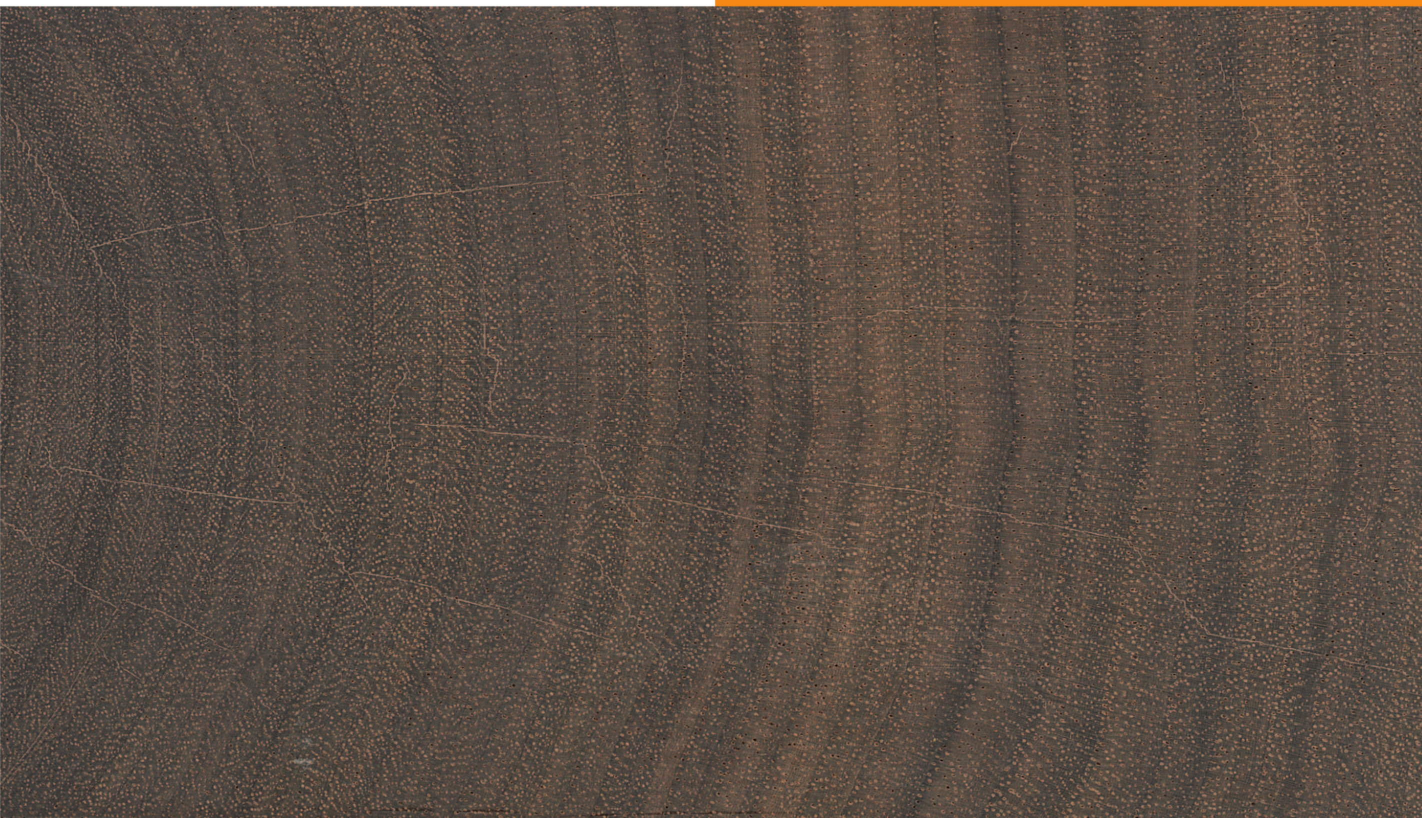
DRVNA INDUSTRIJA

SCIENTIFIC JOURNAL OF WOOD TECHNOLOGY



Juglans nigra L.

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Editorial

About a year ago, I received a friendly e-mail from the editor-in-chief of the journal *Drvna industrija* inviting me to take on the role of guest editor for a special issue. I took some time to think about it and responded positively a few days later. I decided to accept the invitation because I saw it as a great honour and recognition of my previous work, but also as a great challenge for me to learn how the work of an editor of a journal is done. So, the current issue of *Drvna industrija* came into being in less than a year.

I am a chemist by my background and my first contact with wood science and technology was 33 years ago, when I was a young PhD student at the University of Ljubljana, Biotechnical Faculty, Department of Wood Science and Technology. I knew next to nothing about this field, and the decision to work in the field of wood science and technology was a big jump into the unknown for me. The first steps in entering this field, which was completely new to me, were of course to study professional literature, and so I quickly became acquainted with the journal *Drvna industrija*. The articles in the journal were interesting, and the reason why I carefully examined each new issue and read the selected articles was also the easy accessibility of the journal and the fact that it was written in Croatian, a language I understood well. Publications of my colleagues' research results in journals with SCI impact factor were quite rare at that time, so publishing in *Drvna industrija* was an extremely important way for Slovenian researchers to communicate with the scientific community. The journal did not yet have an SCI impact factor, but it already had a wide regional reach in its field. Since then, I have looked forward to each new issue of the journal with interest, following the remarkable diversity of the interdisciplinary field of wood science and technology, its great development and progress, and at the same time the remarkable progress of the journal itself. A major breakthrough came in 2010 when the journal received an SCI impact factor of 0.146. Since then, the journal has made very good progress, moving from the bottom of the ranking of journals in the field of "Materials Science, Paper and Wood" to the border between the third and second quartile in the journal SCI-IF ranking. The journal is now making an extremely important contribution to the field of wood science and technology in the wider regional area and has a global reach.

For me personally and for all my colleagues at the Department of Wood Science and Technology at the Biotechnical Faculty in Ljubljana, the journal *Drvna industrija* means more than just the opportunity to publish our research results. It is a symbol of the long-standing excellent relationship between the Department of Wood Science and Technology and the Faculty of Forestry and Wood Technology at the University of Zagreb. Researchers from the Department of Wood Science and Technology continue to publish extensively in the journal *Drvna industrija*, and we have strengthened the bonds of our friendship through our reviewing activities and participation in the journal's editorial board. These have been deepened by numerous teaching, research and friendship visits to both institutions, meetings at conferences in Zagreb and Ljubljana and various social gatherings. When I researched the history of the study of wood engineering at the University of Ljubljana, I realised that my Zagreb colleagues played an extremely important role in the establishment and development of the study programme of wood science and technology in Ljubljana. As lecturers, they took over some fields of the study for which there were no university lecturers in Ljubljana at the beginning, and helped to train and empower Slovenian university lecturers in wood science and technology. The journal *Drvna industrija* was also essential in this respect. At the Department of Wood Science and Technology in Ljubljana, we are eternally grateful to our colleagues from Zagreb for playing this role.

