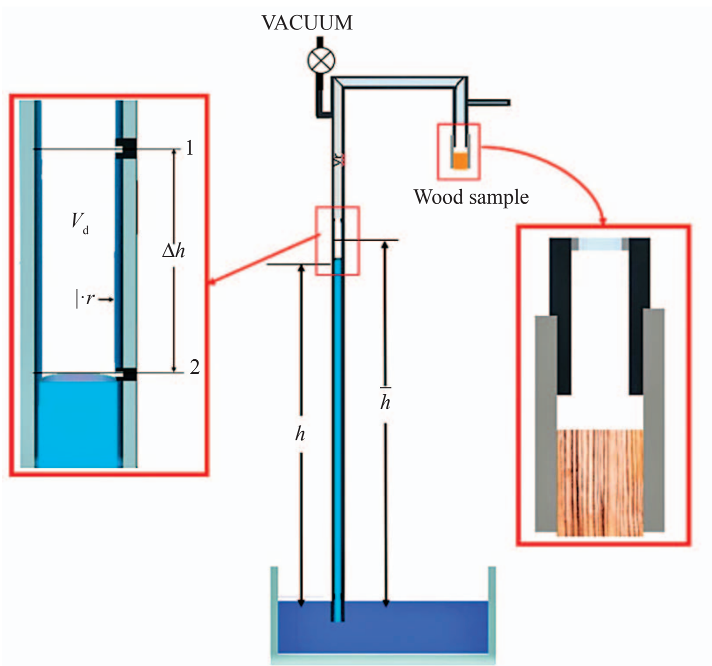

Figure 1 Schematic view of apparatus used to measure air permeability based on falling-water displacement method
Slika 1. Shematski prikaz opreme za mjerenje propusnosti zraka koja se temelji na metodi istiskivanja vode

Where V_r is total volume of apparatus above point 1 including volume of hoses (m^3), and Δh is change in the water drop level during the test (m). Finally, the specific permeability coefficient (K) (m^3 m^{-1}), which is only a function of the porous medium properties, was calculated by:

$$K = k_g \cdot \mu \quad (3)$$

Where μ is the viscosity of air at 20 °C (μ=1.81·10^{-5} Pa·s).

2.5 Statistical analysis
2.5. Statistička analiza

Statistical analysis was conducted using SPSS software (Version 20) by one-way analysis of variance (ANOVA). Duncan multiple range test was used to test the statistical significance at the α = 0.05 level.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Mass loss and compression strength parallel to the grain
3.1. Gubitak mase i čvrstoća na tlak paralelno s vlakancima

P. vitreus caused higher dry mass loss than *B. subtilis* UTB22. The average mass losses were less than 1 % with *B. subtilis* UTB22 and 8.4 % with *P. vitreus* (Table 1). Low mass loss after exposure to *B. subtilis* UTB22 might not be the sole consequence of the bacterial degradation; the leaching of extractives was also observed. Some previous works have reported smaller mass losses after colonization of the wood with the same isolate of *P. vitreus* for six weeks; for example, 4.7 % for black poplar (*Populus nigra*) (Emaminasab *et al.,* 2015), 2.1 % for the heartwood of Norway spruce (Lehringer *et al.,* 2010), and 3.4 % for the sapwood of Norway spruce and Douglas fir (*Pseudotsuga menziesii*) (Schwarze and Landmesser, 2000). Such a variation in the mass loss definitely implies that the fungal activity changes largely with the wood species, the wood block size, and incubation conditions. The compression strength parallel to the grain of the wood specimens was reduced by 16 % and 7 % after incubation with *B. subtilis* UTB22 *and P. vitreus*, respectively (Table 1). Other authors have also reported that a short period of wood incubation by *P. vitreus* caused a slight reduction in mechanical strength (Schwarze and Landmesser, 2000; Schwarze *et al.,* 2006; Lehringer *et al.,* 2010). However, the strength loss after incubation with *P. vitreus* can be significant for some wood species (Schwarze *et al.,* 2006; Emaminasab *et al.,* 2015). Schwarze *et al.* (2006) reported that the impact bending strength of *Abies alba* was reduced by about 31.5 % after incubation of 15 cm^3 test blocks with EMPA 642 isolate of *P. vitreus* for 6 weeks. The unexpected higher loss of strength in bacterial-treated samples, despite their insignificant mass loss, can be attributed to the larger number of tracheids engaged in the decomposition of their pit membrane. This affects the continuity of the tracheid wall and results in a devaluation of some mechanical properties (Bucur, 1995).

3.2 Wood degradation
3.2. Razgradnja drva

Both microorganisms degraded the tracheid bordered pits and cross-field pits, which resulted in an opening of the aspirated pits (Figures 2 and 3). Pit degradation by *P. vitreus* was more severe than that by *B. subtilis* UTB22. The extent of the fungal degradation was not similar for all pits (Figures 3 a, b, and e). The degradation for some pits was limited to the tori (Figure 3b, white arrow), but the membranes of some were completely degraded (Figure 3b, black arrow). Schwarze *et al.* (2006) also showed a similar pit degradation pattern after incubation of *P. abies* and *A. alba* with *P. vitreus* isolates of EMPA 642 and EMPA 643. In contrast to Schwarze and Landmesser (2000), Schwarze *et al.* (2006), and Schwarze *et al.* (2008) who reported a preferential degradation of pit membranes of Norway spruce tracheids at the initial stage of wood colonization with *P. vitreus*, SEM observations in the current study showed some signs of cell wall degradation (Figure 3a, white arrows). Lehringer *et al.* (2010) also found that some bore holes were formed in the tracheid cell walls even at early stages of *P. vitreus* colonization in Norway spruce. Therefore, in agreement with the report of Lehringer *et al.* (2010), a selective degradation of pit membranes with *P. vitreus* as claimed by Schwarze and Landmesser (2000) and Schwarze *et al.* (2006, 2008) is questionable, even at early stages of wood colonization. These contradictory results, however, may be due to different incubation conditions used by the authors, which implies that fungal activity is highly dependent upon the bioincising conditions. The degradation of pit membranes can be more selective by optimizing the incubation parameters (Lehringer, 2011). Degradation by *B. subtilis* UTB22 was confined to the pit tori (Figures 2c-d). A non-uniform

Table 1 Average mass loss, compression strength parallel to the grain, and air permeability of Loblolly pine heartwood blocks after incubation with *Bacillus subtilis* UTB22 and *Physisporinus vitreus* (Pers.: Fr.) P. Karsten isolate 136 for six weeks
Tablica 1. Prosječni gubitak mase, čvrstoća na tlak paralelno s vlakancima i propusnost zraka kroz srž lobodijskog bora nakon šest tjedana inkubacije s *Bacillus subtilis* UTB22 i *Physisporinus vitreus* (Pers.: Fr.) P. Karsten izolat 136

	Mass loss, % *Gubitak mase,* %	**Compression strength parallel to the grain, MPa** *Čvrstoća na tlak paralelno s vlakancima,* MPa	**Air permeability, $m^2 \cdot 10^{-15}$** *Propusnost zraka,* $m^2 \cdot 10^{-15}$	
			Longitudinal *Uzdužno*	Tangential *Tangentno*
Control / *Kontrolni uzorci*	-	32.17 ± 1.2^c	6.70 ± 0.6^a	2.8 ± 0.3^a
Bacillus subtilis	0.63 ± 0.4^a	26.92 ± 2.1^a	52.00 ± 10.8^c	16.4 ± 2.3^c
Physisporinus vitreus	8.42 ± 1.9^b	30.09 ± 2.3^b	15.00 ± 1.6^b	6.4 ± 0.7c^b

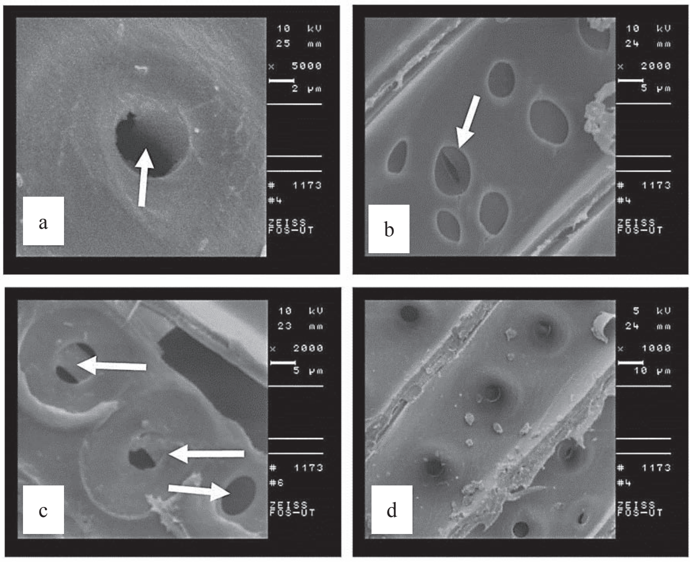

Figure 2 SEM images of wood specimens incubated by *B. subtilis* UTB22: a) partial degradation of tracheid bordered pit torus; b) an opened half-bordered pit aperture in the cross-field area; c) partial degradation of pit tori (leftward arrows) and complete degradation of torus (rightward arrow); d) pit degradation limited to tori

Slika 2. SEM mikrografije uzoraka drva zaraženih bakterijom *B. subtilis* UTB22: a) djelomična razgradnja traheide ograničena na torus jažice; b) poluotvorena jažica na poprečnom presjeku; c) djelomična razgradnja torusa jažice (lijeve strelice) i potpuna razgradnja torusa jažice (desna strelica); d) razgradnja jažice ograničena na torus

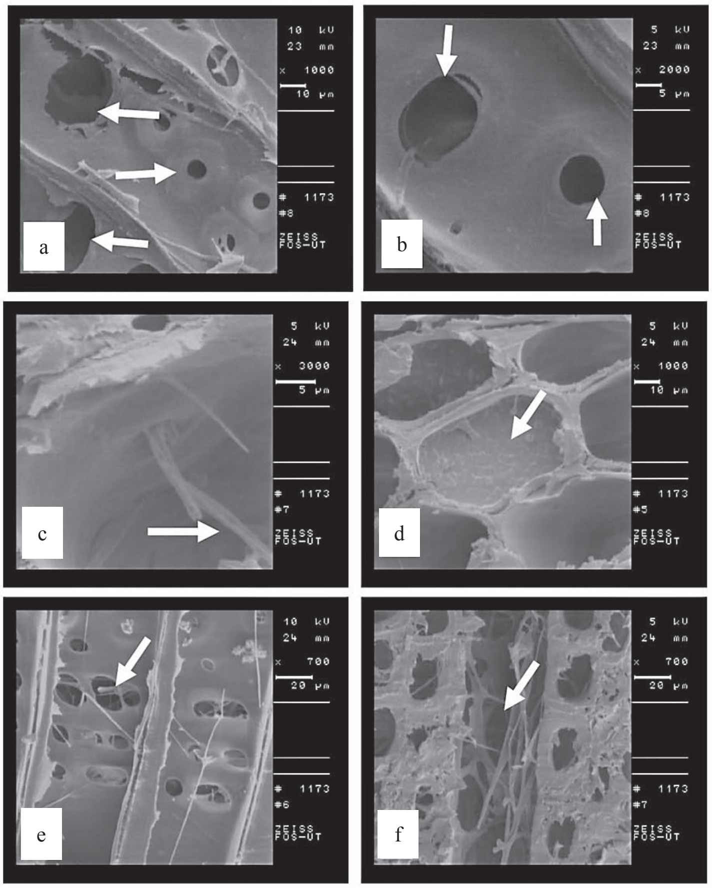

Figure 3 SEM images of wood specimens incubated by *P. vitreus* (Pers.: Fr.) P. Karsten isolate 136: a) totally degraded tracheid bordered pits together with cell wall degradation (leftward arrows) and unaffected bordered pit (rightward arrow); b) a completely digested pit (downward arrow) versus a pit with decomposed torus (upward arrow); c) passing of fungal hyphae through inter-tracheid pits; d) accumulation of fungal hyphae in a tracheid lumen; e) degradation of cross-field pits in the vicinity of hyphae; f) degradation of ray parenchyma cells

Slika 3. SEM mikrografije uzoraka drva zaraženih gljivom *P. vitreus* (Pers.: Fr.) P. Karsten izolat 136: a) potpuna razgradnja traheide na granici s jažicama s istodobnom razgradnjom stanične stijenke (lijeve strelice) i nepromijenjena ograđena jažica; b) potpuno razgrađena jažica (strelica prema dolje) u odnosu prema jažici s razgrađenim torusom (strelica prema gore); c) prolazak hifa gljive kroz jažicu između traheida; d) nakupine hifa gljive u lumenu traheide; e) razgradnja poprečnog presjeka jažice u blizini hifa; f) razgradnja parenhima u drvnim tracima penetration of fungal hyphae in the porous structure of wood (Figures 3a and c) was observed in the current study. A heterogeneous fungal colonization in timbers due to differences in the heartwood percentage, moisture content, and availability of nutrients is one of the main drawbacks of such biological incising methods on an industrial scale (Schwarze *et al.,* 2006; Dale *et al.,* 2019).

In agreement with previous reports (Schwarze and Landmesser, 2000; Schwarze *et al.,* 2006; Lehringer *et al.,* 2010; Fuhr *et al.,* 2011), *P. vitreus* showed a much higher activity in earlywood than in latewood (see Figure 3d for the fungal hyphae accumulation within the cell lumens), which is often attributed to the greater amount and more availability of nutrients in earlywood (Fuhr *et al.,* 2011) as well as the higher resilience of latewood tracheids to decay. At the initial stage of wood colonization, degradation is more obvious in earlywood, but at the advanced stage of fungal activity, latewood was also attacked (Schwarze *et al.,* 2008).

3.3 Air permeability and micro-scale treatability
3.3. Propusnost zraka i impregnacija na mikrorazini

Both *P. vitreus* and *B. subtilis* UTB22 were able to significantly enhance the permeability of Loblolly pine heartwood (Table 1), which was increased by around 56 % in the tangential direction and 124 % in the longitudinal direction after fungal exposure. The improvement was more remarkable when the specimens were exposed to *B. subtilis* UTB22 (485 % for tangential permeability and 676 % for longitudinal permeability). Several studies have also shown that different isolates of *P. vitreus* can significantly improve wood permeability, even after a short period of colonization (Schwarze and Landmesser, 2000; Schwarze *et al.,* 2006; Lehringer *et al.,* 2010; Fuhr *et al.,* 2011; Emaminasab *et al.,* 2015, 2016); however, the changes in permeability vary with the wood species, the fungal isolate, and incubation conditions (Schwarze *et al.,* 2006). The improvement in permeability is mostly attributable to the degradation of the pit membranes (Schwarze *et al.,* 2006; Schwarze and Schubert, 2011; Fuhr *et al.,* 2011). It is believed that the secretion of pectinase enzyme by *B. subtilis* plays an important role in improving wood permeability through the decomposition of pectin compounds and hemicellulose of the pit membrane (Yildiz *et al.,* 2012). Possible reasons for the lower improvement in permeability by *P. vitreus* compared to *B. subtilis* UTB22 are the heterogeneous fungal activity, accumulation of fungal hyphae, and degradation products within the cell lumens (Figure 3d), which may block the fluid flow paths (Emaminasab *et al.*, 2015).

Micro-scale treatability studies under a fluorescence microscope showed that the treatability of the tracheid cells increased after incubation by both microorganisms (Figure 4). The present results clearly demonstrate the higher bioincising efficiency of *B. subtilis* UTB22 compared to *P. vitreus* (Pers.: Fr.) P. Karsten isolate 136. While most tracheid lumina were filled with the impregnating solution after bacterial exposure (Figures 4c, e), many of the lumina remained empty in the wood specimens bioincised by *P. vitreus* (Figures 4b, d).

A non-uniform penetration of the solution into the specimens exposed to *P. vitreus* (Figure 4b) may be

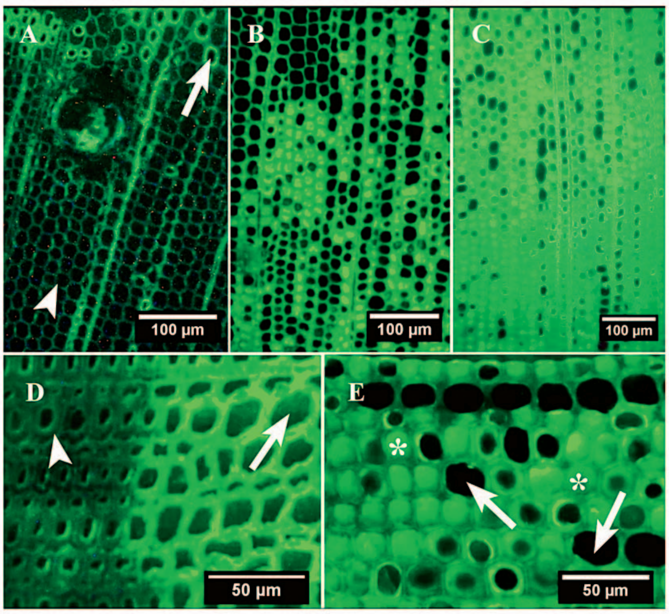

Figure 4 Microscopic treatability of wood samples using fluorescent dye-containing water: A) control, B) and D): fungal treated, C) and E) bacterial treated samples; the green color shows the presence of fluorescence solution; arrowheads, arrows, and asterisks, respectively, show untreated tracheids, tracheids with treated cell walls, and tracheids in which both cell wall and lumen are filled with liquid

Slika 4. Mikroskopska obrada uzoraka drva uz pomoć fluorescentnog bojila otopljenoga u vodi: A) kontrolni uzorak, B) i D) uzorci zaraženi gljivom, C) i E) uzorci zaraženi bakterijom; zelena boja potvrđuje prisutnost fluorescentne otopine; vrhovi strelica, strelice i zvjezdice pokazuju neimpregnirane traheide, traheide s impregniranim staničnim stijenkama i traheide s impregniranim staničnim stijenkama i lumenima

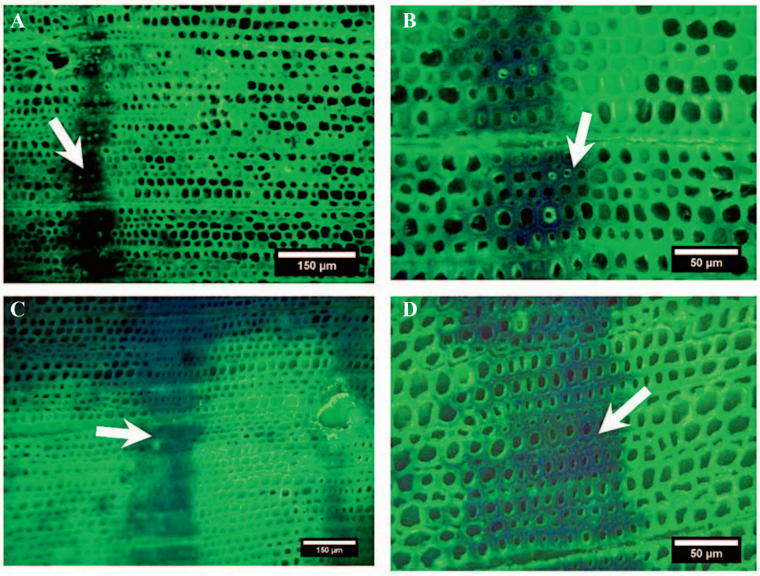

Figure 5 The treatability of earlywood vs. latewood of wood specimens; a) and b) incubated with *B. subtilis* UTB22; c) and d) incubated with *P. vitreus* (Pers.: Fr.) P. Karsten isolate 136. Arrows show untreated latewood
Slika 5. Impregnacija ranog drva u usporedbi s kasnim: a) i b) uzorci zaraženi bakterijom *B. subtilis* UTB22; c) i d) uzorci zaraženi gljivom *P. vitreus* (Pers.: Fr.) P. Karsten izolat 136.; strelice pokazuju neimpregnirano kasno drvo

because of the heterogeneous opening of the aspirated bordered pits by the fungus. Schwarze *et al.* (2006) visually inspected the uptake of bluish dye Neolan Glaucin E-A by Norway spruce and silver fir (*Abies alba*) wood blocks incubated with two isolates of *P. vitreus* (EMPA 642 and EMPA 643), and found that the uptake within *P. abies* was less homogeneous. Lehringer *et al.* (2009a) also reported that the uptake and penetration depth of aqueous modification substances after vacuum impregnation of the bioincised wood were highly variable due to the heterogeneous colonization of the wood by *P. vitreus*.

After bioincising, earlywood showed better treatability than latewood (Figure 5). Only a few latewood cells were treated even after the fungal or bacterial incubation, which indicates the weaker ability of the microorganisms to degrade latewood cells. Therefore, this treatability enhancing strategy may be less successful in improving the treatability of softwoods with high latewood percentage, like the Pinaceae family.

4 CONCLUSIONS
4. ZAKLJUČAK

The present work comparatively studied the degradation pattern, permeability, and micro-scale treatability of Loblolly pine heartwood after incubation with two bioincising microorganisms (white-rot fungus *Physisporinus vitreus* (Pers.: Fr.) P. Karsten isolate 136 and bacterium *Bacillus subtilis* UTB22). Both microorganisms improved treatability significantly, mainly because of degradation of the pit membranes. However, a higher treatability with more uniform impregnation of the wood cells was obtained with *B. subtilis* UTB22 despite a higher mass loss with *P. vitreus*. SEM observations showed that the lower treatability enhancing efficiency of the fungus was possibly due to the heterogeneous pit degradation and blockage of the tracheid lumens with the fungal hyphae. Earlywood was more treatable than latewood after bioincising because of the higher degradation of the former.

It can be concluded that degradation by *B. subtilis* UTB22 was more selective than that by *P. vitreus* (Pers.: Fr.) P. Karsten isolate 136. Degradation by the bacterium was mainly confined to the tori of the pits, whereas some pits were completely degraded by the fungus and the cell walls were also attacked. Based on the current results, a heterogeneous impregnation of the bioincised wood cells can also be an important challenge for scaling up the bio-incising of wood with *P. vitreus* to an industrial scale.

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Finite Element Analysis of Dynamic Characteristics and Bending Stiffness for Cross Laminated Timber Floor Panels with and without Openings

Analiza dinamičkih obilježja i krutosti pri savijanju podnih ploča od križno lameliranog drva provedena metodom konačnih elemenata

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ABSTRACT • *The aim of this paper is to present numerical investigations of dynamic characteristics and bending stiffness for cross laminated timber floor panels with and without service openings. Five-layer panels with the outer layers oriented in the longitudinal direction of the panel have been analyzed. In order to explore the full potential of this floor system using a limited number of measurements and structural tests, models based on the finite element method have been proposed, validated against experimental results and then used to investigate the effect of opening position in the floor on main structural performance parameters. The results showed that, when the need for additional service opening appears, a slight decrease of the main structural characteristics of the cross laminated timber floor panels is achievable with an adequate geometrical position of the opening in the floor.*

Keywords: *cross laminated timber floors; bending stiffness; floors with openings; finite element analysis*

SAŽETAK • *Cilj ovog rada jest prikaz numeričkog modela dinamičkih obilježja i krutosti pri savijanju podnih ploča od križno lameliranog drva (CLT) sa servisnim otvorom i bez njega. Analizirane su peteroslojne ploče s vanjskim slojevima orijentiranim u uzdužnom smjeru ploče. Kako bi se uz pomoć ograničenog broja mjerenja i mehaničkih ispitivanja istražio puni potencijal takvoga podnog sustava, predloženi su modeli utemeljeni na metodi konačnih elemenata koji su validirani u usporedbi s eksperimentalnim rezultatima te primijenjeni za istraživanje utjecaja položaja otvora u podu na glavne parametre ponašanja konstrukcije. Rezultati su pokazali da se odgovarajućim pozicioniranjem naknadnog otvora u podu može postići vrlo malo smanjenje glavnih mehaničkih svojstava podnih ploča od CLT-a.*

Ključne riječi: *podne ploče od CLT-a; krutost pri savijanju; podovi s otvorima; analiza metodom konačnih elemenata*

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1 INTRODUCTION
1. UVOD

Cross laminated timber (CLT) is a sophisticated modern product that has greatly improved the load-bearing capacity of traditional wood as building material. CLT was first developed and used in Germany and Austria in the early 1990s and has been gaining popularity ever since (Blass and Fellmosser, 2004; Laguarda-Mallo and Espinoza, 2014; Espinoza *et al*., 2016). It is increasingly used in construction applications such as floors, walls, beams and has the potential to replace concrete, masonry, and steel in some applications (Pei *et al*., 2016; Wei *et al*., 2020). CLT now has a global reach, with many large buildings erected using this material (Cvetkovic *et al*., 2015; Popovski and Gavric, 2016).

CLT elements are mainly used for walls and floor structures. If used as shear walls in buildings, in-plane-stiffness is an important consideration. In-plane loading for CLT has been extensively studied in the literature (Shahnewaz *et al*., 2017; Wang *et al*., 2018; Danielsson and Serrano, 2018; Song *et al*., 2019). Considering the fact that the out-of-plane stiffness is important for floors, many researchers conducted out-of-plane loading tests and proposed new design procedures (Park *et al*., 2003; Minjuan *et al*., 2018; Okabe *et al*., 2018; Song and Hong, 2018; Pang and Jeong, 2019; Crovella *et al*., 2019). Its dynamic behavior is another characteristic, important for the serviceability of these structures, extensively studied in the literature (Damme *et al*., 2017; Ussher *et al*., 2017; Casagrande *et al*., 2018; Kozaric *et al*., 2019; Huang *et al*., 2020).

The bending stiffness and bending strength of CLT floor panels are important properties because they determine the design of CLT elements. In order to obtain the most rational construction, designers are often guided by the highest possible utilization of stresses, but meeting the structural safety, that is determined by serviceability limit state. However, during the exploitation of the structure, the need for additional opening can appear. Openings in the floors are needed for architectural purposes such as passing service ducts or passing shear walls or continuous structural elements. Adding such openings in the floors, when they were not taken in the account during the design, affects the efficiency of the panel in carrying the assigned loads.

Investigation of the effect of openings in the floor on bending stiffness was not much represented in the existing literature. One of the papers dealing with this problem is the paper by Popovski *et al*. (2016). They conducted experimental investigation of the structural and dynamic characteristics of CLT floor system with and without openings. The results showed the deflection of the CLT panel when the opening is more than 10 % larger than the deflection of the same panel without opening. This is in direct correlation with the stiffness, which is decreased with the presence of an opening. The above study shows the importance of investigating bending stiffness of CLT panels with openings in order to obtain results that can lead to safe and reliable structures.

This paper presents the analysis of bending stiffness of CLT floor panels with and without openings. Modelling was performed using the ABAQUS software package, using the 2D and 3D elements. 3D model was used to investigate the influence of the gaps between boards in non-edge-glued floor panels on the mechanical and dynamical behavior of the panels. The gaps between boards have been shown to influence the shear and torsional stiffness in the panels (Franzoni *et al*., 2016; Franzoni *et al*., 2017). The aim of this paper is to investigate the change of bending stiffness due to the presence of an opening and to make recommendations on the position of the opening so as to cause the least possible reduction of stiffness.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

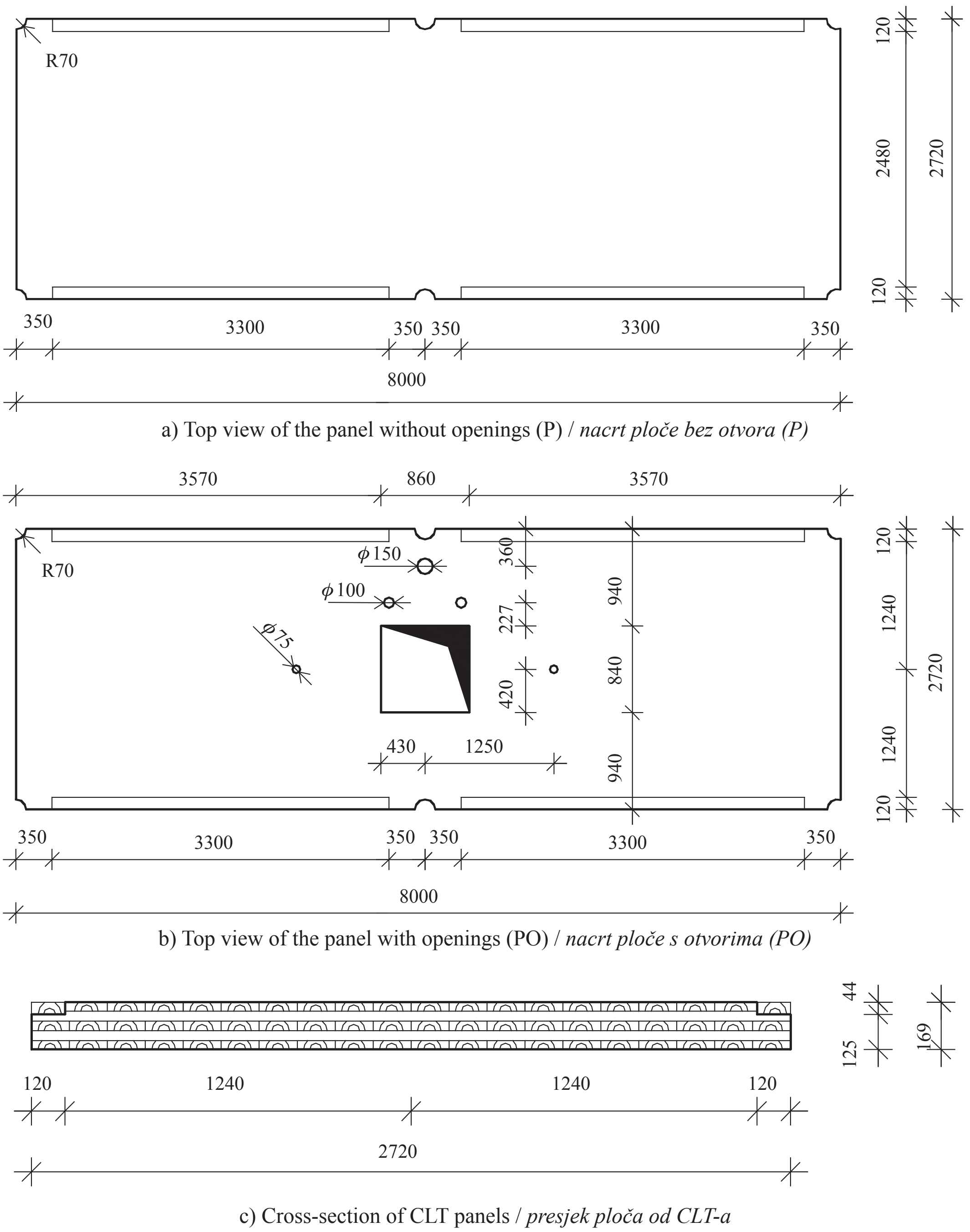
Two types of CLT floor panels from 3 different manufacturers, with and without openings, were analyzed. They are defined as P1-P3 and PO1-PO3, respectively. All CLT panels had 5 layers with the outer layers oriented in the longitudinal direction of the panel with dimensions identical to the point-supported CLT floor system experimentally analyzed by Popovski *et al*. (2016) (Table 1). Geometry of the panels is shown in Figure 1.

A two-point loading method was used for the non-destructive tests. This method considers applying loads at two points that are located in the middle of each span of the floor (Figure 2). Each CLT floor panel was loaded up to its service design load level and the deflection was measured. The estimated service design load for the panels without openings was 148 kN and for the panels with service openings 108 kN. In this research, all panels were loaded with load of 54 kN per span in order to obtain comparable results.

Once the experimental tests were completed, Popovski *et al*. (2016) determined the average density and moisture content of CLT panels. The results showed that the average density of the CLT panels from manufacturer 1 was 473 kg/m^3, while it was 443

Table 1 Thickness of layers in CLT panels and total floor thickness (Popovski *et al*., 2016)
Tablica 1. Debljina slojeva ploča od CLT-a i ukupna debljina poda (Popovski *et al*., 2016.)

Manufacturer *Proizvođač*	Thickness of layers and CLT panels / *Debljina slojeva ploča i CLT-a,* mm					
	Layer / *Sloj*					Total *Ukupno*
	1	2	3	4	5	
1	40	30	30	30	40	170
2	34	34	34	34	34	170
3	32	35	35	35	32	169



a) Top view of the panel without openings (P) / *nacrt ploče bez otvora (P)*

b) Top view of the panel with openings (PO) / *nacrt ploče s otvorima (PO)*

c) Cross-section of CLT panels / *presjek ploča od CLT-a*

Figure 1 Geometrical characteristics of CLT floor panels (Popovski *et al*., 2016)
Slika 1. Geometrijska obilježja ploča od CLT-a (Popovski *et al*., 2016.)

kg/m^3 and 452 kg/m^3 for the panels from manufacturers 2 and 3, respectively. The moisture content of the CLT panels from manufacturer 1 and 2 was similar (about 8.9 %), while the CLT panels from manufacturer 3 had higher moisture content of 13.3 %.

Finite element method (FEM) was applied by using the Abaqus 6.13-1 software package. In order to calibrate the applied modelling method, the numerically determined behavior of the panels was compared with the experimentally obtained values (Popovski *et al*., 2016). Three different simulation models were used: one 2D model and two 3D models (one for edge-glued and one non-edge-glued panel). The 2D model was only used for simulation of edge-glued panels because non-glued edges cannot be modelled under 2D. The adopted mesh was determined by an iterative procedure, until the results of two successive steps differed by less than 1 %.

The 2D model was created using Abaqus internal Composite Layup Manager. The material, thickness, rotation angle, and number of integration points per layer were specified for the lay-up. The model was meshed with S8R elements, an eight-node doubly curved thick shell element with reduced integration (Figure 3).

The 3D models were built up in 5 layers using individual boards assembled to a panel (Figure 4). A

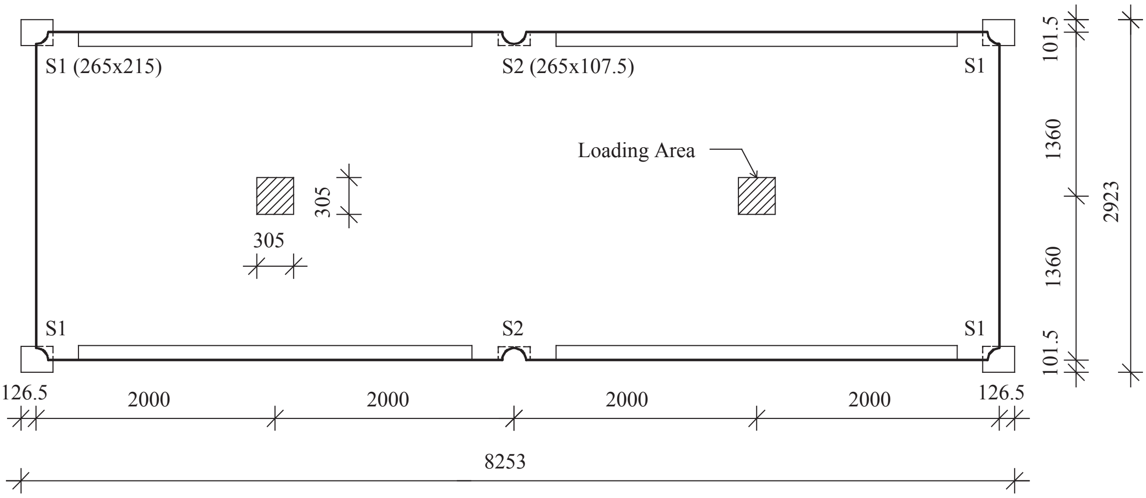
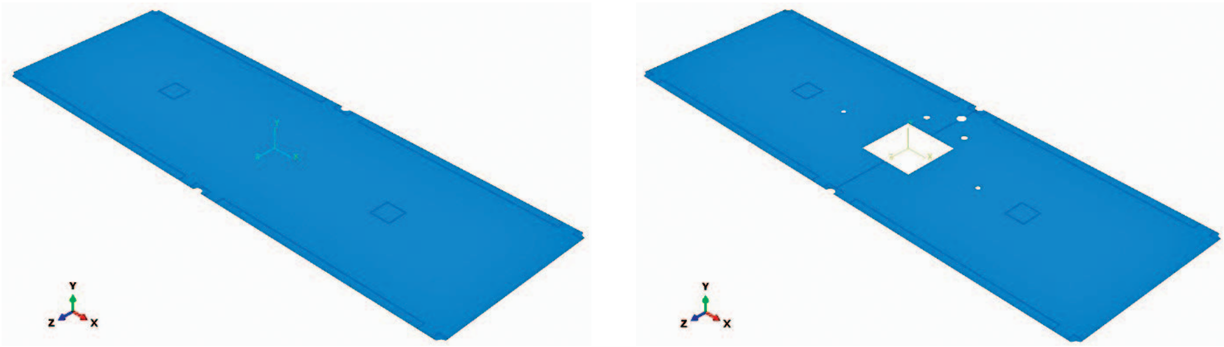


Figure 2 Location of loading points and supports (Popovski *et al*., 2016)
Slika 2. Položaj mjesta opterećenja i oslonaca (Popovski *et al*., 2016.)



a) Panel without openings / *ploča bez otvora*   b) Panel with openings / *ploča s otvorima*

Figure 3 2D model of CLT floor panels
Slika 3. 2D model ploča od CLT-a

Cartesian coordinate system was used to specify the material directions. Glued contact surfaces between the layers were simulated with rigid contact conditions, which did not allow any movement between contact areas. To model the non-glued contact surfaces between the side edges of the boards, flexible contact condition was applied allowing tangential sliding without friction. The model was meshed with the element C3D20R, which is a 20-node quadratic hexahedral element with reduced integration (Figure 5).

Once the FE-model is verified against experiments, the influence of different opening position on the bending stiffness of the CLT floor system was simulated towards the improvement of the design of the CLT floor panels with openings using a limited number of measurements and structural tests. Another 4 different type panels with one single opening in different positions were analyzed (Figure 6). They are defined as PO11-PO14. The service opening for all analyzed panels had the same dimensions and shape, square with sides of 860 mm × 840 mm.

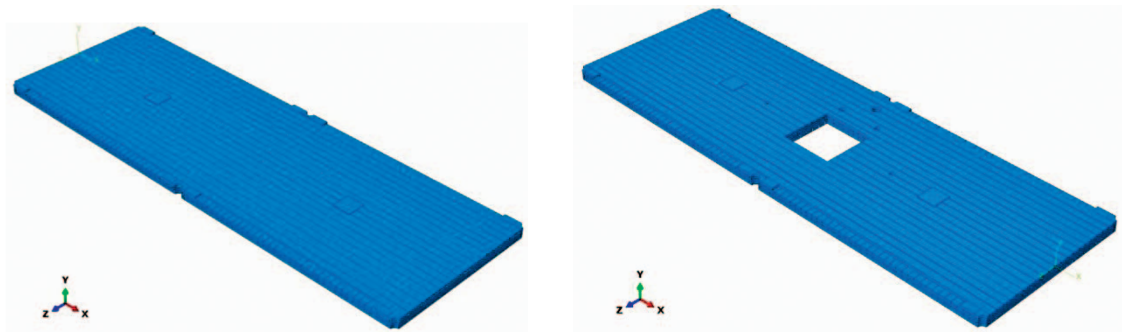
Figure 4 3D model of assembled boards
Slika 4. 3D model sastavljenih ploča

3 RESULTS
3. REZULTATI

The fundamental frequency and the maximum deflection of the CLT floor panels with and without openings are shown in Table 2. The results obtained by numerical analysis in Abaqus (6.13-1) were compared with the experimentally determined data. Deformed shape and deflection values for the CLT floor panels from manufacturer 1 under the two point loads are illustrated in Figure 7. Comparison of the obtained results from 3D non-edge-glued model with variation of opening position in the panel is provided in Table 3.

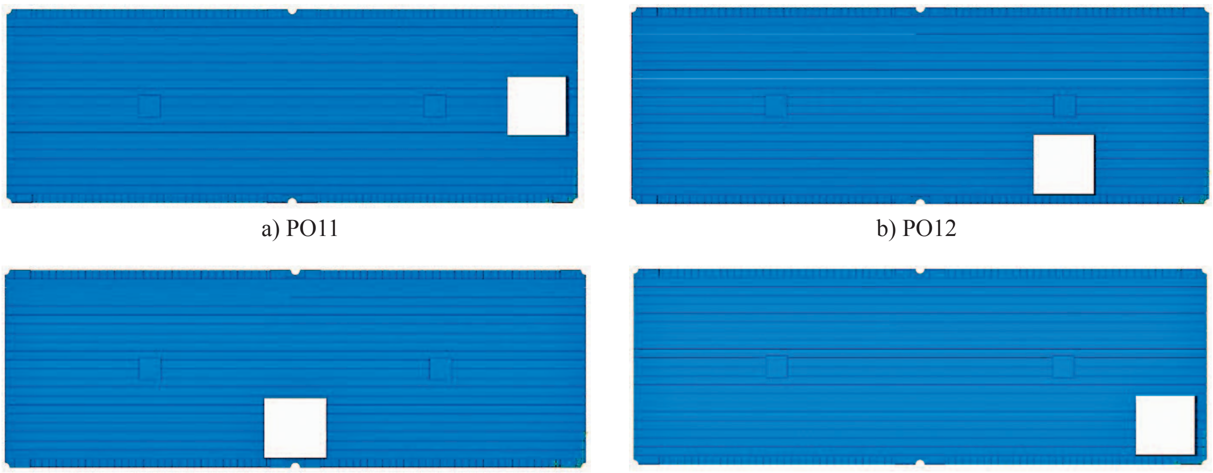
4 DISCUSSION
4. RASPRAVA

The numerically obtained results for the proposed model, shown in Tab. 2, correspond to the results obtained experimentally. The non-edge-glued 3D mod-



a) Panel without openings / *ploča bez otvora* b) Panel with openings / *ploča s otvorima*

Figure 5 3D model of CLT floor panels
Slika 5. 3D model ploča od CLT-a

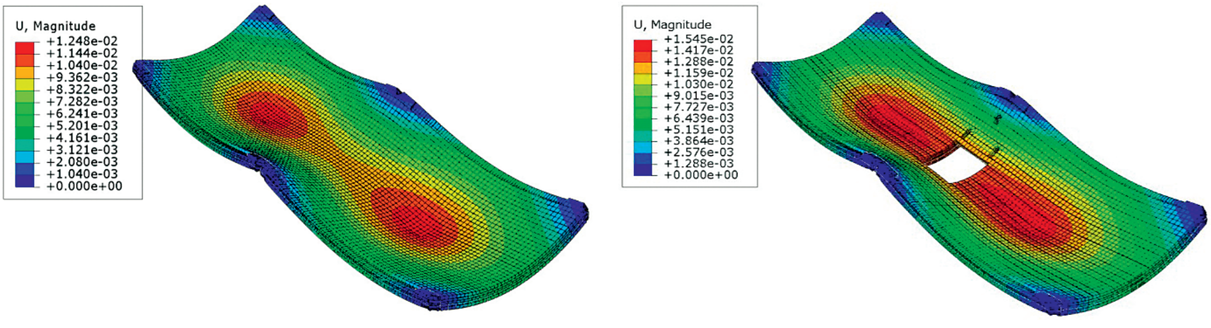


a) PO11 b) PO12 c) PO13 d) PO14

Figure 6 Positions of opening in analyzed panels
Slika 6. Položaji otvora u analiziranim pločama

Table 2 Fundamental frequency and maximum deflection of CLT floor panels
Tablica 2. Osnovna frekvencija i najveći pomak podnih ploča od CLT-a

	Model P1	**No openings** *Bez otvora*			**With openings** *S otvorima*		
		P2	P3	PO1	PO2	PO3	
Frequency, Hz *Frekvencija, Hz*	Experimental results (Popovski *et al*., 2016) *eksperimentalni rezultati (Popovski et al., 2016.)*	16.8	17.5	16.8	15.9	16.0	15.5
	2D	17.1	18.0	17.4	16.5	17.8	17.2
	Difference / *razlika, %*	1.8	2.9	3.6	3.8	11.2	11.0
	3D edge-glued / *3D lijepljeni rub*	17.9	19.4	18.6	17.4	18.7	18.2
	Difference / *razlika, %*	6.5	10.9	10.7	9.4	16.9	17.4
	3D edge-glued / *3D bez lijepljenog ruba*	16.6	17.5	17.1	15.7	16.6	15.3
	Difference / *razlika, %*	-1.2	0.0	1.8	-1.3	3.7	-1.3
Deflection, mm *Pomak, mm*	Experimental results, average Popovski *et al*. (2016) *eksperimentalni rezultati, srednje vrijednosti (Popovski et al., 2016.)*		11.3			12.9	
	2D	12.0	10.6	11.0	13.3	11.8	12.3
	Average / *srednja vrijednost*		11.2			12.5	
	Difference / *razlika, %*		-0.9			-3.1	
	3D edge-glued / *3D lijepljeni rub*	11.2	10.2	10.6	12.5	11.3	11.7
	Average / *srednja vrijednost*		10.7			11.8	
	Difference / *razlika, %*		-5.3			-8.5	
	3D edge-glued / *3D bez lijepljenog ruba*	12.5	10.8	11.3	15.4	13.0	13.7
	Average / *srednja vrijednost*		11.5			14.0	
	Difference / *razlika, %*		1.8			8.5	



a) Panel without openings / *ploče bez otvora*　　b) Panel with openings / *ploče s otvorima*

Figure 7 Deformed shape and deflection values of CLT panels from manufacturer 1
Slika 7. Deformacija i vrijednosti pomaka ploča od CLT-a proizvođača 1

el proved to be the most appropriate model for accurate simulation of CLT panel behavior.

The 2D model is the simplest and fastest to model, but gives slightly higher values of the fundamental frequency than the experimental results, 1.8-3.6 % for panels without openings, while for panels with openings these deviations are much larger (3.8-11 %) and depend on panel manufacturers. The maximum deflection of the CLT floor panels is 0.9 % lower for panels without openings, and for panels with openings this deviation is slightly larger and amounts to 3.1 %. With this model, the gaps between the boards for the non-edge-glued panels cannot be modelled. In 3D modelling, it is possible to account for this feature in calculations when constructing a floor.