Speaking of the present and the creation of this special issue of *Drvna Industrija*, I have seen up close and in detail how difficult and time-consuming it is to prepare each new issue of the journal. It is difficult to obtain high quality articles and to judge whether they are suitable for publication or not. A particular difficulty is finding reviewers and encouraging them to send in their reviews within reasonable deadlines. A demanding task is then to make an overview of the reviews and revised articles. It was particularly difficult to assess whether an article was suitable for publication or not when the reviewers' assessments were contradictory. Last but not least, checking the gallery proofs before submitting them to the press is also a very time-consuming task. This makes me appreciate even more than before the great work that my colleague and friend Prof. Dr. Ružica Beljo Lučić and her team have been doing for many years.

The special issue that I edited as a guest editor is now in front of you, dear readers. The content is very diverse and fully reflects the challenges that wood researchers are facing in the 21st century: from the concern for the preservation of wooden cultural heritage to the introduction of state-of-the-art wood processing technologies, the challenges of the green transition, the development of quality wood processing education and the concern for safety in wood industry. I am sure that every reader will find something interesting and useful in such a variety of content.

Prof. Dr. Marko Petrič

Boštjan Lesar*, Miha Humar, Florjan Osvald[1]

Colour Changes of Weathered Wood Surfaces Before and After Treatment with Iron (II) Sulphate

Promjena boje površina drva izloženih vremenskim utjecajima prije i nakon tretmana željezovim (II) sulfatom

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ABSTRACT • *Outdoor wood is exposed to various factors that can be described as weathering and cause the wood to grey. The ageing processes can vary greatly depending on the exposure. Parts of wood that are not exposed to external factors, e.g. under the overhanging, are less exposed, and the weathering process is therefore slower. This can be accelerated by solutions based on iron ions. In this way, the wood greys quickly and evenly. However, after iron treatment, the colour also depends on the previous exposure of wood to weathering. In our study, the colour change was observed as a function of weathering time. Before treatment, the samples were exposed to outdoor weathering for different periods of time and then treated with a 5 % solution of pure iron (II) sulphate and commercial iron (II) sulphate. It was determined that the pre-weathering time affected the final colour change, as the samples exposed for five weeks before treatment have comparable colour to naturally weathered wood. At the beginning of exposure, iron (II) sulphate limits mould growth, but after two months, staining fungi develop on the treated samples as well. The growth of blue stain fungi on the treated samples did not significantly affect the colour and visual appearance of the wood treated with iron-based solutions.*

KEYWORDS: *ageing; weathering; iron (II) sulphate; colour change; blue stain fungi*

SAŽETAK • *Drvo za vanjsku uporabu izloženo je različitim čimbenicima koji se mogu opisati kao vremenski utjecaji, a uzrokuju sivljenje drva. Procesi starenja mogu uvelike varirati ovisno o izloženosti drva tim utjecajima. Na dijelovima drva koji nisu izravno izloženi vanjskim čimbenicima, npr. ispod prevjesa, proces starenja je sporiji. Taj se proces starenja može ubrzati otopinama na bazi iona željeza tako da drvo brzo i ravnomjerno posivi. Međutim, boja nakon tretmana željezom također ovisi o prethodnoj izloženosti drva vremenskim utjecajima. U ovom smo istraživanju promatrali promjenu boje kao funkciju trajanja izloženosti drva vremenskim utjecajima. Uzorci su prije tretmana različito vrijeme bili izloženi vremenskim utjecajima, a zatim su tretirani 5 %-tnom otopinom čistog željezova (II) sulfata i komercijalnog željezova (II) sulfata. Utvrdili smo da je prvotna izloženost vremenskim uvjetima utjecala na konačnu promjenu boje jer su uzorci koji su bili pet tjedana izloženi vremenskim utjecajima prije tretmana imali sličnu boju kao i prirodno drvo izloženo vremenskim utjecajima. Na početku izlaganja željezov (II)*

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sulfat ograničavao je razvoj plijesni, ali su se nakon dva mjeseca izlaganja i na tretiranim uzorcima drva razvile gljive promjene boje. Razvoj gljiva plavila na tretiranim uzorcima nije znatnije utjecao na boju i vizualni izgled drva tretiranog otopinama na bazi željeza.

KLJUČNE RIJEČI: *starenje; izlaganje vremenskim utjecajima; željezov (II) sulfat; promjena boje; gljive plavila*

1 INTRODUCTION
1. UVOD

Outdoor wood is exposed to various weather conditions that cause the wood to turn grey (Feist, 1988; Williams, 2005; Humar *et al*., 2020; Kropat *et al*., 2020; Zabel and Morrell, 2020; Altay, 2023). The weathering processes can vary greatly depending on the exposure (Nejad and Cooper, 2017). Parts of wood that are not exposed to wetting, e.g. under overhang, are much less exposed to weathering factors. Therefore, the weathering processes are slower (Zimmer *et al*., 2018), which results in an uneven colour of wooden facades. Many customers do not like such an uneven visual appearance of wooden façades and other vertical surfaces (Zimmer *et al.* 2018). Uneven greying affects the aesthetic properties of buildings with wooden facades or other wooden objects exposed outdoors. This is a common reason for choosing other materials such as polymers and minerals that maintain a uniform colour over a longer period of time. Such material changes have an impact on lower wood consumption and the entire wood supply chain. One of the possible solutions for a more even colour is the use of pigmented coatings or wood-transparent coatings with UV absorbers. However, a coating has one major disadvantage: it requires regular maintenance (Nejad and Cooper, 2017), which is expensive and time-consuming. On the other hand, it was already proven that treatment of wooden facades, with an ion-based solution, results in more even and faster greying and prevents uneven discolouration of the building claddings (Hundhausen *et al*., 2020; Lesar and Humar, 2022). Iron-treated wood is also suitable for mimicking ancient wood (Høibø and Nyrud, 2010; Sun *et al*., 2023). However, the colour of wood treated with iron ions also depends on whether the wood has previously been exposed to weathering or has been aged by UV light (Nejad and Cooper, 2017; Zimmer *et al*., 2018). In the case of spruce wood, for example, wood freshly treated with iron (II) ions takes on a slightly greener hue compared to naturally aged spruce surface. The colour can even turn brown, if the wood is exposed only to solar radiation and not precipitation or condensation of surface water (Hundhausen *et al*., 2020). The finding of Hundhausen and co-workers in 2020 show that staining proceeds also on wood without the presence of wood extractives, possibly due to the oxidization of iron (II) that is promoted by photo-induced phenoxyl and ketyl radicals from photolysis of lignin ether bonds. Respective findings explain colour differences of iron (II) sulphate-treated façades that are partly protected by, for instance, a roof overhang.

The effect of iron (II) sulphate on greying is most notable in the initial phases after treatment (Lesar and Humar, 2022). The presence of transparent biocides (e.g. boric acid, quaternary ammonium compounds) had no effect on the colour of wood treated with iron (II) sulphate, nor on iron leaching from treated wood outdoors. However, the biocides positively affect the durability of the treated wood (Lesar and Humar, 2022). Positive effects on decay resistance and mould growth were also confirmed during hydrothermal treatment with different iron compounds (Aleinikovas *et al*., 2021). However, after several months of outdoor exposure, blue stain fungi develop on the surface of wood treated with iron (II) sulphate as well. On the other hand, iron (II) sulphate protects the wood surface exposed to weathering. The surface of the control samples was much more damaged than that of the treated samples (Lesar and Humar, 2022). Long-term outdoor weathering also shows that iron-treated wood is not damaged by wasps as untreated wood (Lesar *et al*., unpublished results) (Figure 1).