The largest deviations from the experimental results appear in using the 3D edge-glued model. In edge-glued CLT panels, the side edges of the boards in each layer are glued together, which yields a fully solid plate without gaps, indicating greater stiffness, and therefore their fundamental frequency is much higher than the experimental results. For panels without openings, 6.5-10.7 % higher value is obtained, for panels with openings the difference goes up to 17.4 %. The difference between the numerical results and the experimental results is slightly smaller for the maximum deflection of the CLT floor panels, being 5.3 % for panels without openings and 8.5 % for panels with openings. The 3D non-edge-glued model simulates the most similar behavior of CLT panels. The largest difference in panels with an opening is 3.7 % for the fundamental frequency and 8.5 % for the maximum deflection.

3D non-edge-glued model, that has been shown to best represent the actual behavior of CLT panels in a parametric study of the influence of the opening position on the dynamic characteristics and bending strength of the CLT floor panels, was investigated. Based on the results presented in Table 3, it can be concluded that, although the presence of openings in the floor reduces the fundamental frequency and bending stiffness of the panels, choosing the favorable position of that opening can minimize the influence. This can be achieved by placing an opening in the middle of the panel or along the axis of the shorter edge of the panel.

4 CONCLUSIONS
4. ZAKLJUČAK

In this study, the main structural performance parameters of the CLT floor panels with and without openings were investigated and numerical models based on the finite element method were proposed. The model parameters were calibrated based on the previously published experimental results in the literature. The high correlation between the numerical and experimental data produces the opportunity to investigate and predict the structural behavior of these floor systems through various numerical parametric studies, since the experimental investigation is generally costly and requires a significant amount of time.

In the parametric study, the influence of the opening position on dynamic characteristics and bending strength of the CLT floor panels was investigated. During the exploitation of the structure, the need for additional service openings can appear. Adding such openings in the floors, when they were not taken into account during the design, affects the efficiency of the panel in carrying the assigned loads. The results of the

Table 3 CLT floor panels with different positions of opening
Tablica 3. Podne ploče od CLT-a s različitim položajima otvora

Model	**3D non-edge-glued (manufacturer 1)** / *3D bez lijepljenog ruba (proizvođač 1)*			
	Frequencys, Hz *Frekvencija*, Hz	**Difference, %** *Razlika,* %	**Deflection, mm** *Pomak,* mm	**Difference, %** *Razlika,* %
P1	16.6	/	12.5	/
PO1	15.7	-5.4	15.4	23.2
PO11	15.4	-7.2	15.3	22.4
PO12	15.3	-7.8	17.5	40.0
PO13	14.0	-15.7	16.0	28.0
PO14	13.6	-18.1	16.3	30.4

numerical analysis show that the slight decrease of the main structural characteristics of the CLT floor panels is achievable with an adequate geometrical position of the opening in the floor.

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Effect of Artificial Ageing on Adhesive Bonds from Heat Treated Spruce

Utjecaj umjetnog starenja na lijepljeni spoj toplinski modificirane smrekovine

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ABSTRACT • *The influence of artificial ageing on bonded heat-treated spruce lamellas was investigated. Heat-treated spruce lamellas with different degrees of thermal modification were bonded with PVAc and MUF and then exposed to 500 artificial weathering cycles, combined with rain, UV and IR radiation. The colour change of the exposed surface, weight change, delamination of the bonded joints and adhesive bond strength were measured. Artificial weathering caused cracking and delamination of the bonded joints and reduced the bond strength of both adhesives. The results show that delamination was higher for PVAc adhesive than MUF, but increased for both adhesives with the temperature of heat treatment of wood. The shear strength of bonds on the exposed side of the samples after the artificial weathering was lower than the average strength of the whole sample.*

Keywords: *heat treated spruce; adhesive bond; artificial ageing; shear strength; delamination; colour change*

SAŽETAK • *U radu je istražen utjecaj umjetnog starenja na lijepljene lamele toplinski modificirane smrekovine. Lamele toplinski modificirane smrekovine različitih stupnjeva toplinske modifikacije lijepljene su PVAc i MUF ljepilom, a zatim su tijekom 500 ciklusa izložene vremenskim utjecajima u kombinaciji kiše te UV i IR zračenja. Mjerena je promjena boje izložene površine, promjena mase te delaminacija i čvrstoća lijepljenog spoja. Umjetno izlaganje vremenskim utjecajima uzrokovalo je pucanje i delaminaciju lijepljenih spojeva te smanjenje čvrstoće spoja lijepljenog obama ljepilima. Rezultati pokazuju da je delaminacija bila veća na slojevima s PVAc ljepilom nego na slojevima lijepljenim MUF ljepilom, ali je povećana na slojevima s oba ljepila, uz povećanje temperature toplinske modifikacije drva. Nakon umjetnog izlaganja vremenskim utjecajima smična je čvrstoća lijepljenog spoja na izloženoj strani uzoraka bila niža od prosječne čvrstoće cijelog uzorka.*

Ključne riječi: *toplinski modificirana smrekovina; lijepljeni spoj; umjetno starenje; smična čvrstoća; delaminacija; promjena boje*

1 INTRODUCTION
1. UVOD

Thermal modification of wood can influence the gluing process with adhesives (Kariz, 2011) and reduce the performance of glued joints (Sernek *et al*., 2008). The shear strength of glued joints is usually reduced due to the lower strength of the modified wood (Esteves and Pereira, 2009) and the lower adhesion to the modified wood surface. Studies had shown that heat treated (HT) wood can be adequately bonded to most industrial wood adhesives with some modifications of the bonding process (longer pressing time, lower wood moisture content) and/or adhesive (higher

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solids content) (Militz, 2002; Mayes and Oksanen, 2003). What is less known is how the bonds in heat-treated wood change over time due to modified wood properties.

The ability of a joint to maintain satisfactory long-term behaviour, often in severe environments, is an important prerequisite for an adhesive joint, as the joint should be able to bear the intended loads for the intended life of the joint (Custódio *et al*., 2009). Over the years, the strength of the joints may decrease. Water and thus increased moisture content (MC) of the wood as well as the temperature lead to a reduction of the mechanical properties of the joined part (wood) (Green *et al*., 1999) and of the adhesive (Vick, 1999) as well as of the bonded joint (Blanchet *et al*., 2003).

Factors that influence the durability of structural adhesive joints can be divided into three categories: environment (temperature, humidity, solar radiation), materials (the joined part, adhesive and intermediate phase between the two) and stresses (stresses to which the joint is exposed during or after exposure to the operating environment and which affect both durability and residual strength) (Custódio *et al*., 2009, Follrich *et al*., 2011). In general, the durability of wood and wood products in dry conditions is good, i.e. with a (MC) below 15 %.

Chemical degradation of the adhesive line in the form of hydrolysis of the adhesive can occur through increased temperatures (e.g. heat or energy such as ultraviolet rays) in combination with moisture. These processes can also be influenced by acidic or alkaline environments. The resistance to hydrolysis depends on the type of adhesive: urea-formaldehyde (UF) adhesives are less resistant to hydrolysis than melamine-urea-formaldehyde (MUF) or phenolresorcinol-formaldehyde adhesives (PRF) (Marra, 1992).

Climate changes increase the hygroscopic exchange, which increases the shrinkage and swelling of wood. Alternating shrinkage and swelling leads to breakage of adhesive bonds and causes breaks in the bond or in the wood, but different adhesives react differently to artificial ageing with standard changing climate conditions (hot/cold, low/high RH): PUR adhesive can retain most of the initial strength, followed by MUF/UF adhesive and others (EPI, MUF, PRF) (Follrich *et al*., 2011). The PVA bond strength gradually decreased due to ageing processes (Blanchet *et al*., 2003). In addition, during swelling or shrinking, the induced stresses in the bond line and in the interface between two materials can be reduced by the creep behaviour of the adhesive used (Follrich *et al*., 2011).

The methods used to assess the durability of wood-based materials include long-term and short-term tests. Long-term assessments, such as outdoor exposure tests, have many disadvantages, such as being time-consuming and difficult to perform and are related to the test site. Short-term assessments evaluate changes in mechanical properties after accelerated ageing treatments, such as immersion in water, cooking, steaming, freezing or drying. Such accelerated ageing tests are artificial, but in recent decades many attempts have been made to correlate deterioration due to outdoor ageing with accelerated ageing in the laboratory (Kojima and Suzuki, 2011).

Most of the research on the ageing of adhesive bonds carried out so far has attempted to simulate the effect of ageing of adhesive bonds by using standard procedures that include different ageing cycles, usually combining cycles with changing climatic conditions such as (50 °C / 95 % relative humidity (RH), -20 °C / 65-70 % RH and 75 °C / 15 % RH) or with soaking in cold/boiling water, soaking in water under vacuum or pressure conditions, air drying at high temperature (70-100 °C) (Kojima and Suzuki, 2011).

The influence of the ageing process on the bonds is evaluated by measuring the shear strength of the bond, the internal bond strength or by measuring the delamination of the bond. This could predict the change in bond strength due to differences in temperature and moisture content of the wood, which are likely to be the main effects on the shear strength of the bond. On the other hand, these methods eliminate the influence of sunlight and rain on the bonded surfaces, which could be important for outdoor furniture or doors/windows.

The aim of this study was to measure the effect of artificial ageing on glued joints made of heat-treated spruce wood, which was produced with different adhesives. Normally the ageing of wood joints is done by standard methods, including soaking in cold or boiling water, freezing, but we wanted to use the ageing method for surface coating to see what effect UV and IR radiation and rain have on glued joints.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Heat treatment of wood and bonding
2.1. Toplinska modifikacija i lijepljenje

The spruce wood lamellae (*Picea abies* Karst) were heat treated at five different temperatures: 150, 170, 190, 210, 230 °C using the method of heat treatment with initial vacuum phase (Rep et al. 2004). Modified lamellas were then conditioned in standard climate (T = 20 °C, RH = 65 %) to achieve an equilibrium moisture (MC) before bonding with adhesives. Untreated samples were also prepared, conditioned and bonded in the same conditions to serve as control.

Two cold-curing adhesives with higher water resistance were used: PVAc Rakol GXL 4, class D4, (H. B. Fuller) and MUF adhesive, (Casco adhesives AB). The lamellas were planed prior to bonding to ensure an even and fresh surface. The adhesive application rate was 180 g/m^2. Bonding was performed in a press at room temperature and a pressure of 1 N/mm^2. The pressing time of 60 minutes for PVAc and 3 hours for MUF adhesive was determined according to the manufacturer's recommendations (30 min for PVAc and 2h for MUF) and extended due to the expected slower bonding of modified wood (Kariz *et al*., 2013). The glued lamellas were then conditioned in standard climate for a few months and then samples were sawn measuring 90 mm × 34 mm × 40 mm, each sample containing two adhesive joints

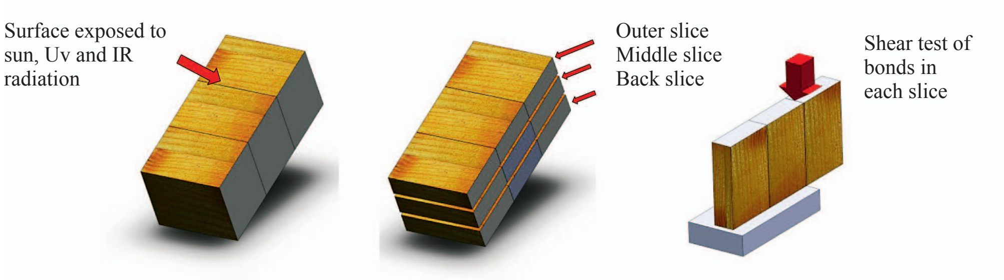

Figure 1 Left: position of specimens during artificial ageing cycles; middle: slicing specimens for bond shear testing; right: shear test of aged bonds
Slika 1. Lijevo: položaj uzoraka tijekom ciklusa umjetnog starenja; sredina: piljenje uzoraka za ispitivanje smične čvrstoće; desno: ispitivanje smične čvrstoće izloženog uzorka

(Figure 1 left). The cross sections were coated with two-component epoxy paint to prevent higher water absorption by these surfaces.

2.2 Artificial ageing
2.2. Umjetno starenje

The artificial ageing method was similar to the method used to test wood coatings for artificial weathering using fluorescent UV lamps, IR heating and water. The samples were exposed to artificial weathering in a special test chamber with water spray, UV and IR lamps. The duration of irradiation of IR was adjusted to reach 60 °C on white surfaces of the samples. The artificial weathering cycle lasted 1 hour and included 22 minutes of rain, 9 minutes of conditioning, 27 minutes of UV and IR irradiation and 2 minutes of conditioning. Every 100 cycles during the test, the samples were weighed, checked for cracks in the bond line and rotated in the chamber to eliminate the effect of possible uneven wetting and UV/IR irradiation. A total of 500 artificial weathering cycles were performed.

2.3 Colour measurement
2.3. Mjerenje boje

Colour change is not directly connected with bond strength change, but visually shows the severity of the ageing process and was thus measured to evaluate the possible correlation between surface colour change and bond strengths decrease. The colour of the samples was measured before and after the artificial ageing process. The samples were always conditioned in standard climate before the measurement to establish equilibrium *MC*. The colour of the surface was measured with the spectrophotometer SP62X-rite GmbH- Optotronik. The CIELAB system was used. In the CIELAB system, the L^* axis represented the brightness (L^* varied from 100 (white) to zero (black)), and a^* and b^* were the chromaticity coordinates. ($+a^*$ was for red, a^* for green, $+b^*$ for yellow, $-b^*$ for blue). The obtained L^*, a^* and b^* values were used to calculate the colour changes ΔE* according to Eq 1 (ISO /DIS 7724-3:1997):

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

Where ΔL^*, Δa^* and Δb^* were the differences between the colour values of the samples before and after the artificial ageing.

2.4 Bond delamination
2.4. Delaminacija lijepljenog spoja

Every 100 ageing cycles, the samples were weighed to measure water absorption, and checked for bond cracks. Crack lengths were measured on cross sections and photos were taken of delaminated bonds. The average length and sum of delaminations for all specimens with the same adhesive and heat treatment were calculated.

2.5 Shear strength
2.5. Smična čvrstoća

After ageing, we wanted to measure the influence of artificial ageing on the shear strength. The samples were cut into three 8 mm thick discs: outer disc - exposed to rain and UV/IR radiation, middle disc and rear disc. Each joint in each slice was loaded on a universal testing machine until failure, and shear strength and the percentage of wood failure were determined. For each adhesive and thermal treatment, 7 samples with two adhesive bonds (Figure 1 left) were tested - thus 14 adhesive bonds (40 mm × 8 mm) and in total (separated slices for outer, middle and back slice) 252 bonds were tested. Average values were calculated as the average in each slice and the average in the whole sample (disregarding the position in the samples).

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

During the artificial ageing cycle, the samples were exposed to UV and IR radiation. The first visual change was the fading of the colour. The colour change shows severity of the ageing process (Kržišnik *et al.*, 2018) and may also indicate some changes in surface structure and degradation, which could also be associated with the degradation of the adhesive bond. The surfaces of the samples turn grey and there were fewer colour differences between the control and most modified samples after ageing. The control samples became grey (decrease in L-value) and most modified samples (HT210, HT230) became lighter (increase in L-value) (Table1).

The colour change ΔE^* shows that the highest colour change occurred in the control samples, followed by the most heat treated samples - these were the

Table 1 Surface colour before and after ageing process, CIELAB system
Tablica 1. Boje površine prije i nakon starenja, CIELAB sustav

Sample *Uzorak*	**Before ageing** / *Prije starenja*			**After ageing** / *Nakon starenja*			ΔE*
	L*	a*	b*	L*	a*	b*	
Control	82.90	3.88	22.43	67.87	7.50	22.96	15.48
HT150	81.08	5.14	25.35	68.30	6.50	21.54	13.41
HT 170	77.67	6.04	26.25	66.75	6.82	21.32	12.01
HT 190	67.12	9.91	29.05	60.75	6.68	21.79	10.18
HT 210	48.63	11.92	25.00	54.82	6.24	16.51	11.94
HT 230	40.79	11.70	22.31	52.62	6.41	16.06	14.38

Table 2 Weight increase (%) in samples during ageing process. Values for standard deviation in brackets
Tablica 2. Povećanje mase (%) uzoraka tijekom starenja; u zagradama su vrijednosti standardne devijacije

	Sample *Uzorak*	**Number of ageing cycles** / *Broj ciklusa izlaganja*				
		100	200	300	400	500
Group of samples *Grupa uzoraka*	Control	13.7 (1.1)	18.1 (1.8)	16.1 (1.9)	15.8 (2.4)	18.9 (2.5)
	HT 150	15.6 (4.0)	19 (2.0)	17.6 (2.7)	19.5 (3.2)	21.1 (4.5)
	HT170	14.1 (4.7)	17.4 (5.2)	17.8 (6.7)	18.1 (6.9)	19.3 (6.1)
	HT190	10.3 (2.4)	12.5 (1.9)	12.4 (3.8)	13.7 (3.1)	13.9 (2.2)
	HT210	8.9 (1.0)	12.1 (1.6)	10.8 (2.3)	12 (2.1)	13.2 (2.6)
	HT230	6.2 (1.0)	7 (1.2)	9.2 (5.1)	8.5 (1.8)	8.8 (2.0)

samples that initially showed the greatest difference - with the control samples being the lightest and the most modified samples being the darkest (Table 2). The least colour change was seen in HT190. If the effect of the combination of wetting, UV and IR light was so intense that the colour of the surface was changed, this could also affect the bonds that were exposed to this treatment (Blanchet *et al*., 2003; Follrich *et al*., 2011), especially affecting the formation of bond cracks on exposed surfaces.

During the ageing process, the weight of the samples increased due to water absorption. The highest water absorption after 500 weathering cycles was measured in the case of control and less heat treated samples (HT150, HT170) and was 18.9 % to 21.1 %) and decreased with increasing heat treatment temperature and was lowest for most heat treated woods (HT 230 8.8 %). Statistical analysis showed only difference between control/less treated wood (HT150, HT 170) and more treated wood (HT190, HT210, HT230) groups. Higher water absorption also means higher swelling and stresses in glued joints, which could contribute to reducing bond shear strength with artificial ageing. Also, initial bond strength was determined on samples conditioned in standard climate (thus having *MC* between 4.8 % -HT230 to 10.9 % - control samples (Kariz, 2011), but the strength after ageing was measured on wet samples as the came from last ageing cycle. This higher *MC* also affects the measured bond strength (Bomba *et al*., 2014).

3.1 Bond delamination
3.1. Delaminacija lijepljenog spoja

The PVAc adhesive bonds of HT210 and HT230 began to delaminate during the first 100 ageing cycles and the average crack length increased with the number of ageing cycles (Table 3). The delamination was highest on the most treated woods and the average de-

Table 3 Average delamination length and sum of delaminations (in brackets) in specimens with same adhesive and heat treatment after each 100 ageing cycles (mm)
Tablica 3. Srednja dužina delaminacije i ukupna delaminacija (u zagradama) na uzorcima s istim ljepilom i toplinskom modifikacijom nakon svakih 100 ciklusa starenja (mm)

	Sample *Uzorak*	**Number of ageing cycles** / *Broj ciklusa izlaganja*									
		PVAc					MUF				
		100	200	300	400	500	100	200	300	400	500
Group of samples *Grupa uzoraka*	Control	0.0 (0)	0.0 (0)	0.0 (0)	0.0 (0)	0.0 (0)	0.0 (0)	0.0 (0)	0.0 (0)	0.0 (0)	0.0 (0)
	HT150	0.0 (0)	0.1 (1.4)	0.3 (5.5)	0.5 (7.6)	0.7 (11.1)	0.0 (0)	0.0 (0)	0.0 (0)	0.0 (0)	0.0 (0)
	HT170	0.0 (0)	0.0 (0)	0.4 (0.4)	0.6 (8.0)	0.6 (8.7)	0.0 (0)	0.0 (0)	0.0 (0)	0.0 (0)	0.0 (0)
	HT190	0.0 (0)	0.0 (0)	0.0 (0)	0.1 (1.4)	0.2 (2.5)	0.0 (0)	0.0 (0)	0.2 (2.8)	0.2 (3.4)	0.4 (7.1)
	HT210	3.1 (36.6)	4.1 (49.1)	4.8 (57.6)	5.5 (65.5)	5.6 (66.7)	0.0 (0)	0.0 (0)	1.4 (22.1)	2.1 (33.2)	2.3 (37.3)
	HT230	1.4 (22.1)	2.5 (39.2)	3.4 (53.9)	3.5 (56.1)	4.3 (69.2)	0.0 (0)	0.0 (0)	0.6 (9.2)	0.9 (13.9)	1.5 (24.6)

lamination crack length was 5.6 mm for HT210 and 4.3 mm for HT230 after 500 cycles. The samples of less heat treated wood (HT150, HT170, HT190) showed only slight delamination with an average crack length less than 0.8 mm. The bonds of control samples (non-modified wood) did not show any cracks.

The delamination of MUF adhesive bonds started later than that of PVAc bonds, after 200 ageing cycles, and it was much lower. The highest delamination was also found at HT210, and the average crack length was 2.3 mm after 500 ageing cycles. Some delamination was also observed on HT230 (max. average crack length 1.6 mm) and HT190 (max. average crack length less than 0.4 mm). Other samples glued from less heat treated wood (HT150, HT170) and control samples showed no delamination.

One of the reasons for poorer performance of the PVAc adhesive could also be caused by the combined effect of increased water content and temperature. The PVAc adhesive is thermoplastic and softens at higher temperatures. During exposure, the samples on the IR radiation surface heat up to simulate solar radiation. The IR radiation lamps were adjusted so that the surface of the control samples reached a maximum of 60 °C. As the samples from heat-treated spruce had a darker colour, they heated up more and so the maximum temperature on the surface of most heat-treated samples (HT230) was up to 72 °C. This higher temperature on more treated samples could be one of the reasons for worse results in case of PVAc adhesive joints. PVAc adhesives have a low temperature resistance and soften above 50 °C. A lower cohesive strength of the adhesive leads to delamination of the joint due to stresses acting on the joints as the wood, which is alternately exposed to drying and wetting, shrinks and swells.

Delamination increased for both adhesives with the degree of heat treatment (except HT230 and HT210) (Kariz *et al*., 2012) and the number of ageing cycles. The comparison between PVAc and MUF adhesive bonds showed that delamination was higher for PVAc adhesives than for MUF adhesives. The reason for this could be the properties of the adhesive - PVAc is thermoplastic and softens above 50 °C. So, heating the surface of IR softens the adhesive and stresses due to swelling and shrinkage could cause the joints to open. The modified wood with darker surface colour (higher L^* colour value) heated more than lighter control wood and this could increase the bond delamination of the modified wood.

3.2 Shear strength
3.2. Smična čvrstoća

The average shear strength of PVAc adhesive bonds decreased in all sample groups except HT230 with artificial ageing. The decrease was between 4.6 % (HT190) and 8 % (control samples). The average shear strength of HT230 increased by 20.1 %. Lower strength of aged joints is expected since stresses during exposure to heat and water decrease bond strength (Bengtsson *et al*., 2003, Zhao *et al*., 2011, Todaro *et al.,* 2015).