This study is a follow up of the paper: Performance of Iron (II)-Sulphate-Treated Norway Spruce and Siberian Larch in Laboratory and Outdoor Tests (Lesar and Humar, 2022). The aim of this work is to elucidate the importance of the pre-treatment prior to iron (II) sulphate impregnation. The key objective is to ensure that the colour of the treated wood is comparable to the colour of naturally weathered wood. In this study, untreated wood was exposed to outdoor weathering for various periods of time. Then the samples were treated with 5 % solution of iron (II)sulphate. Afterwards, the samples were exposed to the weathering again, and the colour changes, mould growth and other surface properties were assessed.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

2.1.1 Wooden material
2.1.1. Drvni materijal

The specimens used for the study were made of Norway spruce (*Picea abies* (L.) H. Karst) and European larch (*Larix decidua* Mill.). The dimensions of

Figure 1 Model façades exposed outdoors for 5 years. Above red line control samples, below iron (II) sulphate treated wood. Left façade exposed to nord, right façade exposed to south. Surface damage by wasps is clearly visible on control samples.
Slika 1. Modeli fasada pet godina izloženih vanjskim utjecajima. Iznad crvene crte nalaze se kontrolni uzorci, a ispod nje je drvo tretirano željezovim (II) sulfatom. Lijeva fasada bila je izložena prema sjeveru, a desna prema jugu. Na kontrolnim su uzorcima jasno vidljiva oštećenja površine, uzrokovane osama.

the specimens were 40 mm × 110 mm × 10 mm, with the radial orientation of the fibres in the radial direction (45±15)°. In total, 85 specimens were prepared. Of these, 45 were spruce samples, and 40 larch samples.

2.1.2 Treatment solutions
2.1.2. Otopine za tretiranje

The Norway spruce and larch specimens were treated with a 5 % iron (II) sulphate solution in distilled water. Two different sources of iron (II) sulphate were used. The concentration of the iron (II) sulphate solution was chosen based on previous studies (Lesar and Humar, 2022). Initially, iron (II) sulphate heptahydrate ($FeSO_4 \times 7H_2O$) (IS) for laboratory purposes in the form of blue-green crystals prepared by Carlo Erba was used. For comparison with the laboratory-grade iron (II) sulphate, a solution was also prepared with commercial, iron (II) sulphate with purity > 98 % and an iron (Fe) content of 20 %. (Agrolit, Slovenia). For commercial purposes, the name green vitriol (GV) was used. The green vitriol was in the form of granules. A solution of the commercial green vitriol (GV) was prepared according to the same procedure as the iron (II) sulphate solution.

2.2 Methods
2.2. Metode

2.2.1 Treatment
2.2.1. Tretman

The test specimens were treated by immersing each specimen in a solution of laboratory grade iron (II)sulphate or green vitriol for 10 seconds. After the treatment, the uptake of the solution was determined gravimetrically. The samples were then dried for 24 hours under laboratory conditions. The samples were then exposed outdoors.

2.2.2 Outdoor exposure
2.2.2. Vanjsko izlaganje

After conditioning, the samples were exposed outdoors at the test field site on special stands. The samples were exposed on the premises of the Department of Wood Science and Technology in Ljubljana, Slovenia (46°02'55.7 "N 14°28'47.3 "E, height above sea level 293 m).

2.2.3 Pre-weathering before iron (II) sulphate treatment
2.2.3. Predizlaganje vremenskim utjecajima prije tretmana željezovim (II) sulfatom

The Norway spruce and larch test specimens were placed on a stand at an angle of 45° and oriented towards the south (Figure 2). On 3 May 2022, the test specimens were exposed to outdoor weathering for 1 to 5 weeks. Each week, five samples of each species were isolated from the test plot and stored in a dark dry space. After the ageing was completed (after five weeks), the samples were scanned with an A3 2400S flatbed scanner. The colour was determined on scanner pictures with the programme Corel Photo PAINT 8 in the CIE Lab colour space. In the figure, the entire surface of each sample was selected, and the colour was measured using the tool in the software and all three CIE Lab components (L^*, a^*, b^*) were extracted. The samples were then treated with solutions of iron (II) sulphate or green vitriol as described in chapter 2.2.1, conditioned for 24 hours and exposed to the test field for a further 21 weeks. At this time, untreated control

Figure 2 Stand with samples facing south at exposure angle of 45°
Slika 2. Stalak s uzorcima izloženima prema jugu pod kutom od 45°

samples of both wood species, which had previously been kept in the dark, were also exposed outdoors alongside other treated samples.

Before and during exposure, the samples were scanned, and the colour was determined as described. Microsoft Excel was used to calculate the colour difference. The total colour difference Δ*E* (Eq. 1) between a reference colour (*L**0, *a**0, *b**0) and a target colour (*L**1, *a**1, *b**1) in the CIE Lab colour space is calculated by determining the Euclidean distance between two colours.

In addition to UV radiation, blue stain and mould fungi also contribute significantly to the discolouration of wood in outdoor applications. Therefore, as part of the study, the presence of blue stain and mould fungi on the samples using an Olympus DSX1000 digital microscope was determined as well. The study was conducted by scanning 1 to 2 representative samples of each treatment after 4, 8, 12, 21 weeks of weathering. The presence of blue stain fungi was assessed in both tests.

2.2.4 Weather data
2.2.4. Podatci o vremenu

The weather also influences the colour change of wood that lies outdoors. For this purpose, weather data were obtained from the archives of the Slovenian Environment Agency (ARSO, 2022). The data came from the ARSO monitoring station, Bežigrad, near the field test site. The amount of precipitation per day, the average temperature in °C and sunshine hours per day were the focus of our interest. For this purpose, archived data from the website of the Slovenian Environmental Agency (ARSO) were obtained for the duration of our experiment between 1 May 2022 and 3 November 2022.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Solution uptake
3.1. Upijanje otopine

The treatment was carried out by immersing each sample in the solution for 10 seconds and then placing the sample on a grid where the excess solution was drained off before weighing. The Norway spruce samples have an average uptake of 32.9 kg/m^3. Pre-weathering has a positive influence on the solution uptake. The highest uptakes were observed at the samples treated, aged for 2 and 4 weeks. On the other hand, the lowest uptake was recorded in spruce samples treated with iron (II) sulphate and aged for 1 week. It should be noted that the differences were not very prominent. However, the positive effect of the weathering on solution uptake is mainly a result of the microcracks on the surface, as reported by (Keržič and Humar, 2022). The larch samples had approximately 10 kg/m^3 lower up-