The average shear strength of the first slice (the first 8 mm of samples) exposed to artificial weathering was lower than the strength of the rest of the adhesive bond. The biggest difference in the case of HT210 is due to the largest cracks in the bond lines. This was to be expected due to cracks occurring in the bond line exposed to swelling and shrinkage. However, high standard deviations show high variability of measurements (Figure 2). The statistical analysis of measurements of aged samples showed that there was no significant dif-

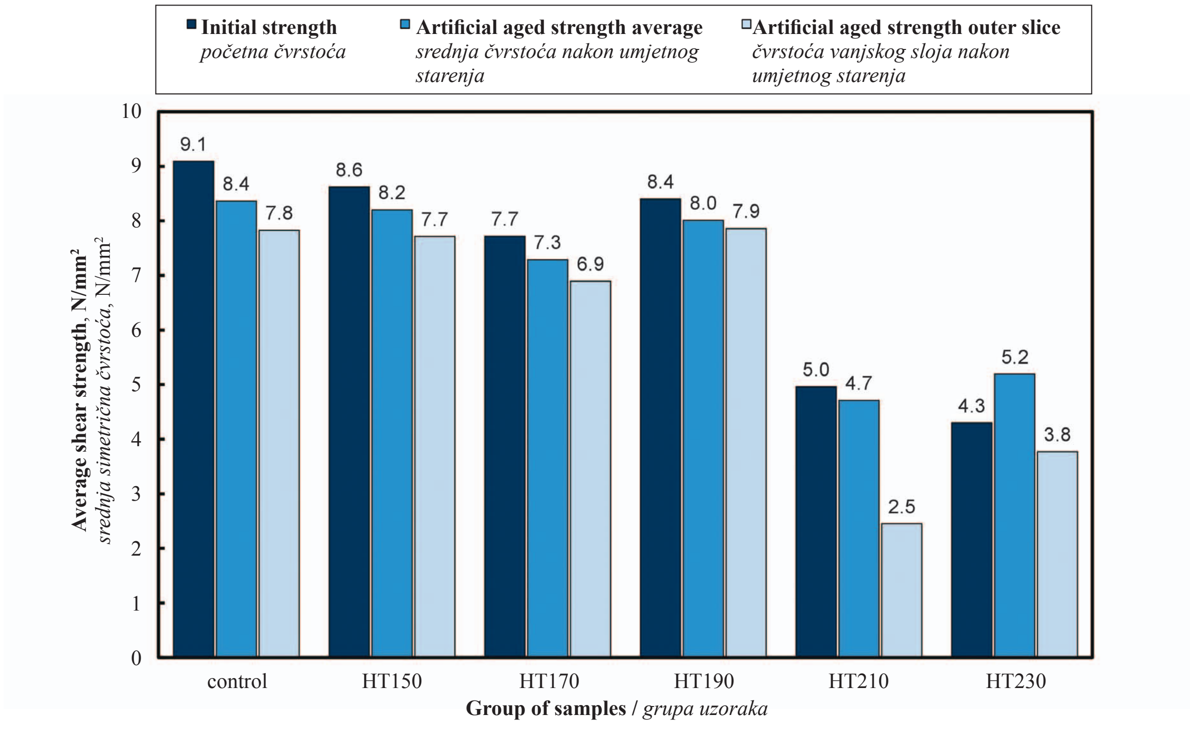

Figure 2 Average shear strength of PVAc bonds before and after artificial ageing
Slika 2. Srednja smična čvrstoća PVAc-om lijepljenog spoja prije i nakon umjetnog starenja

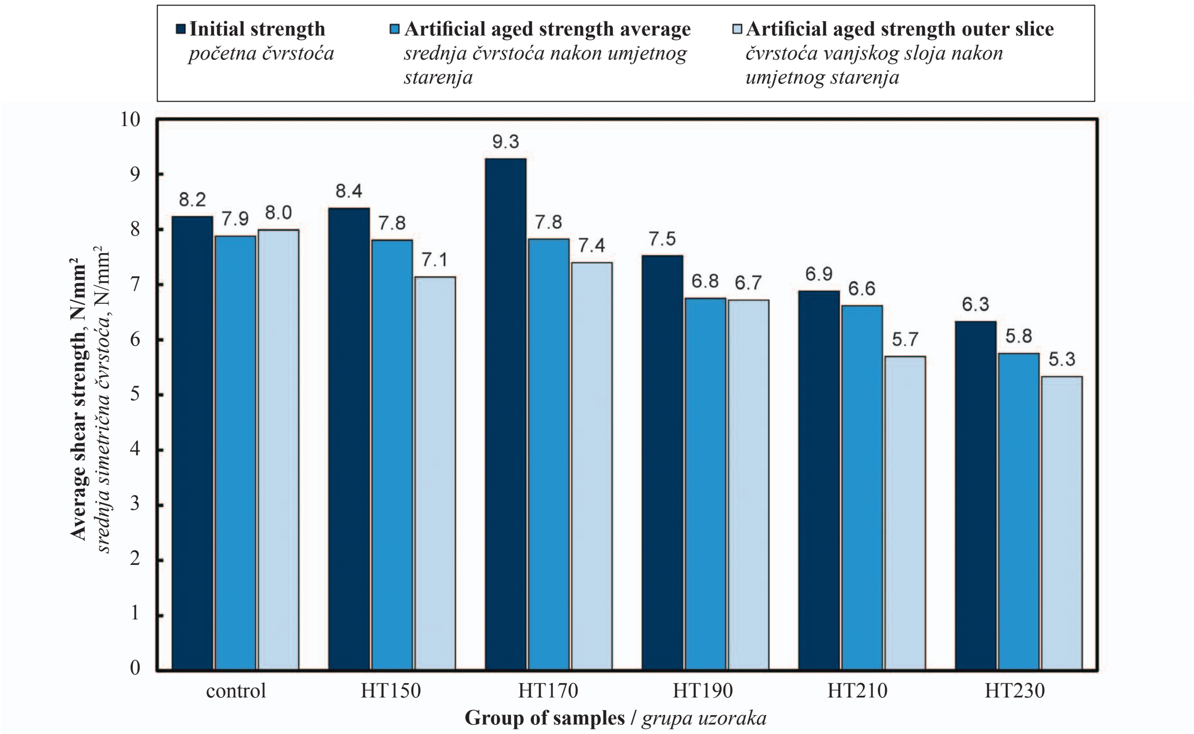

Figure 3 Average shear strength of MUF bonds before and after artificial ageing
Slika 3. Srednja smična čvrstoća MUF-om lijepljenog spoja prije i nakon umjetnog starenja

ference between average strengths between three measured layers (outer slice, middle slice, inner slice). The only exceptions were HT210 samples, where significant difference in strength between the outer and middle slice was observed. Statistical analysis for comparison of strength between the first outer slice and the average strength of the sample only showed significant difference in group HT210 and HT 230.

The average shear strength of MUF adhesive bonds decreased during artificial ageing for all groups of samples (Figure 3). The decrease was between 3.8 % (HT210) and 15.7 % (HT170). The average shear strength of the first slice (exposed to artificial weathering) was lower than the average strength of the rest of the sample (except for the control samples). Statistical analysis showed high variability and significant difference in strength only for HT150 (difference in strength between the outer and inner layer) and HT210 (difference in strength between the outer and middle layer). Statistical analysis for comparison of strength between the first outer slice and the average strength of the sample only showed significant difference in group HT210.

The shear strength of adhesive bonds decreased with artificial ageing (except for PVAc HT230). This decrease was expected, but the actual decrease depends on the type of adhesive used, type of joint and ageing conditions (Follrich *et al*., 2011). The causes of degradation of bond lines can be divided into three categories: environment (climate, temperature, light, fungi, etc.), materials (type of adhesive, wood) and stresses (static loading, stresses from swelling and shrinking) (Gustódio *et al*., 2009), and all can affect the adherend (wood), adhesive or adhesive joint. Since natural ageing and weathering of joints take a long time and are strongly influenced by the macro- and microclimate, several artificial weathering testing procedures are used, making it hard to compare their findings (Follrich *et al*., 2011).

The average percentage of wood failure for MUF adhesive bonds was high and remained high after ageing (Table 4). The selected MUF adhesive is intended for structural joints in glulam, thus a high percentage of wood failure was expected. The average percentage of wood failure for bonds with PVAc adhesive decreased with the temperature of wood thermal modification. Artificially aged samples from the outer layer, made of more severely treated wood (HT210 and HT230), exhibit smaller decrease in wood failure.

4 CONCLUSIONS
4. ZAKLJUČAK

Artificial ageing changed the colour of the exposed surface of wood samples. During ageing cycles, cracks appear in the bond lines and their size increases with the duration of ageing. The time before the bond begins to delaminate depends on the type of adhesive and the degree of thermal modification of wood. In the case of PVAc adhesive, delamination occurred faster than with MUF adhesive. PVAc adhesive is thermoplastic and is softened by temperature, but MUF adhesive has higher temperature and water resistance and this could contribute to better results. The samples from thermally treated wood also started to delaminate before the samples made of untreated wood. Thermally treated wood has a darker colour so that it warmed up during light exposure to higher temperatures than control wood. Higher temperatures on the surface affect

Table 4 Average percentage of wood failure (initial, average after ageing, average after ageing- for outer slice), standard deviations in brackets.
Tablica 4. Srednji postotak loma po drvu (početnog, srednjeg nakon starenja, srednjeg nakon starenja – vanjski sloj); u zagradama su standardne devijacije

		PVAc			MUF		
	Sample *Uzorak*	**Initial WF, %** *Početni WF, %*	**Artificial ageing - average wood failure, %** *Umjetno starenje - srednji lom po drvu, %*	**Artificial ageing -average wood failure-outer slice, %** *Umjetno starenje - srednji lom po drvu - vanjski sloj, %*	**Initial WF, %** *Početni WF, %*	**Artificial ageing - average wood failure, %** *Umjetno starenje - srednji lom po drvu, %*	**Artificial ageing - average wood failure-outer slice, %** *Umjetno starenje - srednji lom po drvu - vanjski sloj, %*
Group of samples *Grupa uzoraka*	Control	100 (0)	100 (0)	100 (0)	100 (0)	100 (0)	100 (0)
	HT150	100 (0)	100 (0)	100 (0)	98 (6)	100 (0)	100 (0)
	HT170	100 (0)	100 (0)	100 (0)	100 (0)	100 (0)	100 (0)
	HT190	99 (3)	98 (7)	100 (0)	98 (6)	100 (0)	100 (0)
	HT210	82 (26)	79 (29)	53 (39)	98 (4)	100 (0)	100 (0)
	HT230	50 (23)	63 (37)	47 (33)	99 (3)	100 (0)	100 (0)

the adhesive, but also mean more drying and shrinking of wood, followed by water shower and cooling and swelling of wood, thus creating stresses on the surface and bonds.

The average shear strength of the joints decreased with increasing the number of ageing cycles (except PVAc HT230), and a small difference in strength between the outer layer and the average of the whole sample was noticed. However, the variability of the results is high and there is no statistically significant difference. Lower measured bond strengths are probably due to several factors: lower strength of heat treated wood, higher MC during testing of aged samples and bond cracks induced by the ageing process.

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Behavioural Aspects of Financial Decision-Making Process of Managers in Wood-Processing Enterprises

Aspekti ponašanja menadžera drvoprerađivačkih poduzeća pri donošenju financijskih odluka

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ABSTRACT • *The study is focused on behavioural aspects in the financial decision-making process of wood-processing enterprises. The main aim was to map this topic and determine the key behavioural factors that lead management to make mistakes. Primary data on this issue were obtained from an empirical survey. The empirical survey was conducted through a questionnaire that contains questions focused on behavioural decision-making aspects. Using statistical methods, three key behavioural factors were determined. By selecting the given behavioural factors, it was established that love, hate, and sadness are the key factors that influence management behaviour and decision-making. In the real business environment, two managers working in a wood processing enterprise were chosen; they were willing to provide us with a review and opinion on the results of the survey. By analysing all the data, it has been concluded that, even though managers are trying to direct their behaviour and activities, they often do not notice the influence of these factors, and sometimes they are unable to make decisions. The managers should be able to direct their behaviour and activities, to provide self-control and take into consideration the fact that these factors are always present. Results determine the key and systematically occurring errors in the financial decision-making process, caused by the influence of the human factor. We have developed a model for activating the three key behavioural factors applied in the financial decision-making process as a tool that can help company managers not to make the wrong decisions.*

Keywords: *finance; behavioural finance; financial decision-making process; wood-processing enterprises; psychological factors*

SAŽETAK • *U ovom su radu prezentirani načini ponašanja menadžera drvoprerađivačkih poduzeća pri donošenju financijskih odluka. Glavni cilj istraživanja bio je mapirati i odrediti ključne čimbenike ponašanja koji navode menadžere na pogrešne odluke. Primarni podatci za ovo istraživanje prikupljeni su empirijskim anketiranjem koje je provedeno upitnikom s pitanjima vezanim za aspekte ponašanja pri donošenju odluka. Primjenom statističkih metoda određena su tri ključna čimbenika ponašanja. Odabirom danih obilježja ponašanja utvrdili smo da su ljubav, mržnja i tuga ključne odrednice koje utječu na menadžere pri donošenju odluka. Izabrana su dva menadžera*

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iz drvoprerađivačkih poduzeća u stvarnom poslovnom okruženju koji su bili voljni dati osvrt i mišljenje o rezultatima anketa. Analizom svih podataka zaključeno je da i oni menadžeri koji su vrlo čvrsti u svojim odlukama i koji pozorno kreiraju svoje ponašanje i aktivnosti najčešće nisu svjesni utjecaja danih čimbenika ponašanja, pa stoga ne mogu donijeti pravu odluku. Svaki bi menadžer trebao pravilno usmjeravati svoje ponašanje i aktivnosti, imati samokontrolu i uzimati u obzir činjenicu da uvijek postoje čimbenici koji utječu na njihove odluke. Rezultati definiraju ključ i pogreške koje se sustavno pojavljuju u procesu donošenja financijskih odluka, a nastaju pod utjecajem ljudskih čimbenika. Kreirali smo model aktiviranja triju ključnih čimbenika ponašanja i procesa donošenja financijskih odluka kao alata za izbjegavanje pogrešnih odluka menadžera.

Ključne riječi: *financije; financijsko ponašanje; proces donošenja financijskih odluka; drvoprerađivačka poduzeća; psihološki čimbenici*

1 INTRODUCTION
1. UVOD

Behavioural economics, currently one of the main economic directions, incorporates psychologically realistic thinking into economic decision-making analyses. In economic decisions, it is not enough to rely solely on numbers and mathematical and statistical methods, but also on memories and intuition in creating estimates of future results. Studies on investment behaviour of a number of authors at different times and places encouraged this new widely accepted inter-disciplinary field of finance: Behavioural Finance (Dervishaj, 2018; Kapoor and Prosad, 2016; Sedaghati, 2016; Shefrin, 2001; Thaler, 2005; Sewell, 2011). Behavioural aspects are not only related to psychology, they are also based on emotional and cognitive perception. The position of a manager brings responsibility, the daily pressure of duties and tasks forces him to act spontaneously and not just think about rational decisions.

Various scholars have studied factors of financial behaviour and their impact on financial decision making (Rehan and Umer, 2017; Omarli, 2017; Shu and Sulaeman, Yeung, 2016; Thaler and Sunstein, 2008; Laibson, 2009). Behavioural financing is topical, and it has begun to develop slowly. The interest of the solved problem stems from the failure to explore the present factors that affect each subject. Therefore, we decided to pay attention to it with the intent to bring some insights into this issue.

The aim of the study was to analyse behavioural factors in the financial decision-making, and consequently to determine the key and systematically occurring errors in the financial decision-making process, caused by the influence of the human factor, as a starting point for preventing wrong financial decisions in the wood-processing industry. Our findings are summarized in the developed model that can eliminate wrong decisions of company managers.

1.1 Theoretical fundamentals
1.1. Teorijske pretpostavke

Traditional finance is based on neoclassical economy. It is assumed that individuals are against risk, have perfect rationality and focus on maximizing the function of personal benefits. Therefore, efficient markets arise. Behavioural financing notes that individuals can sometimes have risky behaviours (FAW, 2018). The traditional theory of corporate finance shares several features with neoclassical financial economics. The manager is focused on maximizing shareholder wealth (Shefrin, 2001). Neoclassical financing theories rely on the skills and motivation of managers themselves, while they behaviourally monitor the behaviour of markets, not just individuals (Ross, 2004). Traditional financing monitors the behaviour of individuals - rational managers, investors and market participants. This leads to markets where prices reflect all available relevant information (Sedliačiková *et al*., 2015).

Behavioural finance recognizes that the way information is presented to participants has a major impact on decision-making and can lead to emotional and cognitive biases. As decisions are not always optimal, this leads to markets that are temporarily or permanently inefficient (FAW, 2018). Proper economic decision-making of individuals is influenced by limited human rationality, social priorities, and a lack of human self-control (Thaler and Sunstein, 2008). Behavioural economics is known as a combination of psychology and economics for understanding finance (Laibson, 2009). Behavioural finance is the result of the structure of three different sciences (Ricciardi and Simon, 2000):

- psychology as a science that analyses the processes of behaviour and thinking, such as the processes of physical, mental and external environment of a person,
- finance as a system of creating and using resources,
- sociology as a science of the social behaviour of a person or a group of people.

Behavioural theories assume that the behaviour of an individual or group is influenced by previous learning. It is about acquiring certain forms of behaviour (Verešová *et al*., 2014). Behavioural finance is associated with psychological and sociological factors that influence the decisions of financial entities (Shefrin, 2001). Sewell (2011) explains behavioural finance as the influence of psychology on the behaviour of financial professionals, who subsequently have an impact on the market itself. Kahneman and Tversky (1979) noted that the behaviour of an individual in theory differs from practice, and classical financial models cannot explain or predict all financial decisions. Economic rationality in behavioural finance is criticized. Behavioural finance became popular after 2002, when Kahneman (2002) won the Nobel Prize in Economics for the behavioural theory, where he integrated knowledge from psychological research in the field of economics that focused on human behaviour and analysis of the human judgment. Behavioural finance is based on research into human and social recognition and the study of emotional tolerance. It seeks to identify and understand economic decisions. Classical financial behaviour is limited by human rationality, behaviourally explaining the psychological effect on financial markets and activities. It claims that financial phenomena are better explained due to the fact that financial market participants are not rational and their decisions are not limited (Bikas *et al*., 2013). Behavioural finance is a type of modern financial theory that states that investors and other interest groups and financial market participants do not behave rationally. There is also the influence of a lot of information on decision-making (Thaler and Sunstein, 2008). Thaler and Sunstein (2008) say that people create financial evaluations using internal calculations and formulas. Not only are people irrational, but this irrationality can sometimes be predicted in advance. Doing business properly and behaving economically depends on people. Behavioural finance is the study of how the phenomenon of psychology affects financial behaviour (Shefrin, 2005). An essential part of behavioural finance is to observe how classical financing and behavioural financing differ in rationality. Decision-making is not only dependent on a careful evaluation of information or statistics, but also on rational decisions. It is a matter of shortening the process of making one's own decision, but only at the expense of less effort in making mathematical or statistical calculations (Lehrer, 2011).

According to the Baker *et al*. (2004), there are two different approaches to finance:

- irrational managers and an efficient/productive market,
- limited rationality - irrational investors and rational managers.

The first approach - irrational managers - focuses on the analysis of the irrational behaviour of managers in the context of an efficient financial market ® excessively optimistic (Fairchild, 2010). Weinstein (1980) describes optimism as people's belief that they are more likely to experience future positive life events and less likely to experience negative situations. In terms of managerial decisions, these decisions play against shareholders/investors. These are intentional and unintentional decisions that reduce value. These are the decisions that intentionally reduce the value of the company, in which managers try to bypass shareholders, and this leads to conflicts that can be resolved by adjusting incentives. However, these decisions do not arise due to different interests between shareholders and managers. These are the consequences of managers' mistakes. These mistakes occur due to psychological aspects and should be preceded by training in the area of management and education (Fairchild, 2010).

The approach of irrational managers is currently less developed. It assumes that the managers' behaviour is defined, but preserves the rationality of investors even if they limit the management mechanisms that they can use to restrict managers. It monitors prejudices of optimism and overconfidence. The simple model shows prejudices of senior managers who believe that their companies are underestimated, encourage overinvestment from internal sources and show preferences for internal and external financing, especially by internal capital. As in the case of investor irrationality, the real economic losses associated with managerial irrationality have yet to be quantified but are significant (Baker *et al*., 2007).

By irrational managerial behaviour, we mean behaviour that deviates from rational expectations and expected maximization of utility value. The manager believes that he is actually close to maximizing the fixed value. In order to have fewer rational managers, corporate management must limit their ability to make rational decisions (Eckbo, 2007).

In regard to the financial policy, an optimistic manager never sells shares unless he has to. If there is an upper limit of the leverage effect (e. g. greater than zero), optimism predicts "accumulation". Financing decisions: the manager relies on internal capital and debt but uses only his own resources. This is his last chance (Baker *et al*., 2007).

The second approach – irrational investors – focuses on situations when investors are systematically irrational and rationally well-informed thanks to managers. This approach assumes that managers will be able to distinguish between price and intrinsic value (Baker *et al*., 2004). According to Graham and Harvey (2001), price represents what investors pay and intrinsic value is what they get. We are talking about the rational goals of managers in irrational markets. Rational managers maintain a balance between the three goals. These are market timing, supply and an increase of the intrinsic value. Market timing refers to decisions aimed at the proper valuation of, for example, the issue of overvalued or repurchased undervalued shares. Supply refers to decisions aimed at rising stock prices above intrinsic value. Increasing intrinsic value is a clear matter - it is about increasing the market value of the company (Fairchild, 2010).

The possibility of learning from mistakes or experience is characteristic for behavioural financing. Managers or other executives are people who may be influenced by their own optimism and overestimation of certain future events. It is well known that self-confidence can lead to mistakes in decision making and overconfident managers are able to underestimate the risk of future results (Baker and Nofsinger, 2010).

The Slovak Republic is relatively independent of importing the natural resources, being built on a domestic resource base of sustainable character. Therefore, it can permanently show an active balance of foreign trade. Regarding the positive situation related to natural resources, their suitable geographic location, and their acceptable energetic demands for processing wood, the wood-processing industry represents an important field of industry for the Slovak national economy, thus enabling further development of small and medium enterprises (Hajdúchová *et al*., 2016). Wood-processing industry (WPI) is composed of the wood, furniture, and pulp and paper industries. These are based on processing wood, i.e. domestic ecological resource (Halaj *et al*., 2018).

The wood-processing industry can positively assess the development of labour productivity. It is the most significant in the pulp and paper industry, where long-term growth above the average of industrial production is recorded, especially in periods with high inflows of foreign direct investment into the mentioned sector (Merková *et al*., 2011). However, the wood-processing industry of Slovakia notes no innovation development focused on the increase of the competitiveness of production and efficiency, and without providing financial resources needed to implement innovative plans, it can expect a significant decrease in the competitiveness and long-term recession (Merková and Drábek, 2010). The share of R&D capacities is gradually reduced, and thus it fails to engage the capacities into innovative projects (Merková *et al*., 2012). The bioeconomy strategy, launched by the European Commission, and the transition to a stronger, circular and low-carbon economy have posed new actions and requirements towards a greater and more sustainable use of natural resources by sustainably increasing the primary production and conversion of waste into value-added products, enhanced production and resource efficiency (Antov *et al*., 2020).