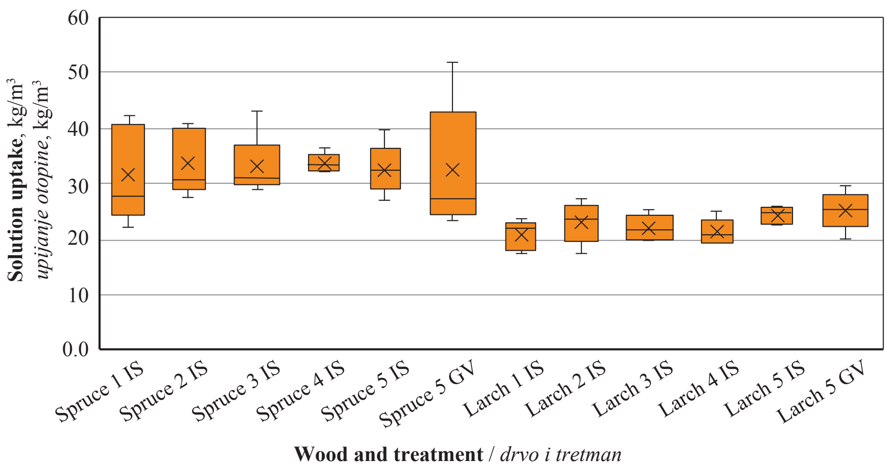

Figure 3 Influence of pre-weathering on solution uptake of spruce and larch wood (IS- iron (II) sulphate; GV- green vitriol). Numbers are weeks of pre-weathering.
Slika 3. Utjecaj predizlaganja smrekovine i ariševine vremenskim uvjetima na upijanje otopine (IS – željezov (II) sulfat; GV – zeleni vitriol). Brojevi označavaju tjedne predizlaganja vremenskim utjecajima.

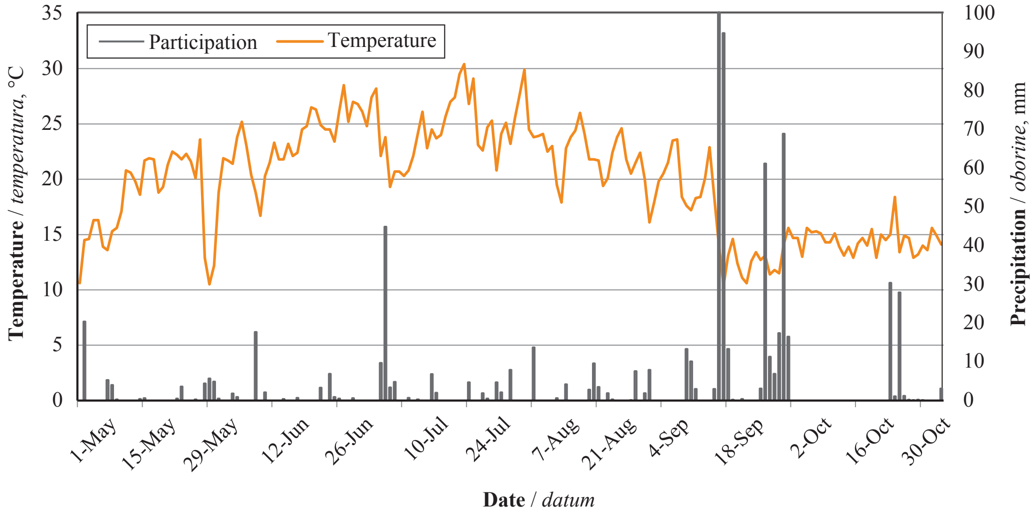

Figure 4 Average daily temperature and daily amount of precipitation from 1 May to 3 November 2022
Slika 4. Prosječna dnevna temperatura i dnevna količina oborina od 1. svibnja do 3. studenog 2022.

take than Norway spruce samples with an average uptake of 22.8 kg/m^3 (Figure 3). This can be mainly attributed to the different densities of the wood (larch has a higher density), as well as porosity and water-permeability of wood. The solution uptakes of various specimens are comparable, although the wood is weathered for different periods of time. The differences are believed to be merely due to the variability of the wood and described microcracks. Similar solution uptakes were reported in previous studies (Lesar and Humar, 2022; Lesar *et al*., 2022).

3.2 Weather data
3.2. Vremenski podatci

As already reported, weather conditions influence the weathering of wood (Feist, 1988; Evans *et al*., 1996; Williams, 2005; Gobakken and Høibø, 2011). Samples were exposed to weathering between 3 May and 3 November 2022. In this period, the average temperature was 19.8 °C, and average precipitation 4.2 mm and average daily solar radiation 7.7. hours (Figure 4 and Figure 5). In the observed period, there were 75 days with precipitation higher then 0.1 mm and the total rainfall in the respective period was 779.9 mm. The highest daily precipitation of 156.9 mm was reported on 16 September.

3.3 Pre-weathering before iron (II) sulphate treatment
3.3. Predizlaganje vremenskim utjecajima prije tretmana željezovim (II) sulfatom

Untreated samples were pre-weathered from 1 to 5 weeks before iron treatment. Total colour change increased with increasing weathering duration for both

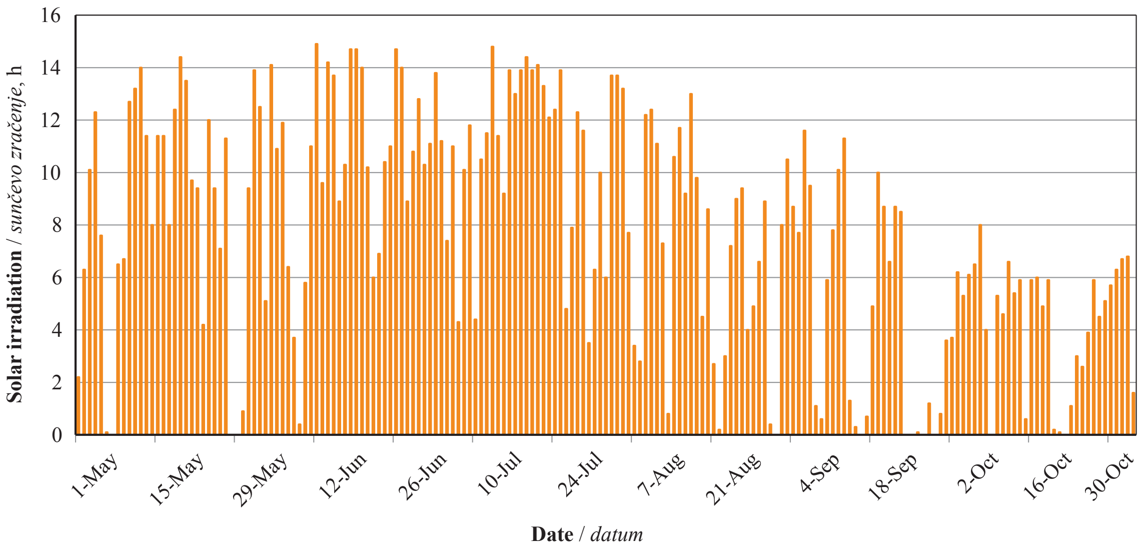

Figure 5 Duration of solar irradiation from 1 May to 3 November 2022
Slika 5. Trajanje Sunčeva zračenja od 1. svibnja do 3. studenog 2022.

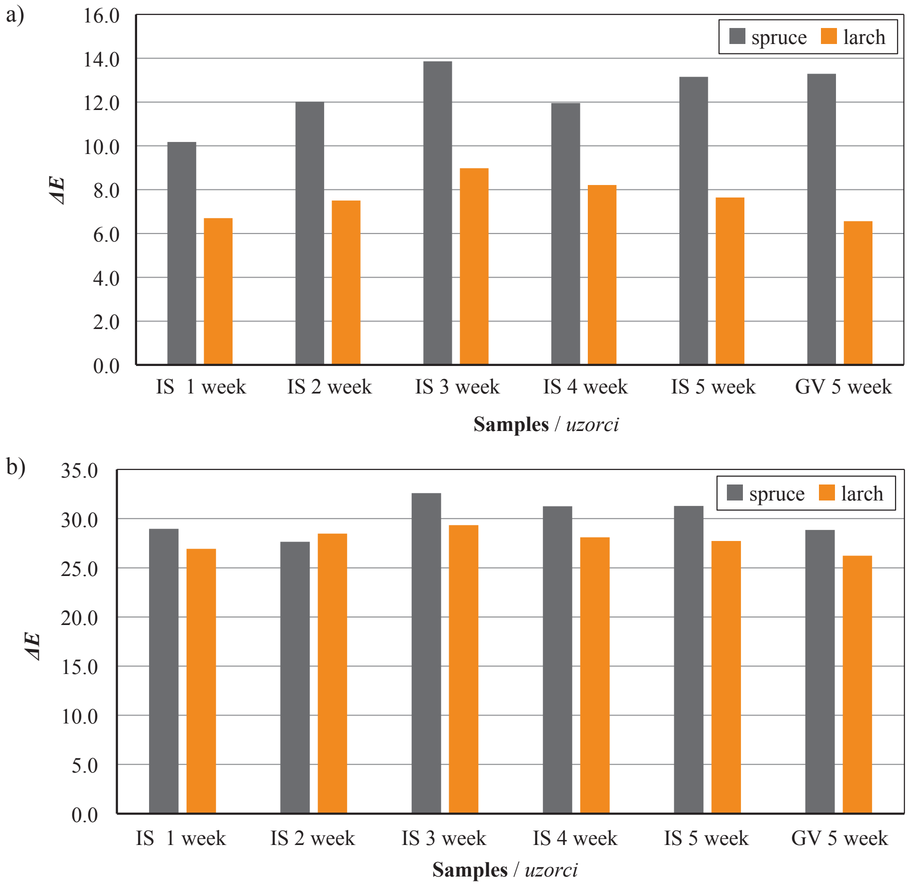

Figure 6 Colour change of Norway spruce and larch samples (a) exposed to weathering from one to five weeks (b) after weathering and treatment with iron (II) sulphate (IS) and green vitriol GV)
Slika 6. Promjena boje uzoraka obične smrekovine i ariševine (a) izloženih vremenskim utjecajima od jednog do pet tjedana i (b) nakon izlaganja vremenskim utjecajima i tretmana željezovim (II) sulfatom (IS) i zelenim vitriolom (GV)

wood species. In general, higher colour changes were determined on Norway spruce wood than on larch wood, predominately as the initial colour of spruce wood was lighter. The most prominent colour change appeared after the first week of outdoor exposure. Namely, colour change (ΔE^*) measured on Norway spruce samples was 10.2, and 6.7 on larch wood (Figure 6a). Pre-weathering of spruce samples and only minor differences for larch samples, exposed to weathering for 5 weeks and later treated with iron (II) sulphate or green vitriol, were observed. Results are in line with previous studies (George *et al.*, 2005; Niklewski *et al.*, 2022; Podgorski *et al.*, 2009).

The most prominent colour changes occurred after treatment with iron (II) sulphate and green vitriol. Colour measurement was performed only one day after the treatment, when the samples were dried. For Norway spruce samples, ΔE^* was 27.6 to 32.6. and for larch wood from 26.2 to 29.3 (Figure 6b). As already observed in a previous study (Lesar and Humar, 2022), colour change of larch samples was higher compared to Norway spruce samples. Larch samples become darker than spruce ones.

However, when the samples are exposed to outdoor weathering, the colour change is the most prominent between the first and second week for both wood species used. This phenomenon was not affected by pre-weathering and iron solution used; it can rather be attributed to the action of iron (II) sulphate on the wood (Figure 7 and 8). Similar patterns of colour change were observed in Norway spruce and larch samples. Comparing Norway spruce and larch samples, it can be seen that the colour change of Norway spruce samples is, on average, 4 units higher than that of larch samples. This can be attributed to the fact that larch is inherently darker compared to spruce, which lowers the colour change. After the third week, the colour change is more uniform. This shows that the most apparent change in colour occurs in the first two weeks of exposure after the iron ion treatment. The samples aged for one week and treated with iron (II) sulphate had the highest colour change, which can be attributed to pre-exposure to UV and rain for the shortest time. On average, the colour change of samples pre-weathered for 5 weeks is 8 units lower compared to samples pre-weathered for 1 week. This can be attributed to the fact that the more the samples have been pre-weathered, the more prominent colour change occurs before treatment with IS and GV solutions, respectively. The colour changes of the samples pre-weathered for 5 weeks are very uniform

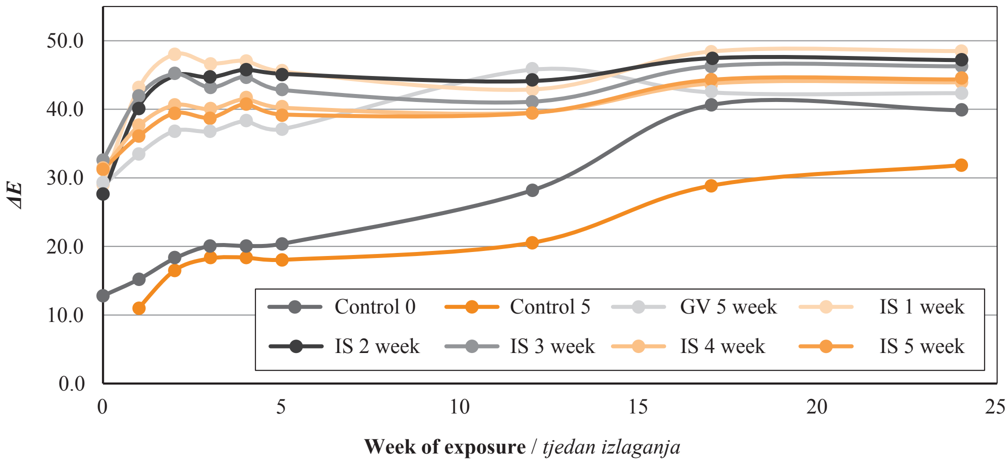

Figure 7 Colour change (Δ*E**) of control and iron (II) sulphate and green vitriol treated Norway spruce wood samples, exposed outdoor at 45° to south
Slika 7. Promjena boje (Δ*E**) kontrolnih uzoraka smrekovine tretiranih željezovim (II) sulfatom i zelenim vitriolom izloženih na otvorenome i prema jugu pod kutom od 45°

after the third week of exposure, as the additional colour change varies only by two units. The colour change of the control samples also shows a maximum colour change in the first three week of exposure, followed by a slight increase of colour change till the end of the experiment. However, none of the control samples exposed at the beginning of May or 5 weeks later did not reach the exact colour change as the treated samples. Untreated samples would require additional time to reach the final appearance.