1.2 Objectives and hypotheses
1.2. Ciljevi i hipoteze

The aim of the work was to develop a model of key behavioural factors that cause errors in the financial decision-making of managers in wood-processing enterprises. The essence was to determine the key, systematically occurring errors in the financial decision-making process, caused by the influence of the human factor, as a starting point for preventing wrong financial decisions.

The main hypothesis can be defined as follows: We assume that the model of key behavioural aspects will be suitable for identifying the main causes of differences in decision-making in neoclassical and behavioural financing, and will serve as a starting point for preventing wrong financial decisions.

We have added five partial hypotheses to the main hypothesis, which are as follows:

H_1: We assume that managers do not observe their decision-making behaviour and are unable to proceed with the self-control in thinking.

Richard Thaler (2005) studied the behaviour of people and found out that that people do things that should not happen when making decisions in classical economics. He wanted to find explanations and wrote a study called Dump Stuff People Do. He was also inspired by Kahneman and Tversky (1979), and so he became one of the fathers of behavioural economics (Záborský, 2017).

H_2: We assume that human behaviour is important in evaluating economic decisions.

Adam Smith (2012) claimed this idea and described it in the work Theory of Moral Sentiments. Behavioural financing is the subject of many empirical observations, and the author's effort was to explain the deviations of behavioural from rational decision-making.

H_3: We assume that people are rational and their thinking is reasonable. Emotions such as fear, love or hate are a source of cases when people change their behaviour and deviate from rationality.

Tversky and Kahneman (1974) described systematic errors in the thinking of normal people and at the same time analysed the origin of such errors in the mechanism of cognition. Thus, they found that emotional and psychological factors are a source of change in the behaviour of people, but only when it comes to borderline situations of decision-making and understanding of something unknown. In this case, we can also apply it to the target decision subjects, i. e. managers of enterprises.

H_4: We assume that the key factors that most influence the managers in decision-making are knowledge as a cognitive factor, certainty as psychological and happiness as an emotional factor.

Standard economic theory is designed to offer mathematically set solutions and to perceive man as an economically rational subject. They are based on idealized financial behaviour. Behavioural finance, in turn, attempts to imitate the phenomenon of the human psyche and is based on observed behaviour (Pompian, 2006).

Akerlof and Shiller (2010) argue that behavioural finance has emerged as a new trend in economics that focuses on the economic aspects of deviations from the rational behaviour of subjects, especially the impact of cognitive distortions, psychological and emotional states of the subject. The key factors of human behaviour that influence decision making are:

- Emotional: love, hate, sadness, happiness, helplessness, panic, depression, despair, anxiety.
- Cognitive: knowledge, learning, ability to concentrate attention, ability to know, logical thinking, human character, short-term and long-term memory processes.
- Psychological: power, safety, security, personality, shame, self-esteem, freedom, self-realization, friendship, health, attractiveness.

H_5: We assume that the model of identified key psychological, cognitive and emotional factors will become a tool for correct decision-making of company managers.

The hypothesis can be proved because a comprehensive theoretical analysis followed the issue of behavioural aspects of financial decision-making of company managers and clearly showed that the manager thinks rationally if he does not take into account the state in which he is at the moment of making the decision.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

The schedule in Table 1 presents the methodology of the whole research, describing interconnections and sequence of goals, hypotheses, research methods and ways of testing.

Table 1 Connection and continuity of objectives and hypotheses of the work
Tablica 1. Veze i slijed ciljeva i hipoteza u radu

<table>
<tr><th>Objective
Cilj</th><th>Hypothesis
Hipoteza</th><th>Method
Metoda</th><th>Way of testing
Način testiranja</th></tr>
<tr><td colspan="4">O1: Data collection in the first part of the work is used to examine the current state of the issue at home and abroad with the focus on behavioural aspects of financial decision-making of company managers, concretization of differences between neo-classical and behavioural financing and definition of financial management tasks.
O1: Prikupljanje podataka prvi je dio istraživanja kako bi se ispitalo trenutačno stanje problema u zemlji i inozemstvu, uz usmjerenost na aspekte ponašanja menadžera poduzeća pri donošenju odluka, odredile razlike između neoklasičnog financiranja i financiranja na temelju ponašanja te da bi se definirale zadaće financijskog menadžmenta.</td></tr>
<tr><td rowspan="2">O2: Based on an empirical survey conducted in the form of a questionnaire in the practice of Slovak enterprises, analyse the issue of cognitive, psychological and emotional influences on financial decision-making of managers.
O2: Na osnovi empirijskog istraživanja putem upitnika u slovačkim poduzećima, analizirano je stanje s obzirom na kognitivne, psihološke i emocionalne utjecaje na donošenje financijskih odluka menadžera.</td><td>H_1: We assume that managers do not observe their decision-making behaviour and are unable to proceed with the self-control in thinking.
H_1: Pretpostavlja se da menadžeri ne obraćaju pozornost na svoje ponašanje pri donošenju odluka i da ne mogu zadržati samokontrolu u razmišljanju.</td><td>Questionnaire (question number 10)
upitnik (pitanje broj 10)</td><td>Descriptive and graphical methods
opisne i grafičke metode</td></tr>
<tr><td>H_2: We assume that human behaviour is important in evaluating economic decisions.
H_2: Pretpostavka je da je pri vrednovanju financijskih odluka važno ljudsko ponašanje.</td><td>Questionnaire (question number 11)
upitnik (pitanje broj 11)</td><td>Descriptive and graphical methods
opisne i grafičke metode</td></tr>
<tr><td colspan="4">O3: Thanks to empirical research conducted through Questionnaires, evaluate and examine the effects of behavioural factors on the thinking of company managers and their influence in decision-making through mathematical and statistical methods.
O3: Na osnovi empirijskog istraživanja putem upitnika trebalo je vrednovati i ispitati utjecaje čimbenika ponašanja na način razmišljanja menadžera te putem matematičkih i statističkih metoda utvrditi njihov utjecaj na donošenje odluka.</td></tr>
<tr><td rowspan="2">O4: Identify the key, systematically appearing errors in financial decision-making of managers due to the impacts of human factors.
O4: Identificirati ključne pogreške koje se sustavno pojavljuju pri donošenju financijskih odluka menadžera pod utjecajem ljudskog čimbenika.</td><td>H_3: We assume that people are rational and their thinking is reasonable. Emotions such as fear, love or hate are a source of cases when people change their behaviour and deviate from rationality
H_3: Pretpostavka je da su ljudi racionalni i da razmišljaju razumno. Emocije poput straha, ljubavi ili mržnje razlozi su zbog kojih ljudi mijenjaju način ponašanja i odstupaju od racionalnog postupanja.</td><td>Questionnaire (question number 9)
upitnik (pitanje broj 9)</td><td>Descriptive and graphical methods
opisne i grafičke metode</td></tr>
<tr><td>H_4: We assume that the key factors that most influence the managers in decision-making are knowledge as a cognitive factor, certainty as psychological and happiness as an emotional factor.
H_4: Pretpostavka je da su ključni čimbenici koji utječu na racionalno ponašanje menadžera pri donošenju odluka znanje kao kognitivni čimbenik, sigurnost kao psihološki čimbenik i sreća kao emocionalni čimbenik.</td><td>Questionnaire (question number 7)
upitnik (pitanje broj 7)</td><td>Contingency tables, Pearson’s chi-square test, Cramer´s V and Pearson’s contingency coefficient C
tablica predvidivosti, Pearsonov χ^2-test, Cramerov V- koeficijent i Pearsonov koeficijent predvidivosti C</td></tr>
<tr><td>O5: Develop a comprehensive model of systematically occurring errors in decision-making of company managers, which will serve as a tool for correct decision-making
O5: Kreirati sveobuhvatan model pogrešaka koje se sustavno pojavljuju pri donošenju odluka menadžera, koji će poslužiti kao alat za ispravljanje pogrešaka pri donošenju odluka</td><td>H_5: We assume that the model of identified key psychological, cognitive and emotional factors will become a tool for correct decision-making of company managers.
H_5: Pretpostavka je da će model identificiranih ključnih psiholoških, kognitivnih i emocionalnih čimbenika postati alat za izbjegavanje donošenja pogrešnih odluka menadžera</td><td></td><td></td></tr>
</table>

2.1 Research sample
2.1. Uzorak istraživanja

The basic set was made of 2549 enterprises from the wood-processing industry – according to the classification SK NACE. The enterprises were from the following sectors: 16 – Manufacture of wood and wood products, 17 – Manufacture of paper and paper products; and 31 – Manufacture of furniture. The questionnaire survey was conducted during January and February 2020. We determined the size of the research sample on the basis of a mathematical relation. In our case, the research sample or sample size will represent the minimum number of respondents who were included in the research.

$$n \geq \frac{(z^2 \cdot p \cdot q)}{\Delta^2} \tag{1}$$

The variable *n* in the formula represents the minimum number of respondents; the variable *z* represents the reliability coefficient; the variable *p* and *q* represent the percentage numbers of addressed respondents. These two values express the number of those respondents who understand or do not understand the *issue* (Kozel *et al*., 2006).

The research methodology offers several ways of compiling a sample. We have chosen a purely random selection. The set of respondents had to be divided into two halves. The distribution of the set of respondents is related to the variables *p* and *q*. The product of their values must be maximal (50 %: 50 %). The variable Δ represents a value that means the maximum acceptable error (Kozel *et al*., 2006).

To reach higher reliability of the research, at the level of 95.4 %, we decided to state the value of the variable $z=2$. The value of the maximum error to identify the representativeness of the sample was set to 5 % (Kozel *et al*., 2006).

$$n = \frac{2^2 \cdot 0.5 \cdot 0.5}{0.05^2} = 400 \tag{2}$$

Sample size should be represented by at least 400 respondents (Kozel *et al*., 2006). 403 respondents participated in the questionnaire survey. The total number of addressed respondents was 2549. Thus, the return rate of the questionnaires was 15.81 %, which presented 403 completed questionnaires from the enterprises in the field of wood-processing industry.

2.2 Research questions
2.2. Pitanja u istraživanju

The research questionnaire consisted of 11 questions. This paper presents the research methodology and solution conclusions focused on two main and three complementary questions from the questionnaire. We also defined the types of questions used in the survey, because the type of question is important for choosing the statistical method.

Question 4: the objective was to select subjects in terms of their position within the enterprise. This question became the basis for the compilation of the questionnaire. Question 4 was close-ended, selective, polytomy, with the possibility of choosing one of three variants: manager, employee, owner.

We focused on the structure of the research sample. Question 4 is significant to determine a model of key behavioural aspects in the financial decision-making process. Given that, the work is focused on the issue of behavioural factors from the perspective of managers of wood-processing enterprises. This question helped us to select the subjects and then focus only on them.

Research question 7 was focused on determining the key behavioural factors that have a decisive influence on the change of managers' behaviour and, due to these changes, can also cause wrong decisions.

Question 7: Which of the following cognitive, psychological and emotional factors most influence the rational behaviour of managers in decision-making? Question 7 was close-ended, named, polytomy, with a choice of several variants – behavioural factors.

Three categories of behavioural factors were mentioned in this question - cognitive, psychological and emotional. We have selected a few examples for each category. In this question, it was necessary to follow the degree of dependence between the job position of respondents and the given categories of factors. Thus, individual behavioural factors became the second variable for the statistical determination of dependence.

Complementary questions were the following:

Question 9: In the case of borderline (unknown) decision-making situations, emotions such as fear, love or hate are a source of change in my behaviour.

Question 10: I control my behaviour in decision-making so that self-control is present in thinking.

Question 11: I think that my behaviour is one of the important aspects in explaining economic decisions.

Bipolar Likert scaling method was used in the above questions 9, 10 and 11 and the format of a typical five-level Likert item was applied (Likert, 1932):
- Strongly disagree
- Disagree
- Neither agree nor disagree
- Agree
- Strongly agree

Using statistical methods, we wanted to point out significant dependencies between:
- Job position (question 4 in the first part),
- Behavioural factors (question 7a, 7b, 7c in the second part of the questionnaire).

2.3 Research methods
2.3. Metode istraživanja

The following selected statistical methods were applied in the research:
- two-dimensional classification of the statistical file based on categorical characters - contingency tables,
- dependence of categorical features - Pearson's chi-square test and degree of dependence - Cramer's V and Pearson's contingency coefficient C.

We subjected the obtained primary data to mathematical-statistical analysis, where the results of the questionnaire survey were transformed into a contingency table in the text editor Microsoft Excel. We filtered the question related to the job position and the question about behavioural factors into the table. Using the statistical software STATISTICA 10, we examined the contingency and association of individual items. Pacáková *et al. (2009)* promote the application of Pearson's chi-square test at a significance level of 5 % for contingency and association research, and the application of Cramer´s V and Pearson's contingency coefficient C for associations.

Association presents a dependence between two alternative categorical characters, where contingency is a dependence between two categorical characters, of which at least one character has more than two variations (Pacáková *et al*., 2009). When verifying the dependence/independence of the two qualitative characters, in this case the job position and individual behavioural factors, we had to get the data from the two-dimensional sample set in a contingency table. The association of categorical characters was tested using the Pearson's chi-square test and the degree of dependence using the Cramer´s V and Pearson's contingency coefficient C.

The Pearson's chi-square (c2) test is a universal test for discrete and continuous distribution functions with a sufficiently large range (*n*). Pearson's chi square or good fit test is based on a frequency table. This frequency table tests the null hypothesis (Budíková *et al*., 2010). The comparison of actually found and theoretical numbers is the basic idea of Pearson's chi-square good fit test. The null hypothesis compared to the alternative hypothesis can be verified by a testing characteristic. It is valid (Pacáková *et al*., 2009):

- H_0: there is no dependence between two categorical characters,
- H_1: there is a dependence between two categorical characters (there is contingency).

If the p-value is lower than the significance level of 5 %, we reject H_0 and accept H_1, and we speak of a statistically significant dependence i. e. contingency and association. We consider the statistical significance and the results are applicable. If the p-value is equal to or higher than 5 %, H_0 cannot be rejected. The difference between the observed and expected frequencies is the cause of random selection, and we do not speak about statistical significance between the characters (Pacáková *et al*., 2009).

Cramer's *V* and Pearson´s contingency coefficients *C* indicate the degree of dependence. The coefficient takes values between 0 and 1. The closer it is to 1, the stronger is the dependence between the two characters. On the contrary, the closer it is to 0, the weaker is the dependence (Schmidtová and Vacek, 2013). The significance of the values of the Cramer´s coefficient is defined by a scale: between 0 and 0.1 - negligible dependence, between 0.1 and 0.3 - weak dependence, between 0.3 and 0.7 - medium dependence, between 0.7 and 1 - strong dependence (Pacáková *et al*., 2009).

3 RESULTS
3. REZULTATI

3.1 Findings from statistical analysis
3.1. Rezultati statističke analize

We tested 27 behavioural factors that influence decision making:

- Emotional: love, hate, sadness, happiness, helplessness, panic, depression, despair, anxiety.
- Cognitive: knowledge, skills, ability to concentrate attention, ability to know, logical thinking, human character, short-term and long-term memory processes.
- Psychological: power, safety, security, personality, shame, self-esteem, freedom, self-realization, friendship, health, attractiveness.

Through Pearson's chi-square test, statistical dependences of two variables were found. By selecting individual dependencies, 11 factors that are statistically dependent on the job position were determined. Further on, the degree of dependence was followed through the Cramer´s *V* and Pearson's contingency coefficient *C*. Here we examined how the dependence manifests itself, whether the results are applicable and whether they are statistically significant, and determined dependence between factors. Our selection narrowed down to 4 factors: the ability to concentrate attention, love, hate, sadness. At the same time, it can be stated that these factors and the position of the manager in the company are key factors for changing behaviour. The table of residual frequencies showed that the manager is influenced by these four factors. One factor is from the category of cognitive factors and others from the category of emotional factors (Table 2).

Dependence of job position and influence of behavioural factors on financial decision-making process (O4 and O7a - ability to concentrate attention) (Table 3): We reject the H_0 with the risk of the 5 %-error that there is no dependence between the influence of behavioural factors on the job position and we accept the H_1. The reliability of our decision to reject H_0 is confirmed by the fulfilment of the condition of good approximation. The approximation can be used when missing information makes it impossible to obtain an accurate result. The value of Cramer´s V at the level of 0.5894 represents a medium dependence between the job position of the respondents and the influence of behavioural factors on the financial decision-making process. Furthermore, it can be stated that the ability to concentrate attention influences the behaviour of individual subjects and can also lead to wrong decisions. From the

Table 2 Residual frequencies (ability to concentrate attention)
Tablica 2. Frekvencije osipanja (mogućnost koncentracije)

	Observed minus expected frequencies *Dobivene manje očekivane frekvencije*			
	Manager *Menadžer*	**Owner** *Vlasnik*	**Employee** *Zaposlenik*	**Line (sums)**
0	5.51613	-4.81886	-0.697270	0.00
1	-5.51613	4.81886	0.697270	0.00

Table 3 Dependence O4 and O7a and ability to concentrate attention
Tablica 3. Ovisnost O4 i O7a o mogućnosti koncentracije

Statistics / *Statističke metode*	**Statistics: O4 and O7a Ability to concentrate attention** *Statistika: O4 i O7 s mogućnošću koncentracije*		
	Chi-square / χ^2	***Df***	***p***
Pearson's Chi square / *Pearsonov χ^2-test*	139.9888	2	<0.00001
M-V chi square / *M-V χ^2-test*	41.92583	2	<0.00001
Phi / φ	0.5893782		
Contingency coefficient / *koeficijent predvidivosti*	0.5077514		
Cramer´s *V* / *Cramerov V-test*	0.5893782		

residual frequencies for the respective factor it results that, while the manager is not influenced by the ability to concentrate attention, the owner and employee are.

Dependence of job position and influence of love on financial decision-making process (O4 and O7c): At the significance level of 5 %, H_0 can be rejected even with this factor and the alternative hypothesis H_1 can be accepted. The value of Cramer´s *V* is 0.2114, which represents a weak dependence between the job position of respondents and the influence of love on the financial decision-making process (Table 4). There is also statistical significance between the two variables. Residual values show that, while a manager assumes the effect of love on a change in his behaviour, employees and owners are not influenced by this behavioural factor.

Dependence of job position and influence of hate on financial decision-making process (O4 and O7c): At the level of 5 % significance, H_0 with this factor can also be rejected, where the basis was to keep the condition of good approximation since the value of Pearson's chi-square is 0.02797 (Table 5). In this way, it can be said that there is dependence and thus the influence of hate as an emotional factor affects the job position of the addressed subjects. The important thing is that through residual frequencies, it was established that the manager is influenced by hate in decision-making and that it changes his behaviour, which is not the case with the owner and employee. While the manager is affected by hate, other subjects are not.

Dependence of job position and influence of sadness on financial decision-making process (O4 and O7c): In examining behavioural factors, it was observed that sadness is the last factor that influences the behaviour of subjects in different job positions (Table 6). This factor is one of the emotional behavioural factors. Based on the expected frequencies, it can be concluded that there is also statistical significance between the variables and that the results are applicable (Table 7). It can also be determined that this factor affects the behaviour of a particular subject - the company manager. This can be seen this through the table of residual frequencies. While the manager is affected by sadness, the employee and company owner are not.

To understand the whole research and model construction and the answers to all the hypotheses given in the conclusion, some other results can be added. At the same time, however, we decided to omit the results of other questions with no statistically significant result, where the analysis did not lead to essential conclusions.

Table 4 Dependence O4 and O7c love
Tablica 4. Ovisnost O4 i O7c o ljubavi

Statistics / *Statističke metode*	**Statistics: O4 and O7c love** *Statistika: O4 i O7 s ljubavlju*		
	Chi-square / χ^2	***df***	***p***
Pearson's Chi square / *Pearsonov χ^2-test*	18.01184	2	0.00012
M-V chi square / *M-V χ^2-test*	16.09091	2	0.00032
Phi / φ	0.2114105		
Contingency coefficient / *koeficijent predvidivosti*	0.2068388		
Cramer´s V / *Cramerov V-test*	0.2114105		

Table 5 Dependence O4 and O7c hate
Tablica 5. Ovisnost O4 i O7c o mržnji

Statistics / *Statističke metode*	**Statistics: O4 and O7c hate** *Statistika: O4 i O7 s mržnjom*		
	Chi-square / χ^2	***df***	***p***
Pearson's Chi square / *Pearsonov χ^2-test*	7.152895	2	0.02797
M-V chi square / *M-V χ^2-test*	10.10468	2	0.00639
Phi / φ	0.1332258		
Contingency coefficient / *koeficijent predvidivosti*	0.1320590		
Cramer´s *V* / *Cramerov V-test*	0.1332258		

Table 6 Dependence O4 and O7c sadness
Tablica 6. Ovisnost O4 i O7 o tuzi

Statistics / *Statističke metode*	**Statistics: O4 and O7c sadness** *Statistika: O4 i O7 s tugom*		
	Chi-square / χ^2	***df***	***p***
Pearson's Chi square / *Pearsonov χ^2-test*	15.88575	df=2	0.00036
M-V chi square / *M-V χ^2-test*	14.86026	df=2	0.00059
Phi / φ	0.1985415		
Contingency coefficient / *koeficijent predvidivosti*	0.1947404		
Cramer´s V / *Cramerov V-test*	0.1985415		

Table 7 Residual frequencies (Dependence O4 and O7c: love, hate, sadness)
Tablica 7. Frekvencija osipanja (ovisnost O4 i O7: ljubav, mržnja, tuga)

	Observed minus expected frequencies / *Dobivene manje očekivane frekvencije*			
	Dependence O4 and O7c: love / *Ovisnost O4 i O7: ljubav*			
	Manager / *Menadžer*	Owner / *Vlasnik*	Employee / *Zaposlenik*	Line (sums)
0	-9.38710	5.72953	3.65757	0.00
1	9.38710	-5.72953	-3.65757	0.00
	Dependence O4 and O7c: hate / *Ovisnost O4 i O7: mržnja*			
	Manager / *Menadžer*	Owner / *Vlasnik*	Employee / *Zaposlenik*	Line (sums)
0	-5.93548	3.49380	2.44169	0.00
1	5.93548	-3.49380	-2.44169	0.00
	Dependence O4 and O7c: sadness / *Ovisnost O4 i O7: tuga*			
	Manager / *Menadžer*	Owner / *Vlasnik*	Employee / *Zaposlenik*	Line (sums)
0	-9.22581	6.92556	2.30025	0.00
1	9.22581	-6.92556	-2.30025	0.00

The statement in question 9 argued that, in the case of unknown decision-making situations, emotions such as fear, love or hate are a source of change in behaviour. 58.1 % (219 managers) strongly agree with the statement and 17.2 % (65 managers) agree. It follows that love, fear and hate are the reason for most managers to change their action and behaviour. We have connected this statement with hypothesis H_3. The hypothesis assumed that people are rational and their thinking is reasonable. However, these emotions are a source of cases where they change their behaviour in crucial situations. This hypothesis was partially confirmed by the given statement. In the previous section, where question 7 was statistically evaluated, it can be seen that these factors are a source of change in the behaviour of business managers.