3.4 Visual appearance of weathered control and treated samples

3.4. Vizualni izgled izloženih kontrolnih i tretiranih uzoraka

Figures 9 and 10 show the visual changes in Norway spruce and larch samples exposed outdoors. In the first row there is an untreated control sample of Norway spruce or larch that has been pre-weathered for 5 weeks. This is followed by the samples pre-weathered for 1 week and treated with a 5 % iron (II) sulphate solution, the samples pre-weathered for 3 weeks and treated with a 5 % IS solution, and the samples pre-weathered for 5 weeks and treated with the same 5 % IS solution, and the sample treated with 5 % (GV) green vitriol is in the last row. All samples were exposed for 21 weeks after treatment.

Figures 9 and 10 confirm the results of the colour change measurements. The control sample shows a distinct colour change until the second week of exposure, after which the colour remains fairly uniform until the 12th week when further distinct colour changes occur. However, for the samples that were pre-weathered and treated with iron-containing solutions, a visible colour change was observed shortly after treatment. The one-week-pre-weathered sample is darker and browner than the three- or five-weeks pre-weathered samples. The five-week pre-weathered samples show even shades of grey. It was established that the one-week pre-weathered samples become

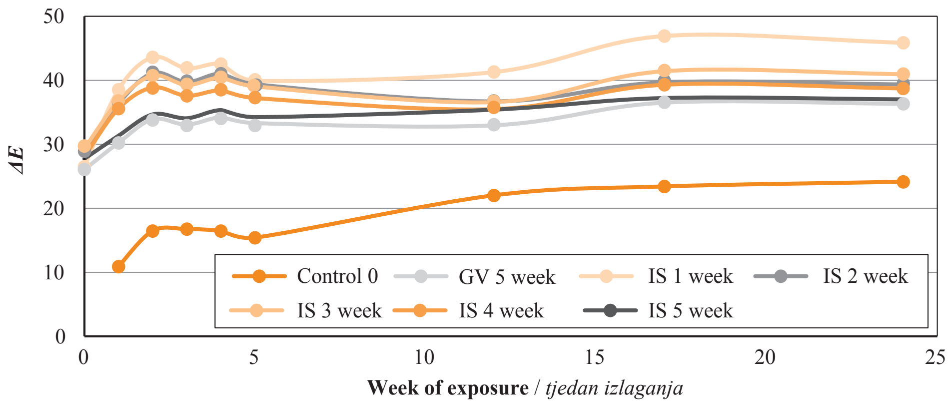

Figure 8 Colour change (Δ*E**) of control and iron (II) sulphate and green vitriol treated larch wood samples, exposed outdoor at 45° to south
Slika 8. Promjena boje (Δ*E**) kontrolnih uzoraka ariševine tretiranih željezovim (II) sulfatom i zelenim vitriolom, izloženih na otvorenome i prema jugu pod kutom od 45°

Figure 9 Visual appearance of control and iron treated Norway spruce wood samples. The first column shows wood samples before the test, the second column shows samples aged outdoors for 1, 3 and 5 weeks, the third column shows samples immediately after treatment with iron (II) sulphate (IS) and green vitriol (GV), the fourth to eleventh columns show colour changes during outdoor exposure from week 1 to week 24.
Slika 9. Vizualni izgled kontrolnih uzoraka i uzoraka smrekovine tretiranih željezom. Prvi stupac prikazuje uzorke drva prije ispitivanja, u drugom su stupcu uzorci izloženi na otvorenome jedan, tri i pet tjedana, treći stupac predočuje uzorke neposredno nakon tretiranja željezovim (II) sulfatom (IS) i zelenim vitriolom (GV), a uzorci od četvrtog do jedanaestog stupca pokazuju promjene boje tijekom vanjskog izlaganja od 1. do 24. tjedna.

browner, while the five-week pre-weathered samples become grey. This indicates that the UV-induced radical changes continue long after initiation, although wood was stored in a dark place. The five-week pre-weathered sample achieves a better approximation to the colour of naturally aged wood. For the five-week pre-weathered samples, there was only a slight difference in colour between IS and GV treatment. For

Figure 10 Visual appearance of control and iron treated larch wood samples. The first column shows wood samples before the test, the second column shows samples aged outdoors for 1, 3 and 5 weeks, the third column shows samples immediately after treatment with iron (II) sulphate (IS) and green vitriol (GV), the fourth to eleventh columns show colour changes during outdoor exposure from week 1 to week 24.
Slika 10. Vizualni izgled kontrolnih uzoraka i uzoraka ariševine tretiranih željezom. Prvi stupac prikazuje uzorke drva prije ispitivanja, drugi stupac uzorke izložene u vanjskom prostoru jedan, tri i pet tjedana, u trećem su stupcu uzorci neposredno nakon tretiranja željezovim (II) sulfatom (IS) i zelenim vitriolom (GV), a uzorci od četvrtog do jedanaestog stupca pokazuju promjene boje tijekom vanjskog izlaganja od 1. do 24. tjedna.

larch wood, the difference was slightly more prominent. It is believed that this difference is mainly related to the more prominent colour variations in fresh wood samples.

If the samples of spruce and larch are compared, the relation between colour and ageing can be seen in both species. Although larch samples are clearly darker than spruce samples, there is a clear correlation between ageing and colour change in both species analysed. This can be observed in specimens that have been pre-weathered for one week showing more browning and those pre-weathered for five- weeks showing more greying. Another reason for the darker colour of larch samples is that they have a higher proportion of extractives that react with iron (Hundhausen, 2020).

3.5 Observation of blue stain fungi
3.5. Promatranje gljiva plavila

During the experiment, the presence of blue stain fungi and moulds on the surface was monitored. The analysis was performed on the exposed control, IS-treated and GV-treated samples. The sample surfaces were observed with a digital microscope. The measure-

Figure 11 Observation of mould growth on Norway spruce wood samples. Columns show wood samples exposed outdoors for 4, 8, 12 and 21 weeks after treatment. The first raw control samples (0) were exposed at the same time as the treated samples, the second raw control samples were exposed for 5 weeks before treatment, raw samples 3, 4, 5 were aged for 1, 3 and 5 weeks before treatment with iron (II) sulphate (IS) and raw samples 6 for five weeks before treatment with green vitriol (GV). The bar is 400 µm long.
Slika 11. Promatranje razvoja plijesni na uzorcima obične smrekovine. Stupci prikazuju uzorke drva izložene na otvorenome 4, 8, 12 i 21 tjedan nakon tretmana. Prvi netretirani kontrolni uzorci (0) izloženi su u isto vrijeme kad i tretirani uzorci, drugi netretirani kontrolni uzorci izloženi su pet tjedana prije tretmana, netretirani uzorci 3., 4. i 5. izlagani su jedan, tri i pet tjedana prije tretmana željezovim (II) sulfatom (IS), a netretirani uzorci 6. izlagani su pet tjedana prije tretmana zelenim vitriolom (GV). Žuta je linija dugačka 400 µm.

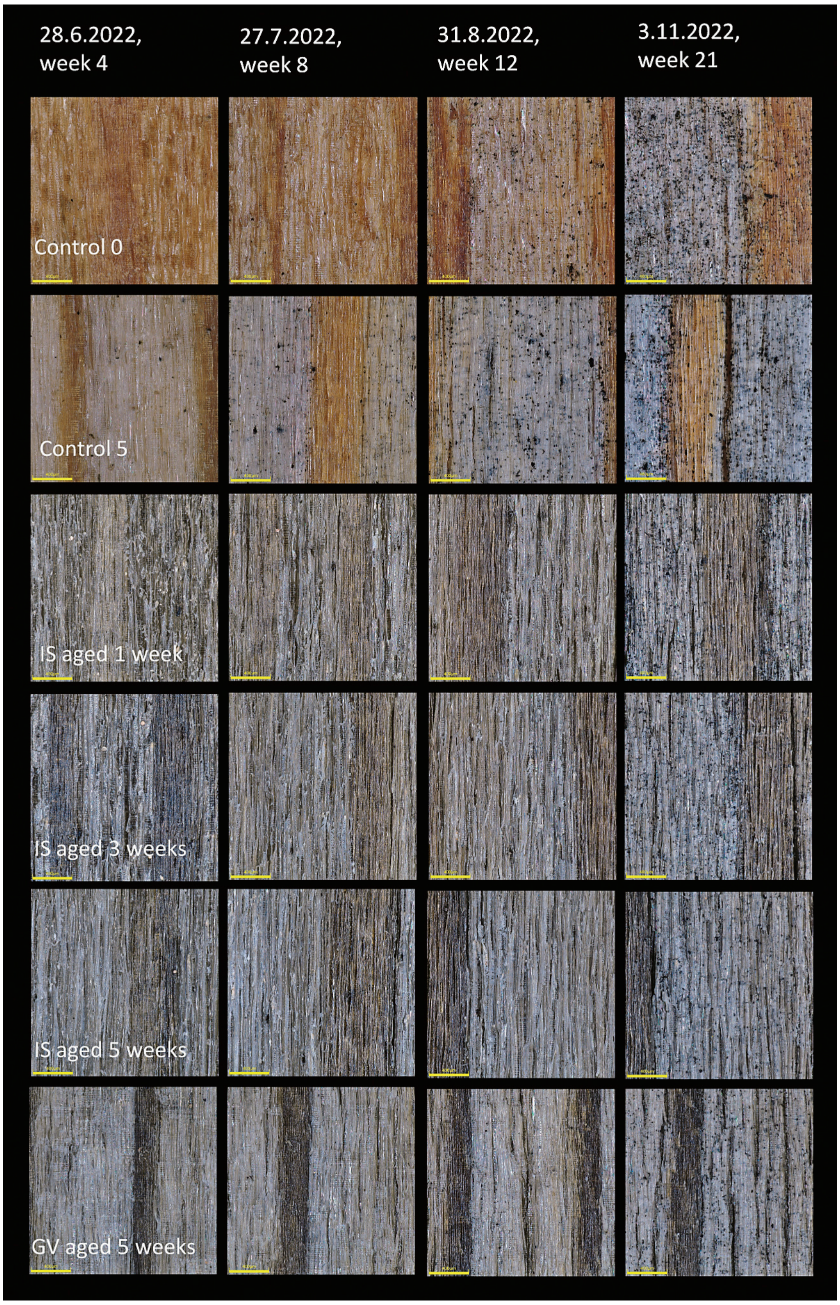

Figure 12 Observation of mould growth on larch wood samples. Columns show wood samples exposed outdoors for 4, 8, 12 and 21 weeks after treatment. The first raw control samples (0) were exposed at the same time as the treated samples, the second raw control samples were exposed for 5 weeks before treatment, raw samples 3, 4, 5 were aged for 1, 3 and 5 weeks before treatment with iron (II) sulphate (IS) and raw samples 6 for five weeks before treatment with green vitriol (GV). The bar is 400 µm long.

Slika 12. Promatranje razvoja plijesni na uzorcima ariševine. Stupci prikazuju uzorke drva izložene na otvorenome 4, 8, 12 i 21 tjedan nakon tretmana. Prvi netretirani kontrolni uzorci (0) izloženi su u isto vrijeme kad i tretirani uzorci, drugi netretirani kontrolni uzorci izloženi su pet tjedana prije tretmana, netretirani uzorci 3., 4. i 5. izlagani su jedan, tri i pet tjedana prije tretmana željezovim (II) sulfatom (IS), a netretirani uzorci 6. izlagani su pet tjedana prije tretmana zelenim vitriolom (GV). Žuta je linija dugačka 400 µm.

ments were performed after 4, 8, 12 and 21 weeks of outdoor exposure.

The figures (Figure 11 and 12) show the presence of blue stain fungi on the aged samples of Norway spruce and larch. The samples that were pre-weathered before treatment, control-5 and control-0 were exposed at the test site on the same day as the treated samples. The samples of control-5 showed the first signs of blue stain fungi already after nine weeks (5 weeks pre-weathering + 4 weeks of weathering), in both wood species. After 8 weeks, the first signs of blue stain fungi were also observed on the control-0 larch samples, that were not pre-weathered, which is comparable to control-5 samples weathered for 4 weeks. On the other hand, the first signs of blue stain fungi were observed on the control-0 Norway spruce samples after 12 weeks of weathering. The strongest growth of blue stain fungi occurred between week 12 and 21 for both wood species. These changes on the surface were also detected by colour measurements (Figure 7 and 8) and the visual appearance of the surfaces (Figure 9 and 10). Such pronounced growth was to be expected as it occurred during the autumn months with a lot of rain (Figure 4). This is in line with other research stating that the colour of the wood fluctuates due to the stronger growth of blue stain fungi in the autumn months (Kržišnik *et al*., 2018). The growth of blue stain fungi was more intense in the early wood than in the late wood. This can be observed especially in the control and treated larch samples (Figure 12).

The presence of blue stain fungi on the treated samples was influenced by the pre-weathering duration before treatment. All samples were treated on the same day with the same concentration (5 %) of iron (II)sulphate solution. The first signs of blue stain can be observed already four weeks after treatment (IS-treated samples were 5 weeks pre-weathered before treatment), just like control 5. This can be observed in Norway spruce and larch samples. These are only small patches of blue-stain fungi predominately due to inoculation of the samples during pre-weathering. On other iron-treated samples, no signs of blue stain fungi were found until week 12. Like the control samples, the most prominent growth of blue stain fungi was observed on both wood species used between 12 and 21 weeks after exposure. Nevertheless, the surface area covered by blue stain fungi was lower in the treated samples than in the untreated control samples. The biocidal activity of iron can only be observed at the beginning of the exposure; later, the iron was leached from the wood (Lesar and Humar, 2022; Lesar *et al*., 2022; Zimmer *et al*., 2020), and its biological activity was insignificant. At the end of the exposure, there was no statistically significant difference in the growth of blue stain fungi between the different ageing times of the samples before treatment. Figures 13 and 14 show that there was no difference between IS and GV treatment. The growth of blue stain fungi on the treated samples has no significant influence on the colour and visual appearance of the wood treated with iron.