With question 10, we wanted to find out whether managers try to guide their decision-making behaviour through self-control in thinking. Self-control in thinking is a factor showing how to manage a certain burden and effort that comes with decision-making. The manager must try to control himself, to be able to handle situations that are at some point a source of irrational behaviour. Self-control can also be considered as a certain personality characteristic of a person. Descriptive statistics show that 87 % of managers can control themselves and guide their decision-making behaviour. 11.2 % of managers are partially affected, but they agree with this statement. 0.5 % (2 managers), who are clearly unable to guide their behaviour, did not agree. Hypothesis H_1 was linked to this question. We assumed that managers did not monitor their behaviour and decision-making and could not direct their behaviour to self-control. This hypothesis was refuted because managers agreed with the statement.

The statement in question 11, that behaviour is one of the important aspects in explaining economic decisions, is connected to hypothesis H_2. Human behaviour varies depending on several factors. Behavioural factors have been mentioned above. These are largely a source of behaviour change. They can change the expected results and, of course, they are also a source of wrong decisions. Following the results related to the statement, 78.8 % (297 managers) strongly agree with this statement. This percentage of respondents is aware of changes in behaviour as well as of the possibility of deviations and errors that occur in financial decisions.

3.2 Model concept
3.2. Koncept modela

Based on the presented results of mathematical-statistical research, we developed a model for determining the key behavioural factors that cause errors in the financial decision-making process of company managers (Figure 1). Then, we applied the first model to the second model of systematically occurring decision-making errors of managers for the purpose of eliminating wrong decisions. By connecting the two models (presented in Figure 1), we obtained a tool that can eliminate wrong decisions of company managers.

4 DISCUSSION
4. RASPRAVA

By directed statements, we aimed to point out that behavioural aspects can cause significant devia-

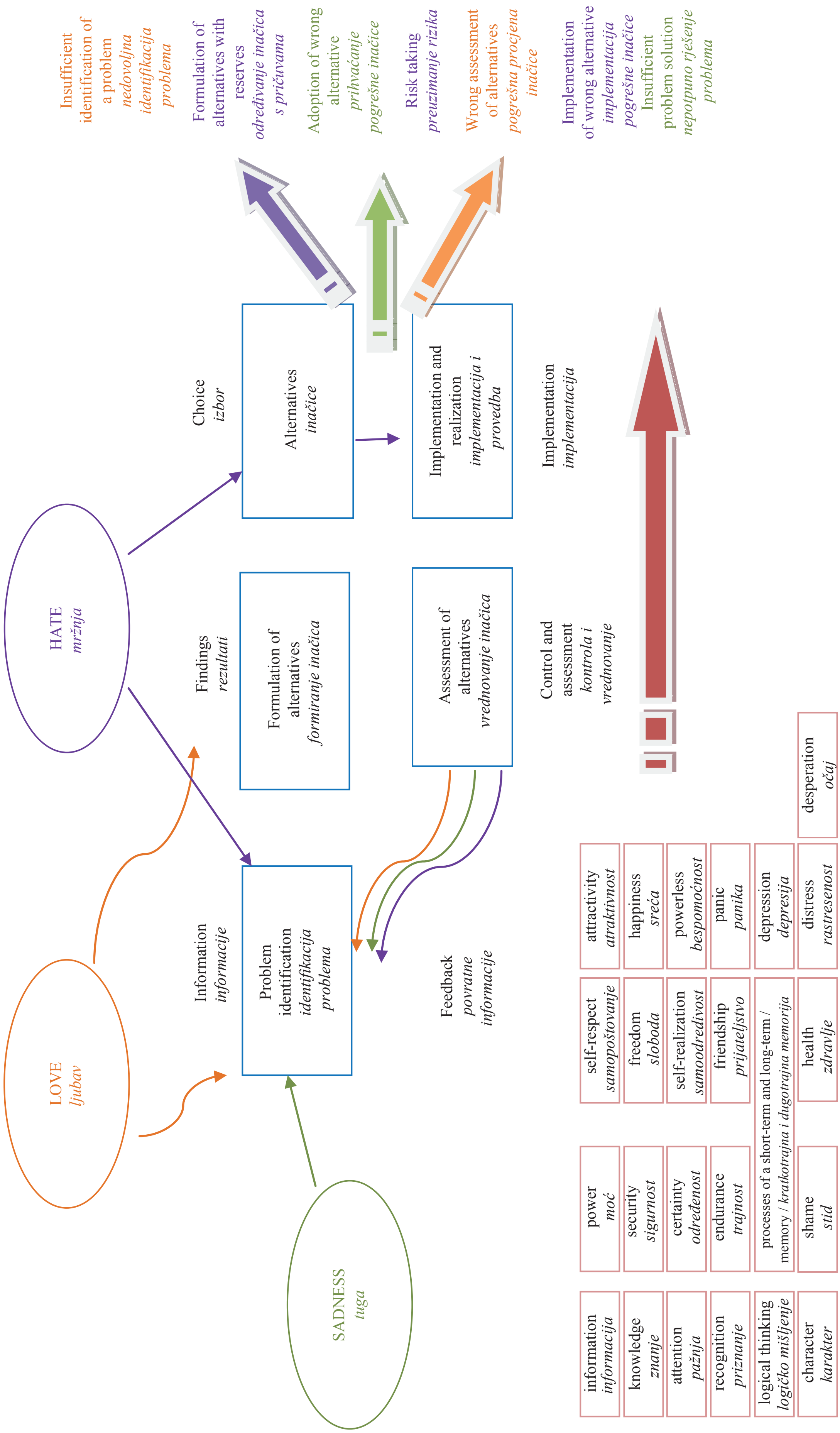

Figure 1 Model of actuation of three key behavioural factors and their application in decision-making process (Source: authors)
Slika 1. Model s tri ključna čimbenika ponašanja i njihov utjecaj pri donošenju odluka (izvor: autori)

tions in the behaviour of managers and thus bring inaccurate to erroneous results in their decisions. Our findings focused on factors of behavioural finance, which are related to investment influence in decision-making in wood processing industry. We have discussed this topic with other authors (Dolan, 2002; Franco and Sanches, 2016; Virlics, 2013), also including developing countries (Asab *et al*., 2014). Baláž (2009) provided many incentives for discussion and questions about the essence, meaningfulness and topicality of behavioural economics. In the second statement, it is clear that intuition influences managers in the decision-making process when considering most of the answers. Nevertheless, if they have enough time to make decisions and are unsure of the outcome, they will rather rely on quantitative and analytical methods. However, the use of one's intuition is not refuted. Over the past several decades, evidence has shown that financial decision-making in the economy at all levels often departs from the predictions of models of rational information-processing (Frydman and Camerer, 2016). More than half of the managers stated that decisions based on their intuition brought profit-maximizing results. Managers also agree that, in the case of clear and well-structured problems, they can omit some steps in the decision-making process. In this case, we can make it clear that managers rely on their intuition. However, it is not clear to what extent and specifically which behavioural factors are the source of change in their behaviour. Due to this fact, we included specific behavioural factors divided into three groups among the statements. Thus each manager had the opportunity to choose the factors that most influence him when making decisions.

Following the previous statement, a question concerning the style of decision-making was included in the second part of the questionnaire. In the context of the sophistication of financial relationships, investment alternatives and risk, the human factor has become increasingly important in investor decision-making (Hitka *et al*., 2019; Lorincová *et al*., 2019; Wang and Ruhe, 2007). More than half of the managers reported a behavioural style of decision-making; however, each of the four options contained an answer. The ninth statement focused specifically on three categories of behavioural factors. It was fear, love and hate. This statement was supported by the hypothesis: Behavioural factors affect business decision making (Merigo, 2015; Tur-Porcar *et al*., 2018). Results of Shu *et al*., (2016) identify bereavement as an underexplored life experience that can significantly influence investors' performance and behaviour. From our research, it can be concluded that love and hate are decisive factors in changing behaviour. This has also been confirmed on the basis of the statistical dependence addressed in question 7.

The last two statements were aimed at manager's self-control in thinking and perception of behaviour. Both statements are linked and the managers are aware of their actions. They agreed that their behaviour influences economic decisions and try to prevent wrong decisions by applying self-control. Functional freedom depends on three compensatory dimensions: it is greatest when the decision-maker is highly rational, when the structure of the decision is highly underdetermined, and when the decision process is strongly based on conscious thought and reflection (Lau and Hiemisch, 2017).

Based on the findings and benefits of the issue, we recommend further examination of behavioural factors and their impact on the financial decision-making process of company managers. As this issue is current and interesting, we think that our results will be applicable to further research in the use of specific behavioural factors and their impact. We believe that our results can influence the perception of managers of wood processing enterprises and their future direction in the area of decision-making. We are convinced that thanks to the implemented empirical research, most managers will try to change their behaviour and think about their decisions.

5 CONCLUSIONS
5. ZAKLJUČAK

Decisions based on hypotheses resulting from the mathematical-statistical research in the wood processing industry are as follows:

We reject hypothesis H_1. Research shows that 87 % of managers are able to direct their behaviour and actions providing self-control in thinking.

Hypothesis H_2 was confirmed. We can assume that managers are aware of their behaviour and perceive the changes resulting from financial decisions.

We can confirm the validity of hypothesis H_3. In the case of unknown decision-making situations, emotions such as fear, love or hate are a source of behavioural change. Two behavioural factors - love and hate, which are the source of change in the decisions of company managers, have been confirmed.

We reject hypothesis H_4. We could determine three key behavioural factors - love, hate and sadness, which affect the financial decision-making process of managers in the wood-processing enterprises.

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Slavica Petrović[1]

The Influence of Heating Degree Days on Fuelwood Consumption in Households in Selected Countries of Central and Southeastern Europe

Utjecaj broja stupanj-dana grijanja na potrošnju ogrjevnog drva u kućanstvima u nekim zemljama srednje i jugoistočne Europe

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ABSTRACT • *Serbia is one of the few European countries that does not keep official statistics and does not have data on heating degree days. A heating degree day (HDD) represents a measure to quantify the energy needs for heating a building. In order to create a database, six meteorological stations in Serbia had been selected, for which the heating degree days were calculated for every year in the period 2010-2018. The months with the highest values of heating degree days were also determined for each year of the analyzed period. In addition to the annual level, heating degree days in the heating seasons over the analyzed period were calculated for the six selected stations, as well as the length and the average air temperature of each heating season. In Serbia, heating season officially lasts from October 15 to April 15. To determine the influence of the calculated annual heating degree days on fuelwood consumption in households in Serbia, over the period 2010-2018, multiple econometric models were formulated. The influence of the annual values of heating degree days on fuelwood consumption for household space heating in Slovenia and Croatia was analyzed, as well. The analysis of energy consumption in the households of the selected countries showed that wood fuels are mostly used for heating, primarily fuelwood. This is the reason why this type of fuel was selected for the research.*

Keywords: *heating degree days; heating season; fuelwood; households; space heating*

SAŽETAK • *Srbija je jedna od malobrojnih europskih zemalja koja ne vodi službenu statistiku i nema podatke o broju stupanj-dana grijanja. Broj stupanj-dana grijanja mjera je koja se primjenjuje za određivanje energije potrebne za grijanje objekta. Kako bi se formirala baza podataka, odabrano je šest meteoroloških postaja u Srbiji za koje je izračunan broj stupanj-dana grijanja za svaku godinu u razdoblju 2010. – 2018. Osim toga, određeni su mjeseci s najvećim brojem stupanj-dana grijanja za svaku godinu analiziranog razdoblja. Osim na godišnjoj razini, izračunan je broj stupanj-dana grijanja u sezonama grijanja tijekom analiziranog razdoblja za šest odabranih*

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postaja, kao i trajanje i srednja temperatura svake sezone grijanja. Sezona grijanja u Srbiji službeno traje od 15. listopada do 15 travnja. Izrađeni su višestruki ekonometrijski modeli kako bi se u kućanstvima u Srbiji u razdoblju 2010. – 2018. utvrdio utjecaj izračunanih godišnjih vrijednosti broja stupanj-dana grijanja na potrošnju ogrjevnog drva. Također je analiziran utjecaj godišnjih vrijednosti broja stupanj-dana grijanja na potrošnju ogrjevnog drva u Sloveniji i Hrvatskoj. Analiza potrošnje energije u kućanstvima u promatranim zemljama pokazala je da se za grijanje najviše iskorištava drvna biomasa, osobito ogrjevno drvo. To je i bio razlog zašto je ta vrsta goriva odabrana za istraživanje.

Ključne riječi: *broj stupanj-dana grijanja; sezona grijanja; ogrjevno drvo; kućanstva; grijanje*

1 INTRODUCTION
1. UVOD

Heating degree days can be explained as a way of climate influence on energy consumption for household space heating. Calculation of heating degree days is based on the height of the daily average air outdoor temperature values and the length of the heating season. Both of these variables depend on the type of climate that is characteristic of a particular area. Heating degree days have been analyzed in a large number of countries around the world (Belova *et al*., 2018; Shen *et al*., 2016; Spinoni *et al*., 2015; Sivak, 2009; Matzarakis *et al*., 2004). In Croatia, this type of research has been conducted for different areas in the country, as well as different time periods (Cvitan *et al*., 2012; Slijepčević *et al*., 2019).

There are three types of climate in Serbia, namely continental, temperate continental and mountain. Continental climate is characteristic of the Pannonian Plain and its peripheral area, up to an altitude of 800 m. This type of climate is characterized by warm summers and severe winters, and warmer autumns than springs (source: Republic Hydrometeorological Service of Serbia, 2020/a). The border between the continental and temperate continental climate traverses the valley of the river Zapadna Morava, then enters the valley of the river Južna Morava, goes over the Leskovac valley and the valley of the river Nišava (Geotesla, 2021). Temperate continental climate prevails in the area south of this border up to an altitude of 1400 m. This type of climate is characterized by moderately warm and dry summers, cold winters and warmer autumns than springs. Mountain climate that prevails at an altitude over 1400 m is characterized by long, severe and cold winters with plenty of snowfall and short and cool summers (Geotesla, 2021). In Croatia, there are also three main climate areas, namely: continental, mountain and maritime. Continental Croatia has a temperate continental climate, while at higher altitudes in the mountainous districts of Gorski kotar, Lika and the Dinaric Alps, mountain climate prevails. Maritime climate is characteristic of the coastal parts of the country, and is characterized by mild winter and warm summers, without extreme temperature differences between the seasons (Croatian Meteorological and Hydrological Service, 2021). Slovenia also has a temperate continental climate in most parts of the country, except in the coastal areas by the sea and in the mountains, where the maritime and mountain climate prevail (Slovenian Environmental Agency, 2021).

In addition to climate, there is a similarity between the countries in the final energy consumption in households. In 2018, households were the largest end-users of final energy in Serbia and Croatia, and the third largest in Slovenia. In households in Serbia 38.5 % of final energy was consumed, 34.4 % in Croatia, and 21.6 % in Slovenia (Eurostat, 2020; Food and Agriculture Organization - FAO and United Nations Development Program - UNDP projects, 2011, author's calculation). Space heating is the major use of energy in households. In 2018, households in Croatia consumed 68.3 % of energy for this purpose, in Slovenia 61.2 %, while there was no data for Serbia.

The main goal of this research was to define the dependency between heating degree days in Belgrade, Novi Sad, Niš, Vranje, Loznica and Zlatibor and fuelwood consumption in households in Serbia. In accordance with the aim, the subject of the research was heating degree days in the six selected meteorological stations.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

Specialized terminology to calculate heating degree days in the six selected meteorological stations in Serbia was used, the main determinants of which are presented below. According to Živković *et al*. (1998), the heating day is the day when the average daily outdoor air temperature is lower than 12.0 °C, and the heating limit temperature (t_{gg}) is the temperature that defines the beginning and end of the heating period. The heating limit temperature is 12.0 °C in Serbia. The heating period represents the number of days, from the beginning to the end of heating, and is also called the heating season (Directive on Energy Efficiency of Buildings, 2011). Calculation of heating degree days was performed based on air temperature of 20.0 °C in the heated room (Directive on Energy Efficiency of Buildings, 2011).

2.1 Calculation of heating degree days
2.1. Izračun broja stupanj-dana grijanja

The heating season in Serbia officially lasts from October 15th of the observed year to April 15th of the following year. However, the heating season can start in the period October 1st-14th of the observed year, if the average daily outdoor air temperature is below 12.0 °C in three consecutive days. Also, the heating season will be extended, if the average daily outdoor air temperature is below 12.0 °C in three consecutive days in the period from April 16th to May 3rd. These rules were followed when calculating heating degree days in Belgrade, Novi Sad, Nis, Vranje and Loznica. In the mountainous areas, on the mountain Zlatibor to be precise, the length of the heating season was determined differently. In accordance with the heating limit temperature, the calculation of the heating degree days included the months of May and September. The heating season in September began when the daily average outdoor air temperature was lower than 12.0 °C in three consecutive days. The end of the heating season was announced in May when the daily average air temperature was equal or higher than 12.0 °C in three consecutive days. If, after three consecutive days with the temperature above 12 °C, the daily average air temperature falls below 12.0 °C again, the heating will be switched on, only if the low temperatures last for three or more days. During the course of the heating seasons, days when the average daily air outdoor temperature was equal to or greater than 12.0 °C were not included in heating degree-days.

According to Živković *et al*. (1998), the calculation of annual (January to April, October to December) heating degree days over the period 2010-2018 was conducted according to the following formula:

$$HDD = Z \cdot (t_u - t_{gg}) + \sum(t_{gg} - t_{sn}) \quad (1)$$

Where: Z – number of days in the month when the difference between the limit heating temperature and the average daily outdoor air temperature was lower than 12.0 °C; t_u – air temperature in heated spaces in residential buildings in the heating season (20.0 °C); t_{gg} – heating limit temperature (12.0 °C); t_{sn} – average daily outside temperature over the heating period.

In Slovenia and Croatia, the same values of the heating temperature limit and the air temperature in heated space in residential buildings as those recorded in Serbia were used to calculate heating degree days (Slovenian Environmental Agency, 2021; Slijepčević *et al*., 2019). Data for heating degree days for the countries in the period 2010-2018 were taken from the Eurostat database.

2.2 Characteristics of the selected meteorological stations in Serbia
2.2. Obilježja odabranih meteoroloških postaja u Srbiji

The selection of meteorological stations for the calculation of heating degree days was carried out by a combination of the following criteria:

- the number of inhabitants of a city where the meteorological station is located;
- climate (in addition to the influence of basic types of climate, special attention is paid to climate subtypes; their influence is characteristic of certain, smaller areas in the country where specific geographical features change the climate making it different from the climate of the environment);
- the district where the meteorological station is located.

According to the first criterion, meteorological stations located in the three largest cities in Serbia, namely Belgrade, Novi Sad and Niš were selected. In Serbia, 23.09 % of the total population lives in Belgrade, 4.75 % in Novi Sad and 3.62 % in Niš (Statistical Office of the Republic of Serbia, 2021). It is assumed that the largest cities have the greatest influence on final energy consumption in households, especially their suburbs where fuelwood is used the most. Based on the second criterion, a meteorological station in Vranje, a city in southern Serbia, was chosen. The modified Mediterranean climate prevails in Vranje, moderately continental in Belgrade and Niš, and in Novi Sad it varies from moderately continental to continental. To consider the influence of the mountain climate on fuelwood consumption in households, a meteorological station on the mountain Zlatibor was chosen. The research conducted during the implementation of the FAO and UNDP projects has shown that households in Western Serbia are large consumers of fuelwood. This was the reason to select the meteorological station located in the city of Loznica that has a temperate continental climate. The six selected meteorological stations are located in different districts in Serbia. There are 29 districts in Serbia, with Belgrade being an independent territorial unit, Novi Sad belonging to the South Bačka District, Niš to the Nišava District, Vranje to the Pčinja District, Zlatibor to the Zlatibor District, and Loznica to the Mačva District.

2.3 Data sources
2.3. Izvori podataka

The calculation of heating degree days for the selected meteorological stations in Serbia was done for the period 2010-2018. There are no data on fuelwood consumption in households in Serbia for the period before 2010. The analysis of fuelwood consumption in households in Serbia was conducted on the basis of data taken from the European Eurostat database, the census of population, households and dwellings in the Republic of Serbia, as well as certain FAO and UNDP projects. The research conducted for the purposes of these projects has shown that the real data values of fuelwood consumption in households in Serbia are much higher than the official data recorded in the Eurostat database. In 2018, according to projects results, energy consumption from fuelwood in households in Serbia was 60.8 % higher than energy consumption from wood fuel in households, as recorded in the Eurostat database. The implementation of the projects provided data on the total consumption of fuelwood in households, whereby it was not divided according to the purpose of consumption. Therefore, the influence of heating degree days on energy consumption from fuelwood for all purposes in households in Serbia was examined. Also, the influence of heating degree days on fuelwood consumption for household space heating in Slovenia and Croatia was examined.

2.4 Econometric modeling
2.4. Ekonometrijsko modeliranje

Regression analysis was conducted to determine the functional dependency of heating degree days on fuelwood consumption in households in Serbia in the period 2010-2018. Multiple econometric models were formulated with four (out of a total of six) independent variables. Durbin-Watson test (DW) of autocorrelation was a limiting parameter for the number of variables in the models, given that the data series had been used for nine years. K-combinations of n elements, without repetition determined the total number of the analyzed models. To this purpose, the following formula was applied (Rakočević, 1983):

$$C_{\mathrm{n}}^{\mathrm{k}} = \binom{n}{k} = \frac{n!}{k!(n-k)!} \tag{2}$$

Where: k – number of meteorological stations in the multiple econometric model (4); n – total number of meteorological stations (6).

In accordance with the applied formula, 15 multiple econometric models were formulated. The choice of the multiple econometric model to determine the analyzed dependency was made according to the highest value of the adjusted correlation coefficient. Single econometric models were formulated to examine the dependency between heating degree days in Croatia and fuelwood consumption for household space heating in the country, as well as in Slovenia. The choice of the single econometric model was made according to the highest coefficient of determination. The statistical significance of the calculated parameter in the model equation was assessed using Student's t-test. Statistical significance of the correlation coefficient was assessed by F-test. In order to determine the autocorrelation in econometric models, Durbin Watson test was used. Econometric models were developed by using software package STATISTICA 7.0.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

This chapter contains the following segments: Heating degree days in selected meteorological stations in Serbia; Heating degree days over heating seasons in Serbia in the period 2010-2018; Energy consumption in household in Serbia; Energy consumption for household space heating in Croatia and Slovenia; The influence of heating degree days on fuelwood consumption in households in Serbia; and The influence of heating degree days on fuelwood consumption for household space heating in Slovenia and Croatia.

3.1 Heating degree days in selected meteorological stations in Serbia
3.1. Broj stupanj-dana grijanja u odabranim meteorološkim postajama u Srbiji

Over the analyzed period in Serbia, Zlatibor had the highest annual value of heating degree days, Belgrade the lowest, while Novi Sad, Loznica, Niš and Vranje had similar values slightly higher than Belgrade (Figure 1).

The highest values of heating degree days for all the analyzed stations were recorded in 2011 (Table 1). Except for Zlatibor, the lowest values of heating degree days for all the analyzed stations were recorded in 2014. The lowest value of heating degree days was recorded in 2018 on Zlatibor. It was a result of extremely warm April and May, which had 9 heating days. In contrast, in the same period in 2014 there was 43 heating days.

Comparing the last and the first year of the analyzed period, all the analyzed stations had the higher heating degree days in 2018 than in 2010. The largest difference between heating degree days of 529.2 was determined for Zlatibor, followed by Novi Sad - 283, Loznica - 291.4, Belgrade - 143.1, Vranje - 69.1 and the lowest for Niš - 31.8. The highest values of heating degree days were recorded in January, February or December. In January, they were recorded in 2010, 2011, 2013, 2015 and 2017, in February 2012, and in December 2014, 2016 and 2018. The highest values of heating

Table 1 The highest and the lowest value of heating degree days in the period 2010-2018
Tablica 1. Najviše i najniže vrijednosti broja stupanj-dana grijanja u razdoblju 2010. – 2018.

Meteorological stations *Meteorološke postaje*	**Heating degree days** *Broj stupanj-dana grijanja*		
	2011	2014	2018
Zlatibor	3,841.1	-	3,260.4
Vranje	3,092.6	2,277.7	-
Novi Sad	2,905.5	2,158.7	-
Niš	2,814.3	2,169.0	-
Loznica	2,784.3	2,108.8	-
Belgrade	2,579.8	1,969.6	-

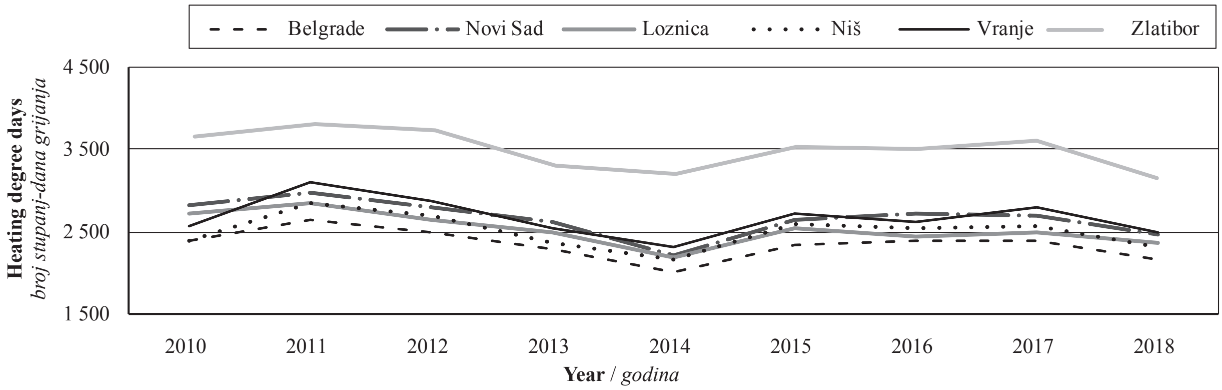

Figure 1 Heating degree days in selected meteorological stations in Serbia in the period 2010-2018
Slika 1. Broj stupanj-dana grijanja u odabranim meteorološkim postajama u Srbiji u razdoblju 2010. – 2018.

degree days were recorded on Zlatibor in January 2017, in February 2012, and December 2016.

3.1.1 Heating degree days over heating seasons in the period 2010-2018
3.1.1. Broj stupanj-dana grijanja tijekom sezone grijanja u razdoblju 2010. – 2018.

The lowest values of heating degree days over heating seasons, over the period 2010-2018, were recorded in Belgrade, and the highest on Zlatibor, while slightly higher values than Belgrade were recorded in Niš, Loznica, Novi Sad and Vranje. The highest values of heating degree days, in all meteorological stations, were recorded in 2011/2012 heating season, and the lowest in 2013/2014. The highest value of heating degree days of 3993.8 was recorded on Zlatibor, followed by Vranje 3191.8, Novi Sad 2970.8, Niš 2905.3, Loznica 2841.9, and Belgrade 2640. In 2013/2014 heating season, the lowest value of heating degree days of 1939.5 was recorded in Belgrade, followed by Niš 2119.4, Loznica 2185.7, Novi Sad 2199.5, Vranje 2293.7 and Zlatibor 3301.6.

The comparative analyses of the heating season length in Belgrade, Novi Sad, Niš, Loznica, and Vranje over the period 2010-2018 showed that it was in the interval between 140 days and 181 days. In Belgrade, Niš and Novi Sad, the longest heating season was 2016/2017, and in Loznica and Vranje 2011/2012. Except in Loznica, where the season 2017/2018 was the shortest heating season, in all the other stations it was the season 2013/2014. Also, 2017/2018 heating season was longer than 2010/2011 only in Belgrade, while in the other stations it was shorter. The shortest heating season over the analyzed period, lasting for 140 days, was recorded in Belgrade. Niš and Loznica have always had longer heating season than Belgrade, as well as Novi Sad and Vranje. The longest heating season in Niš lasted for 169 days, in Loznica 170 days, and in Novi Sad 176 days. The longest heating season of 181 days was recorded in Vranje in 2011/2012. On Zlatibor, the heating seasons have different characteristics compared to the other five analyzed places. The longest heating season of 225 days was 2010/2011, and the shortest of 192 days 2017/2018. Accordingly, over the analyzed period, the heating season length on Zlatibor started to decrease, the largest difference between the heating seasons' length being 33 days. The lowest values of average air temperature during the heating seasons in Novi Sad, Niš, Loznica, Vranje and Zlatibor were recorded in 2011/2012, while in Belgrade it was the case in 2010/2011. The lowest average temperature of 1.07 °C was recorded on Zlatibor, in Vranje slightly higher, 2.36 °C, followed by Niš 2.60 °C, Novi Sad 2.73 °C, Loznica 3.29 °C and Belgrade 3.44 °C. The warmest heating season in Belgrade, Niš, Loznica and Zlatibor was in 2013/2014 and in Novi Sad and Loznica in 2015/2016. Belgrade recorded the highest average air temperature of a heating season of 6.15 °C, followed by Loznica 5.75 °C, Niš 5.58 °C, Novi Sad 5.47 °C, Vranje 5.39 °C, and Zlatibor 4.50 °C (Figure 2).

3.1.2 Energy consumption in households in Serbia
3.1.2. Potrošnja energije u kućanstvima u Srbiji

In 2018, 101.996 TJ of final energy was consumed in households in Serbia (Sources: Eurostat 2020, FAO and UNDP projects, author's calculations). With the exception of 2010, when enormous electricity consumption was recorded, in the remaining years of the analyzed period, the consumption of final energy in households was without major oscillations.

Wood fuels are mostly used for household space heating, followed by fossil fuels and district heating, and, finally, electricity. In the group of wood fuels, fuelwood is mostly used, while the consumption of wood pellets and briquettes is significantly lower. In 2018, energy consumption from fuelwood was higher than in 2010. Unlike fuelwood, energy consumption from fossil fuel for space heating was lower in 2018 compared to 2010. Coal was mostly used, followed by natural gas, and petroleum products as the least used

3.1.3 The influence of heating degree days on fuelwood consumption in households in Serbia
3.1.3. Utjecaj broja stupanj-dana grijanja na potrošnju ogrjevnog drva u kućanstvima u Srbiji

By formulating multiinple econometric models, it was determined that the changes in heating degree days in Novi Sad, Loznica, Zlatibor and Vranje have the greatest influence on fuelwood consumption in households in Serbia. The influence of heating degree days on fuelwood consumption in households in Serbia is best described by a multiple econometric model (the highest value of adjusted correlation coefficient), with Eq. 3:

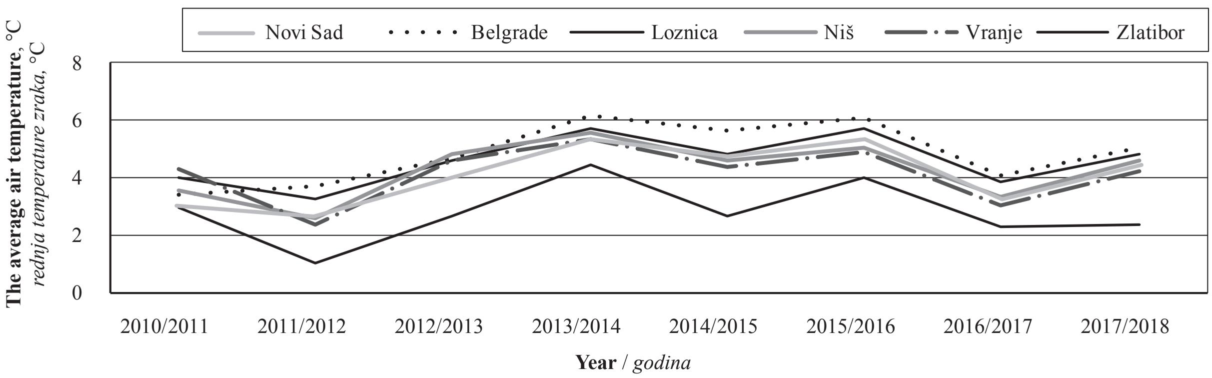

Figure 2 The average air temperature of heating seasons in selected meteorological stations in the period 2010-2018
Slika 2. Srednja temperatura zraka tijekom sezone grijanja u odabranim meteorološkim postajama u razdoblju 2010. – 2018.

Table 2 The values of the basic parameters of the model
Tablica 2. Vrijednosti osnovnih parametara modela

Parameter		**St. error**	***t*-test**	**|*t*-test|**	$F_{(4,4)}$	$F_{test\ (0.05)}$:
a	ln = 12.45253	0.551940	22.56139	$> t_{0.05}$	2.5114	-
b	0.24648	0.148699	1.65755	$< t_{0.05}$		
c	-0.20489	0.13226	-1.54916	$< t_{0.05}$		
d	-0.32017	0.112997	-2.83345	$> t_{0.05}$		
e	0.10211	0.083928	1.21660	$< t_{0.05}$		

$$Y=255896.9 \cdot x_1^{0.24648} \cdot x_2^{-0.20489} \cdot x_3^{-0.32017} \cdot x_4^{0.10211} \quad (3)$$

Where: x_1 - heating degree days in Novi Sad; x_2 - heating degree days in Loznica; x_3 - heating degree days in Zlatibor; x_4 - heating degree days in Vranje.

The calculated value of the correlation coefficient of 0.84 shows a very high correlation between the analyzed variables. In addition, the value of the coefficient of determination shows that 71.5 % of the variations in the fuelwood consumption in households in Serbia may be explained by the changes in heating degree days in Novi Sad, Loznica, Zlatibor and Vranje. The value of DW statistics is 1.72, so it cannot be stated with certainty if the model has an autocorrelation. Since the parameters b, c and e are not significant, larger deviations from the stated value can be expected. The basic parameters of this model are given in Table 2.

The calculated values of individual parameters in the model equation mean that if the variables representing heating degree days in:

- Novi Sad (x_1) and Vranje (x_4) increase by 1.0 %, the fuelwood consumption in households in Serbia will increase by 0.25 % and 0.10 %, and if the variables in
- Loznica (x_2) and Zlatibor (x_3) increase by 1.0 %, the fuelwood consumption in households in Serbia will decrease by 0.20 % and 0.32 %.

The research was based on the assumption that, with the increase in heating degree days in selected meteorological stations, fuelwood consumption in households in Serbia will increase. However, different results were obtained, which showed that the increase in heating degree days in Loznica and Zlatibor causes a decrease in fuelwood consumption in households in Serbia. These results may be explained by the fact that households purchase certain quantities of fuelwood before the start of the heating season. If the heating season is extended and the prepared quantity of wood is consumed, households have two options, either to procure new quantities of fuelwood or to use alternative fuel. When the heating season extends, the demand for energy sources on the local market grows and their price rises accordingly. This rule does not apply to electricity and therefore one part of households uses this energy source as a supplement. Also, some households that use fuelwood for space heating combine it with coal. The price competitiveness of energy source will significantly influence the decision whether to procure additional quantities of wood or coal. Also, it must be taken into account whether fuelwood can be purchased as split or unsplit.

3.2 Energy consumption for household space heating in Slovenia
3.2. Potrošnja energije za grijanje u kućanstvima u Sloveniji

In 2018, energy consumption for household space heating in Slovenia of 27,313.4 TJ was 28.8 % lower than in 2010 (Statistical Office of the Republic of Slovenia, 2020).

Wood fuels are mostly used for space heating, followed by fossil fuels, significantly less district heating and electricity, and geothermal and solar energy as the least used. Fuelwood is mostly used for household space heating, followed by wood pellets, while consumption of wood chips, briquettes and the consumption residues is much lower. In 2018, the consumption of fuelwood was lower than in 2010, unlike the consumption of wood pellets and the consumption of wood chips, briquettes and residues. Also in 2018, the consumption of fossil fuels was lower than in 2010. Extra light oil and LPG are mostly used for space heating, followed by natural gas, while coal consumption is symbolic.

3.2.1 The influence of heating degree days on fuelwood consumption for household space heating in Slovenia
3.2.1. Utjecaj broja-stupanj dana grijanja na potrošnju ogrjevnog drva za grijanje kućanstava u Sloveniji

Over the period 2010-2018, heating degree days in Slovenia dropped from a record 3136.59 to 2579.61. The lowest value of 2345.72 was recorded in 2014 (Eurostat, 2021). In Slovenia, heating degree days are specially defined for 13 areas. The highest value of 3457.3 was recorded in 2010 in the area of Gorenjska and the lowest of 1840.74 in the area of Obalno-kraska in 2014.

The influence of the heating degree days on fuelwood consumption for household space heating in Slovenia A is best described by a simple econometric model (the highest value of coefficient of determination), with Eq. 4:

$$Y = 5.64 \cdot x^{1.02} \quad (4)$$

Positive parameter b and its statistical significance show that, with the increase in the heating degree days in Slovenia of 1.0 %, an increase in fuelwood consumption for household space heating of 1.02 % can be expected. The basic parameters of this model are given in Table 3.

The value of the correlation coefficient of 0.84 is quite high and it is statistically significant, so it means

Table 3 The basic parameters of the econometric mode
Tablica 3. Vrijednosti osnovnih parametara ekonometrijskog modela

Parameter		S	t-test	|t-test|	$F_{(1,7)}$	$F_{test (0.05)}$:
a	ln= 1.730259	1,979258	0,874195	$< t_{0.05}$	16,701	+
b	1.020935	0,249821	4,086658	$> t_{0.05}$		

Table 4 The values of other model parameters
Tablica 4. Vrijednosti ostalih parametara modela

Parameter		S	t-test	|t-test|	$F_{(1,7)}$	$F_{test (0.05)}$:
a	ln=6.172837	0.879585	7.017897	$> t_{0.05}$	26.646	+
b	0.587916	0.113894	5.161970	$> t_{0.05}$		

that there is a very high correlation between the consumption of fuelwood for household space heating in Slovenia and heating degree days. The value of coefficient of determination shows that 70.5 % of the variation in fuelwood consumption for household space heating in Slovenia can be explained by the change in heating degree days. The value of DW statistics is 0.99, which means that it cannot be claimed with certainty whether the model has an autocorrelation.

3.3 Energy consumption for household space heating in Croatia
3.3. Potrošnja energije za grijanje kućanstava u Hrvatskoj

In 2018, energy consumption for household space heating in Croatia was at the level of 65.745 TJ, or 18.1 % lower than in 2010 (Eurostat, 2021). This means that in the period 2010-2018, consumption decreased at an average annual rate of 2.5 %.

Wood fuels are mostly used for household space heating, namely fuelwood, while the use of wood pellets and briquettes is significantly lower. Fossil fuels are the second most important energy source for household space heating, but their consumption decreased significantly over the period 2010-2018. Energy consumption from coal decreased the most, by 62.5 %, followed by petroleum products by 52.2 % and natural gas by 21.8 % (Eurostat, 2021).

3.3.1 The influence of heating degree days on fuelwood consumption for household space heating in Croatia
3.3.1. Utjecaj broja stupanj-dana grijanja na potrošnju ogrjevnog drva za grijanje kućanstava u Hrvatskoj

In Croatia, over the period 2010-2018, heating degree days decreased from a maximum of 2,521.22, to 2,076.47. The minimum value of 1,885.28 was recorded in 2014 (Eurostat, 2021). Official data for heating degree days are defined for 23 areas. The highest value of heating degree days was recorded in 2010 in the Lika-Senj area (3,040) and the lowest in 2014 in the amount of 1293 in the Split-Dalmatia area. Zagreb, the capital of Croatia, had a record value of heating degree days of 2715 in 2010, and 2216 in 2018. The lowest value of 1,957 was recorded in 2014 (Eurostat, 2021).

The influence of the heating degree days on fuelwood consumption for household space heating in Croatia is best described by a simple econometric model (the highest value of coefficient of determination), with Eq. 5:

$$Y = 1116.44 \cdot x^{0.59} \quad (5)$$

The positive parameter b and its statistical significance show that, with an increase in heating degree days in Croatia of 1.0 %, an increase of fuelwood consumption for household space heating of 0.59 % can be expected. The value of correlation coefficient of 0.89 shows that there is an extremely high correlation between the analyzed variables, while the value of coefficient of determination shows that 79 % of the variation in fuelwood consumption for household space heating can be explained by the change in heating degree days. Other model parameters have the values from Table 4.

4 CONCLUSIONS
4. ZAKLJUČAK

By formulating multiple econometric models, it was determined that the changes in heating degree days in Novi Sad, Loznica, Zlatibor and Vranje have the greatest influence on fuelwood consumption in households in Serbia. The model equation defines that with the increase in heating degree days in Novi Sad and Vranje, an increase in consumption of firewood in households of 0.25 % and 0.10 % can be expected. Similarly, with the increase in heating degree days in Loznica and Zlatibor, a decrease in fuelwood consumption of 0.20 % and 0.32 % can be expected. By simple econometric models for Slovenia and Croatia, it has been proved that, if the heating degree days in these countries increase by 1.0 %, an increase in fuelwood consumption for household space heating of 1.02 % and 0.59 % can be expected in Slovenia and Croatia, respectively.

In the coming period, it is necessary to expand the research and create database for heating degree days in all districts in Serbia. Creating a database is the first step for all subsequent research in this field. The econometric models have shown the initial dependency of firewood consumption in households in Serbia, Croatia and Slovenia on the heating degree days. Certainly, other factors affect the firewood consumption in households and they must be the subject of further research.

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Determination of Ideal Cooking Conditions for Pulp Production from Avocado Wood (*Persea americana* Mill.) by Kraft Method

Određivanje idealnih uvjeta kuhanja u proizvodnji pulpe od drva avokada (*Persea americana* Mill.) primjenom kraft metode

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ABSTRACT • *In this study, hand sheets were made from pulp produced by the Kraft method using avocado wood. The raw materials were supplied by a fruit orchard and consisted of avocado (Persea americana Mill.) trees that had completed their useful life and were cut during routine thinning maintenance. In order to determine the ideal cooking conditions in the production of pulp from avocado wood via the Kraft method, 16 cooks were carried out by varying the cooking time (T), active alkali (AA), and sulfidity (S) ratios. The general pulp properties, especially the screened pulp yield, pulp viscosity, and Kappa number, were evaluated. The pulp yield was taken as the primary basis in determining the cooking conditions. The ideal cooking conditions were also determined by considering some physical, mechanical, and optical properties of the paper. According to this study, the conditions found to be ideal in pulp production from avocado (Persea americana Mill.) wood via the Kraft method were: 18 % AA, 22 % S, and 75 min T.*

Keywords: *avocado wood (Persea americana Mill.); Kraft method; pulp/paper production; pulp/paper properties*

SAŽETAK • *U ovom su istraživanju izrađeni eksperimentalni papiri od pulpe dobivene sulfatnim (kraft) postupkom od drva avokada. Kao sirovina za proizvodnju pulpe odabrano je drvo avokada (Persea americana Mill.) iz plantažnog uzgoja, posječeno tijekom rutinskog prorjeđivanja nakon završetka životnog vijeka stabla. Radi utvrđivanja idealnih uvjeta kuhanja u proizvodnji pulpe od drva avokada kraft metodom, provedeno je 16 kuhanja, pri čemu su varirani omjeri vremena kuhanja (T), dodatka lužine (AA) i sulfidnost (S). Utvrđena su opća svojstva dobivene pulpe, uz naglasak na prinos prosijane pulpe, njezinu viskoznost i vrijednost kappa broja. Prinos pulpe pritom je uzet kao primarni podatak za određivanje uvjeta kuhanja. Idealni uvjeti kuhanja također su utvrđeni uključivanjem nekih fizikalnih, mehaničkih i optičkih svojstava papira. Prema ovom istraživanju, idealni uvjeti za proizvodnju pulpe od drva avokada (Persea americana Mill.) kraft metodom jesu: AA = 18 %, S = 22 % i T = 75 min.*

Ključne riječi: *drvo avokada (Persea americana Mill.); kraft metoda; proizvodnja pulpe/papira; svojstva pulpe/papira*

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1 INTRODUCTION
1. UVOD

Many types of trees, bushes, and vines are cultivated for fruit production around the world. Although their useful lives and productive periods vary according to the species, some trees require frequent pruning and others require early replacement because of their short span of productivity. For whatever reason, at certain periods a significant biomass supply is available from orchards. Using this potential in the forest industry reduces the pressure on the essential forest trees. Therefore, the issue of utilizing fruit tree wood has attracted the attention of researchers working in the forest industry. For example, in a study conducted in Poland, it was stated that if the residues remaining from the maintenance pruning carried out every year in a 100-ha apple orchard were burned and converted into energy, the net present value of the total income obtained in 10 years would be € 5644 (Arkadiusz *et al*., 2020). In a study conducted in Turkey, the physical and mechanical properties of the trunk wood of date-plum, olive, and loquat trees were examined and it was reported that they could be used in woodturning, carving, and toy production and in the production of massive furniture elements that require high resistance and durability (Topaloğlu and Ustaömer, 2020). A number of studies have investigated the suitability of fruit trees for pulp production. Included among those evaluated have been pomegranate prunings (Gülsoy *et al*., 2015), white mulberry (Gençer *et al*., 2013), common hazelnut (Gençer and Özgül, 2015, 2016), kiwi (Gençer, 2015), wild cherry (Gençer and Gül Türkmen, 2016), wild dogwood (Gençer and Aksoy, 2017), apricot (Gençer *et al*., 2018), date-plum, olive, and loquat (Topaloğlu *et al*., 2019). Most of these types were found to be suitable for pulp production, whereas some were not. For example, Topaloğlu *et al*. (2019) suggested that olive trunk wood was suitable for pulp production, but date-plum and loquat wood should be put to other uses. Similarly, it was suggested that apricot wood was suitable for pulp production, but fruit pericarp should be used in the board industry (Gençer *et al*., 2018). It has been stated that the wood of the olive (*Olea europaea* L.) and fig (*Ficus carica*) trees is similar to that of broad-leaved trees in terms of their chemical components (Odabaş Serin and Kılıç Penezoğlu, 2020a, 2020b). It has been reported that the pulp yields obtained from orange tree (González *et al*., 2011) and kiwi pruning residues (Gençer, 2015) can be used to manufacture paper pulp with satisfactory yields.

In a study conducted on avocado wood, branches were found to have significant biomass potential, but without treatment, it is weak in an outdoor environment because certain of its mechanical and physical properties are low. However, it was recommended for furniture production and indoor use (Fuentes-Talavera *et al*., 2011). The specific gravity of avocado branch wood was found to be (air dry) 0.54 g cm^{-3} at 12 % moisture content and the density to vary from low to medium (Fuentes-Talavera *et al*., 2011). This type of wood, of medium density and with flexible fibers (Kırcı, 2000), can be utilized in pulp production. Runkel ratio shows the collapsibility of the fibers. Collapsed fibers have high fiber bonding area. Therefore, fibers having Runkel ratio of 1 or less are good for papermaking. Runkel ratio of *Persea americana* was 0.95 (Ajuziogu *et al.,* 2010). In addition, it was stated that avocado wood is suitable for pulp production using chemical methods and that the resulting pulp is easy to bleach (Vargas *et al*., 2006). These sample studies demonstrate that it is possible to obtain the maximum benefit from a raw material by carrying out research to find its correct application areas.

Avocado (*Persea americana* Mill.) belongs to the Lauraceae family. In well-maintained orchards where it is grown, a height of 10-15 m and diameter of 40-60 cm are considered ideal in terms of fruit yield. In mixed stands in its natural environment, its height can reach 25-30 m. Although this species is native to Mexico and Central America, some species are commonly grown in many countries having a Mediterranean climate. In our country, avocado is produced in significant amounts in the Mediterranean Region, including the provinces of Antalya, Mersin, and Muğla (Demirkol, 2001; Bayram *et al*., 2006). The Fuerte variety of avocado (*Persea americana* Mill.) is grown in the Gazipaşa district of Antalya Province and trees past the age of useful fruit production were used as the wood raw material in the study. The Kraft method has been reported to be suitable for all types of raw materials in pulp production (Rydholm, 1965). Based on this literature review, the aim of this study was to produce pulp from avocado wood using the Kraft method.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

The avocado (*Persea americana* Mill.) trunk used in this study was obtained from an orchard at an altitude of 4 to 5 m from the sea in Gazipaşa District of Antalya Province, Turkey. The study used trunk wood of 15-year-old Fuerte species trees that the grower had decided to cut during thinning maintenance. Wood discs (10 cm thickness) were taken from bottom, middle, and top of the trunk. These discs were debarked and subdivided into two discs (5 cm thickness). The wood samples were chipped manually for pulping. The chip size was about 50 mm × 15 mm × 5 mm (length × width × thickness). The chemical composition and fiber morphology of avocado wood were determined in our previous study (Altunışık Bülbül and Gençer, 2021). The holocellulose, α-cellulose, klason lignin, hot water solubility, cold water solubility, 1 % NaOH solubility, and ethanol solubility of avocado wood were 73.29 %, 55.05 %, 14.85 %, 2.64 %, 1.51 %, 19.75 %, and 4.50 %, respectively. Fiber length, fiber width, lumen width, cell wall thickness, slenderness ratio, flexibility ratio, and Runkel ratio of avocado wood were 1.06 mm, 25.78 μm, 16.18 μm, 4.80 μm, 41.0, 63.0, and 0.59, respectively.

2.2 Methods
2.2. Metode

By changing the cooking time (T), active alkali (AA) ratio, and sulfidity (S), a total of 16 cooks divided into four groups (A, B, C, D) were completed via the Kraft method using the avocado wood. The cooking time was 75 min for the groups A and C and 90 min for the groups B and D. The active alkali was used in two different ratios: 18 % for the groups A and B and 20 % for the groups C and D. In each group, four different cooks were carried out by changing the sulfidity rate (20 %, 22 %, 24 %, and 26 %). The cooking temperature of softwood chips should be 170°C and above for pulp production using the Kraft method. However, as there is less lignin in hardwood, 160-170 °C is ideal (Rydholm, 1965). Therefore, using the Kraft method, pulp was produced from the avocado wood at a cooking temperature of 165 °C for 90 min.

Avocado wood has been reported to be more easily cooked than other hardwoods such as eucalyptus (Vargas *et al*., 2006). Based on this literature review, the targeted cooking temperature was set as 165 °C. In addition, the study investigated the effects on the yield of reducing the cooking time to 75 min. Pulp production was carried out in an electrically heated laboratory-type cooking vessel at 165 ± 3 °C, with a pressure of 25 kg cm^{-2}, a capacity of 15 L, and at 2 cycles per minute. It was stated that no significant change in pulp yield would occur in the range of 150-170 °C at the least, provided that a regular preheating time and temperature were maintained (Rydholm, 1965). For this reason, the time to reach the maximum temperature in all cooks was kept constant (60 min) and care was taken to keep the vessel temperature below 150 °C until the fiftieth minute of the preheating time. The period for reaching the maximum temperature began after the fiftieth minute, and the maximum temperature was reached in the 60th minute. The pulp-cooking plan for the avocado (*Persea americana* Mill.) wood using the Kraft method is outlined in Table 1.

The total yield was calculated from the pulp washed after the cooking processes listed in Table 1. The fibers released in the mixer were screened in a Somerville vibratory vacuum sieve according to TAPPI T 275 sp-02 (2002) standard and after the residue was separated, the screened pulp yield was calculated according to TAPPI 412 om-02 (2002) standard. Screened pulps were beaten up to (36±4) SR° in a Hollander beater according to TAPPI T200 sp-01 (2001) standard. The degree of freedom of the pulps was determined on a Schopper-Riegler device according to the ISO 5267-1:2012 standard. Values for the Kappa number and viscosity were determined according to TAPPI T 236 om-06 (2006) and SCAN-CM 15-62 (1962) standards, respectively. Ten handsheets of (75±2) g/m^2 in weight were produced from these pulps according to ISO 5269-2:2013 standard.

After conditioning the handsheets at (23±2) °C and (50±2) % relative humidity for 24 h in accordance with TAPPI T 402 sp-03 (2003) standard, they were evaluated to determine opacity TAPPI T 519 om-02 (2002), brightness TAPPI T 525 om-02 (2002), tear index TAPPI T 414 om-98 (1998), tensile index TAPPI T 494 om-01 (2001), and burst index TAPPI T 403 om-02 (2002) according to relevant standards. Elongation and TEA values were also determined while performing the tensile test.

Table 1 Kraft method pulp cooking plan for avocado wood *Persea americana* Mill.)
Tablica 1. Plan kuhanja pulpe od drva avokada (*Persea americana* Mill.) prema kraft metodi

Cook group No. *Br. kuhane skupine*	**Active alkali, %** *Aktivna lužina,* %	**Sulfidity, %** *Sulfidnost,* %	**Cooking time at max. temperature, min** *Vrijeme kuhanja pri najvišoj temperaturi,* min
A1	18	20	75
A2	18	22	75
A3	18	24	75
A4	18	26	75
B1	18	20	90
B2	18	22	90
B3	18	24	90
B4	18	26	90
C1	20	20	75
C2	20	22	75
C3	20	24	75
C4	20	26	75
D1	20	20	90
D2	20	22	90
D3	20	24	90
D4	20	26	90

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

Some of the obtained pulp properties, the brightness and opacity values of the paper produced from these pulps, are given in Table 2.

Dissolution and degradation of carbohydrates develops independently of sulfidity in the cooking solution. Therefore, there was no significant change in viscosity with the increase of sulfidity within the groups. However, the concentration of sodium hydroxide is highly related to cooking time and temperature. In this study, in the cooking of groups where the active alkali ratio was increased (A and C), the decrease in viscosity was more effective than the increase in processing time. Here, the ineffectiveness of the time period was due to the low cooking temperature. However, it is certain that at high temperatures, extension of the time period would have increased the alkali degradation. In this case, a low alkali rate was more advantageous. Alkaline degradation of hemicelluloses, which is more important for their strength properties, is not necessarily parallel to cellulose degradation, but it is likely to develop slightly later. The effect of removing lignin in

Table 2 Some properties of avocado (*Persea americana* Mill.) wood Kraft pulps and brightness and opacity values of the paper produced from them
Tablica 2. Neka svojstva kraft pulpe od drva avokada (*Persea americana* Mill.) i vrijednosti svjetline i neprozirnosti papira proizvedenoga od nje

Groups-AA, % -*T*, min[a] *Grupe - AA, % - T,* min[a]	**Cook No.- Sulfidity, %** *Br. kuhanja - sulfidnost*, %	**Screened yield, %** *Prinos prosijavanja, %*	**Reject, %** *Škart*, %	**Viscosity, cm³/g¹** *Viskoznost,* cm³/g	**Kappa No.** *Kappa broj*	**ISO Brightness, %** *ISO svjetlina,* %	**ISO Opacity, %** *ISO neprozirnost, %*
A-18-75	A1-20	45.46	0.25	1418	25.91	19.84	99.78
	A2-22	47.25	0.33	1413	24.81	19.35	99.82
	A3-24	47.21	0.21	1413	23.50	19.01	99.87
	A4-26	46.73	0.31	1410	22.30	18.52	99.92
B-18-90	B1-20	45.06	0.23	1413	18.84	18.44	99.92
	B2-22	45.46	0.17	1405	18.46	18.42	99.93
	B3-24	46.70	0.08	1405	18.40	17.78	99.93
	B4-26	46.33	0.05	1400	18.32	17.56	99.96
C-20-75	C1-20	46.39	0.18	1356	20.05	18.88	99.86
	C2-22	46.86	0.03	1356	19.97	18.71	99.92
	C3-24	46.30	0.03	1350	19.46	17.72	99.92
	C4-26	44.97	0.05	1345	18.98	17.64	99.96
D-20-90	D1-20	43.11	0.03	1345	18.79	18.52	99.87
	D2-22	44.28	0.16	1355	18.02	18.18	99.89
	D3-24	45.85	0.14	1350	18.42	17.51	99.90
	D4-26	45.93	0.08	1286	18.92	17.31	99.90

[a]AA – Active alkali; T – Cooking time at maximum temperature / *AA – aktivna lužina; T – vrijeme kuhanja na najvišoj temperaturi*

unbleached Kraft pulps is to reduce the strength properties, even at high viscosity (Rydholm, 1965). Although the viscosity was high in group A, low tensile strength resulted because the lignin remaining in the pulp impeded the inter-fiber bonds. A high dose of active alkali at a constant cooking time and temperature greatly lowers viscosity, whereas changes in sulfidity have only a minor effect. This is because of the absence of a significant change in viscosity within the groups at a constant active alkali and cooking time.

Opacity is important for writing and printing paper and is advantageous because paper with high opacity can be used on both sides. The opacity value increased in the groups with a long cooking time and with the increase of sulfidity within the groups. Rydholm (1965) stated that high opacity was especially important in paper produced for printing. Most bleachable sulfate pulps are cooked to 14-18 Kappa numbers. Pulps obtained from deciduous wood can be bleached to brightness values of up to 70 without decreasing the

Table 3 Some properties of paper from avocado (*Persea americana* Mill.) wood kraft pulps according to 36 SR°
Tablica 3. Neka svojstva papira od kraft pulpe drva avokada (*Persea americana* Mill.) prema 36 SR°

Groups-AA, % -*T*, min* *Grupe - AA, % - T,* min*	**Cook No.- Sulfidity, %** *Br. kuhanja - sulfidnost*, %	**Tensile index, N·m/g** *Vlačni indeks,* N·m/g	**Elongation, %** *Produljenje, %*	**TEA**[b]**, j/m²**	**Tear index, m·N·m²/g** *Indeks kidanja,* m·N·m²/g	**Burst index, kPa·m²/g** *Indeks prskanja,* kPa·m²/g
A-18-75	A1-20	60.10	2.25	62.95	3.28	2.94
	A2-22	69.30	2.22	83.95	3.94	2.51
	A3-24	67.27	2.45	93.51	2.85	2.83
	A4-26	68.66	2.35	86.60	3.38	2.49
B-18-90	B1-20	70.95	2.36	92.08	3.28	2.50
	B2-22	73.75	2.47	97.96	2.87	2.80
	B3-24	70.90	2.58	96.62	3.00	2.58
	B4-26	63.26	2.19	76.61	2.92	2.20
C-20-75	C1-20	73.47	2.47	100.00	3.57	3.27
	C2-22	70.20	2.46	99.88	2.91	2.89
	C3-24	68.00	2.50	91.85	2.70	2.80
	C4-26	69.15	2.70	91.42	2.74	2.77
D-20-90	D1-20	75.40	2.01	78.41	3.41	2.92
	D2-22	73.50	2.42	99.82	3.48	2.90
	D3-24	73.88	2.42	105.00	3.41	2.83
	D4-26	73.47	2.58	103.00	3.30	2.73

[a]AA – Active alkali; T – Cooking time at maximum temperature / *AA – aktivna lužina; T – vrijeme kuhanja na najvišoj temperaturi*
[b]TEA: tensile energy absorption / *apsorpcija vlačne energije*

strength properties (Casey, 1960). The groups in this study, except for group A, can be bleached easily.

The most important characteristic features of paper are achieved by beating the pulp. Some properties of paper improve with beating, whereas some deteriorate. The important thing is to provide the required beating conditions to produce paper with the desired properties. There is an old saying in papermaking: "Paper is made in a beater" (Eroğlu, 1990). This study aimed at 36 SR° in the beating process. This was selected because 36 SR° is an average beating in which the physical, optical, and mechanical properties do not change excessively. The targeted SR° value was fully realized in some pulps, whereas there were some deviations (± 4 SR°) in others. The values in Table 3 were determined according to pulps corrected to 36 SR°.

3.1 Effects of cooking time (T)
3.1. Učinci vremena kuhanja (T)

When the cooking time was examined, the highest screened yield was for 75 min as seen Table 2 (A2= 47.25 %). Under these cooking conditions, some of the pulp features were also good. For example, the viscosity of the pulp was high as seen in Table 2 (A1=1418 cm^3/g), and the tear and tensile indices of the paper obtained from these pulps were also high. Although the high Kappa number value of these pulps seemed like a disadvantage, they could be bleached if needed. In other words, the important thing is that the pulp did not lose its strength properties.

3.2 Effects of sulfidity (S)
3.2. Učinci sulfidnosti (S)

When the cooking conditions were examined, a decrease in the Kappa number was seen within the groups (A,B,C and D, in Table 2) that had an increase in sulfidity at a constant AA ratio and cooking time. This indicated that delignification had increased. The viscosity did not change significantly within the groups because the sodium sulfide protected the cellulose from degradation during cooking.

3.3 Effects of active alkali (AA)
3.3. Učinci aktivne lužine (AA)

The decrease in the Kappa number occurred because the long cooking time and the high AA ratio directly increased the delignification. Similarly, the decrease in viscosity was due to the destruction of cellulose. Moreover, it is possible to produce paper having different properties from the same pulp since the properties of the pulp can be changed by beating. This provides the paper manufacturer with the opportunity to meet market demands.

4 CONCLUSIONS
4. ZAKLJUČAK

The most important reason for making use of wood raw materials in pulp production is the high screened yield. In this study, it was observed that, independent of the cooking time and active alkali rate, by increasing the sulfidity rate from 20 % to 22 %, there was an increase in the screened yield in all groups, but when it was increased to 24 %, there was a decrease in some. Therefore, the sulfidity ratio can be accepted as 22 %. Increasing the active alkali rate from 18 % to 20 %, while keeping the cooking time and sulfidity rate constant, reduced the screened yield. This was due to the increased degradation of cellulose. The decrease in viscosity confirmed this statement. Accordingly, the active alkali rate can be taken as 18 %. The yield was reduced by changing the cooking time from 75 min to 90 min (excluding D4) while keeping the active alkali rate and sulfidity constant. In this case, 75 min of cooking time was sufficient.

In this study, in general, the evaluation of the tensile, tear, and burst indices obtained during the cooking period of 75 min showed that some values had increased; however, most decreased when the time was increased to 90 min. The reason is that long-term cooking causes the degradation of carbohydrates and a decrease in viscosity. In this case, a cooking time of 75 min would be appropriate.

The produced paper exhibited high opacity values and would be suitable for use as writing and printing paper. Excluding Group A, their Kappa values provided an easy-to-bleach feature, and if desired, these paper sheets could be easily bleached without damaging their mechanical properties.

Taking into account the screened yield and some mechanical and optical properties of the avocado wood pulp produced via the Kraft method, the ideal cooking conditions were: active alkali rate: 18 %, sulfidity rate: 22 %, cooking time: 75 min. Using the Kraft method in the production of pulp from avocado wood enables cooking under more moderate conditions than with other hardwood types widely used in pulp production. Therefore, we believe that good results may be obtained from this kind of semi-chemical and mechanical production of pulp.

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Drvo afrormozije (*Pericopsis elata* (Harms) Meeuwen)

NAZIVI

Vrsta *Pericopsis elata* (Harms) Meeuwen iz porodice *Fabaceae* (*Leguminosae*) poznata je i pod sinonimom *Afrormosia elata* Harms. Otuda je afrormosia standardni trgovački naziv drva te botaničke vrste u Europi. Lokalni trgovački nazivi toga drva jesu: kokrodua, moholé (Gana); afrormosia, anyeran, ayin, egbi (Nigeria); assamela (Obala Bjelokosti); ején, obang (Kamerun); bohala, wahala, olé (Kongo).

NALAZIŠTE

Afrormozija je prirodno rasprostranjena u tropskim kišnim šumama zapadne Afrike, od Obale Bjelokosti do Konga, gdje je uglavnom nalazimo u skupinama.

STABLO

Stabla afrormozije narastu 30 – 45 m, s deblom dugačkim od 15 do 25 m te prsnog promjera od 0,6 do 1 m. Debla su uglavnom nepravilnoga poprečnog presjeka, katkad su valjkasta, zajedno sa žilištem. Kora je glatka, siva do sivosmeđa i debela oko 1 cm. Na starijim se stablima ljuska, a deblo ispod otpalih ljuski boje je cimeta do crvenkastosmeđe.

DRVO

Makroskopska obilježja

Bjeljika je bjelkasta i široka od 2 do 5 cm. Srž je smećkastožuta, ali i maslinasta, te s vremenom potamni. Tangentne su površine razgranato prošarane. Radijalne površine, osobito one dvostruko usukane žice, prugaste su, sjajne i izrazito lijepog izgleda. Miris svježe posječenog drva je neugodan.

Zone rasta raspoznaju se golim okom. Pore, drvni traci i aksijalni parenhim uočljivi su pod povećalom. Zbog katnog rasporeda drvnih trakova na uzdužnim su površinama vidljive fine vodoravne pruge.

Trupci ravne žice su rijetkost, vlakanca su često dvostruko usukana i tangentno valovita.

Mikroskopska obilježja

Pore drva na poprečnom su presjeku rastresitog rasporeda, prevladavaju pojedinačne, a mogu biti i u parovima te u kratkim radijalnim i kosim skupinama. Promjer im se kreće od 80…110…150 mikrometara, a gustoća od 9…16…23 na mm^2. Udjel pora u drvu je oko 26 %. U porama se nalaze svijetle sržne tvari. Aksijalni je parenhim apotrahealno marginalno raspoređen u širini 1 – 2 stanice ili paratrahealno vazicentrično, aliformno, konfluentno i unilateralno. Udjel aksijalnog parenhima u drvu je oko 15 %. U aksijalnom parenhimu česte su stanice s kristalima. Staničje drvnih trakova homogeno je i katno raspoređeno. Gustoća drvnih trakova je 7…9…12 po milimetru tangentnog smjera. Visina drvnih trakova iznosi 210…270…320 mikrometara, odnosno 11 do 18 stanica. Širina trakova je 16…30…44 mikrometara, odnosno 2 – 3 stanice, a udjel trakova u drvu je oko 15 %. Vlakanca su libriformska i difuznog rasporeda, mjestimice blago radijalno raspoređena. Debljina dvostrukih stijenki vlakanaca je 3,5…4,6…7,9 mikrometara, a promjer lumena 6,0…11,0…13,0 mikrometara. Dužina vlakanaca je 1,13…1,32…1,50 mm, a njihov udjel u drvu iznosi 44 %.

Fizička svojstva

Gustoća standardno suhog drva, ρ_o	oko 650 kg/m^3
Gustoća prosušenog drva, ρ_{12-15}	720…860 kg/m^3
Gustoća sirovog drva, ρ_s	1050…1200 kg/m^3
Poroznost	oko 57 %
Radijalno utezanje, β_r	3,0...3,5 %
Tangentno utezanje, β_t	6,0...7,0 %
Volumno utezanje, β_v	9,4...10,0 %

Mehanička svojstva

Čvrstoća na tlak	66...70 MPa
Čvrstoća na savijanje	120...150 MPa
Čvrstoća na vlak, okomito na vlakanca	oko 2,5 MPa
Tvrdoća HB paralelno s vlakancima	oko 32 MPa
Modul elastičnosti	11 300...13 500 MPa

TEHNOLOŠKA SVOJSTVA

Obradivost

Drvo se mehanički dobro obrađuje i neznatno zatupljuje oštrice alata. Optimalna brzina rezanja pri piljenju je 20 m/s. Blanjanje površina dvostruko usukane žice nešto je teže. Pri uvijanju vijaka i zabijanju čavala preporučuje se prethodno bušenje. Drvo se dobro tokari i reže. Pri lijepljenju treba izbjegavati alkalna ljepila. Površinski se dobro obrađuje, a lakira se izvrsno. Drvo s visokim sadržajem vode korodira u dodiru sa željezom. Prašina drva afrormozije može izazvati dermatitis.

Sušenje

Drvo se polako ali dobro suši, uz mali rizik od izvrtanja i pojave pukotina. Osušeno je drvo mirno, tj. stabilnih je dimenzija.

Trajnost i zaštita

Drvo afrormozije otporno je na napad gljiva truležnica (razred otpornosti 1 – 2) te na napad ksilofagnih kukaca suhog drva. Rizik od napada kukaca ograničen je na bjeljiku. Otporno je na napad termita (razred D). Bjeljika se lako impregnira (razred permeabilnosti 1), a srž je nepropusna (razred permeabilnosti 4). Zbog prirodne otpornosti drvo afrormozije prikladno je za uporabu na mjestima koja su u dodiru sa zemljom i sa slatkom vodom (razred 4). Drvo srži afrormozije ne zahtijeva zaštitu od ksilofagnih kukaca, kao ni u uvjetima povremenoga ili trajnog vlaženja.

Uporaba

Drvo afrormozije upotrebljava se za izradu rezanog furnira, namještaja i dijelova za namještaj visoke kvalitete, za unutrašnju stolariju i obloge (zidne i podne), za vanjsku stolariju i obloge, za tokarenu robu i unutarnja stubišta, a u brodogradnji nalazi primjenu u izradi oplata i brodskog poda. Odličan je nadomjestak za drvo tika (*Tectona grandis*).

Sirovina

Drvo se isporučuje u obliku trupaca dužine 4,0…10,0 m, srednjeg promjera 50 – 120 cm, te kao piljena građa, parket, furnir za oblaganje i furnirske ploče.

Napomena

Afrormozija je od 1992. na popisu ugroženih vrsta međunarodne organizacije CITES (dodatak 2, u propisima EU-a dodatak B). To znači da je za trgovinu trupcima, piljenom građom i furnirima afrormozije potrebna potvrda, tj. dozvola organizacije CITES. Drvo sličnih svojstava imaju i ostale vrste istoga roda te drvo tika (*Tectona grandis* L.f.), drvo ovangkola (*Guibourtia ehie* (A.Chev.) J. Leonard) i drvo aayan, tj. movinguia (*Distemonanthus benthamianus* Baill.).

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Upute autorima

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Primjer

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

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

Primjeri

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

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

Ostale publikacije (brošure, studije itd.)

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

Web stranice

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

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Example:

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

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Examples:

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

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

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Müller, D. 1977: Beitrag zur Klassifi zierung asiatischer Baumarten. Mitteilung der Bundesforschungsanslalt für Forst- und Holzwirtschaft Hamburg, Nr. 98. Hamburg: M. Wiederbusch.

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