4 CONCLUSIONS
4. ZAKLJUČAK

For the treatment, it was sufficient to immerse the samples in the iron aqueous solution for 10 seconds to obtain the desired effect. The solution uptake is different for spruce and larch since larch wood is denser and less permeable. It was noted that the pre-weathering has an influence on the higher uptake of iron-based solutions in both wood species. As expected, pre-weathering of the wood affects the colour change, and the changes are visible faster after continuous exposure. The most prominent colour changes occur in the first few weeks of outdoor exposure. Lower solution uptake of iron-based solutions in larch wood specimens had no effect on the colour change. It is believed that this is due to the higher proportion of extractives in larch that react with iron ions compared to Norway spruce.

During a 21-week observation period of aged samples treated with iron-containing solutions, the colour of larch and Norway spruce wood samples changed significantly during the first two weeks of exposure. After that, the colour changes are less prominent. In addition, samples that were pre-weathered for 1 week before treatment show a stronger colour change than those that were exposed to outdoor weathering for 5 weeks. This is also confirmed when analysing the visual appearance, for both the Norway spruce and larch samples: The samples pre-weathered for 1 week are browner than those weathered for 5 weeks. This indicates that the UV induced changes continue long after the exposure. However, samples pre-weathered for only 5 weeks are more similar to naturally aged wood, i.e. they have a brownish-grey colour. Blue stain fungi are the main cause of wood discolouration, along with UV light and rain. It was found that they are present in all samples but in varying amounts. The growth of blue stain fungi on the treated samples had no significant effect the colour and visual appearance of the wood treated with iron-based solutions. There was no difference in colour change and mould growth between pure iron (II) sulphate and green vitriol solutions prepared from industrial-grade iron sulphate. It can be concluded that it makes sense to use industrial green vitriol for the commercial application for accelerated greying of wood, as it is much cheaper than that for laboratory analysis. Further research is needed to investigate the possibility of artificially ageing or otherwise pre-treating the wood prior to treatment with iron (II) sulphate to achieve a uniform grey colour similar to that of naturally aged wood. This could be a good basis for the commercialisation of the whole process of wood ageing with iron (II) sulphate.

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Efficiency Assessment Based on Data Envelopment Analysis for Occupational Accidents and Diseases in Furniture Industry of Turkey

Procjena učinkovitosti turske industrije namještaja primjenom analize omeđivanja podataka o broju nezgoda na radu i profesionalnih bolesti

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ABSTRACT • *In this study, the provinces in which enterprises operate heavily in the Turkish furniture industry were subjected to an efficiency assessment using the data envelopment analysis (DEA) method based on work accidents and occupational diseases. The data related to the furniture industry was obtained from the Social Security Institution (SSI) and the Ministry of Labor and Social Security (MoLSS). The number of operating businesses and the number of employees insured in each province were evaluated as inputs for the analysis. The total number of workplace physicians and other health personnel in each province, the number of workplace physician and occupational safety centers and A, B, and C-class occupational health and safety specialists were used as outputs. The number of employees who experienced work accidents and occupational diseases, deaths resulting from work accidents and occupational diseases, total temporary disability periods, the number of beneficiaries, and the number of those receiving permanent disability income in each province were evaluated as undesirable outputs. The data was analyzed using the DEA method by modeling the undesired outputs in 6 different ways, and the efficiency of each province was determined. It was seen that Model 6 gave the most ideal results in DEA efficiency assessments. Aydın, Çanakkale, Diyarbakır, Eskişehir, Malatya, Muğla, Trabzon and Zonguldak were determined as the most effective provinces in terms of occupational health and safety. The results were evaluated along with the literature, and recommendations were presented.*

KEYWORDS: *DEA; data envelopment analysis; non-parametric evaluation methods; work accident and occupational health; furniture industry*

SAŽETAK • *U ovom je istraživanju primjenom metode analize omeđivanja podataka (DEA) na temelju broja nezgoda na radu i profesionalnih bolesti napravljena procjena uspješnosti pokrajina u kojima intenzivno posluju*

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poduzeća turske industrije namještaja. Podatci vezani za industriju namještaja dobiveni su od Zavoda za socijalnu skrb i Ministarstva rada i socijalne skrbi Turske. Ulazni podatci za analizu bili su procijenjeni broj operativnih poduzeća i broj zaposlenika u svakoj pokrajini, a izlazni podatci sadržavali su ukupan broj liječnika medicine rada i drugoga medicinskog osoblja u svakoj pokrajini, broj centara za zaštitu na radu te broj stručnjaka zaštite na radu A, B i C klase. Kao nepoželjni izlazni podatci za analizu u svakoj su pokrajini uzeti u obzir broj zaposlenika koji su doživjeli nezgodu na radu i/ili oboljeli od profesionalne bolesti; broj smrti uzrokovanih nezgodama na radu i/ili profesionalnom bolešću; ukupno trajanje privremene nesposobnosti za rad, kao i broj korisnika te onih koji primaju trajnu invalidninu. Podatci su analizirani uz pomoć DEA metode (analize omeđivanja podataka) modeliranjem neželjenih izlaza na šest različitih načina te je određena učinkovitost svake provincije. Pokazalo se da je model 6. dao optimalne rezultate u procjeni učinkovitosti DEA metodom. Utvrđeno je da su Aydın, Çanakkale, Diyarbakır, Eskişehir, Malatya, Muğla, Trabzon i Zonguldaku najuspješnije pokrajine u smislu zdravlja i sigurnosti na radu. Rezultati su analizirani usporedno s literaturom te su dane preporuke za moguća poboljšanja.

KLJUČNE RIJEČI: *analiza omeđivanja podataka (DEA); neparametarske metode evaluacije; nezgoda na radu; turska industrija namještaja*

1 INTRODUCTION
1. UVOD

Occupational health and safety (OHS) is an essential element of the business world. At this point, occupational accidents and diseases (OADs) are an issue that particularly concerns employees in many sectors. Sectors such as manufacturing, production, mining, construction, transportation, underground and underwater contain many risks for employees. The issue of OADs is not only a matter of concern for employees, but also affects and concerns employers, the relatives for whom the employee is responsible, and the government. In Turkey, this issue has been taken more seriously since the adoption of the Occupational Health and Safety Law No. 6331 in 2012 within the framework of the European Union (EU) adaptation laws. Although the law contains important provisions for workplaces and employers to take serious measures regarding OHS and to protect employees, and provides for serious sanctions for non-compliance, the rates of OADs in Turkey are still at low levels. According to Serin *et al*. (2015), in terms of the manufacturing sector, Turkey takes the first place in Europe and second in the world when rankings are examined according to the frequency of occupational accidents. This situation shows the necessity of analyzing occupational accidents seriously in Turkey (Akyüz *et al*., 2016).

During work life, OHS has a priority that requires a national-level effort involving not only preventive measures taken by employers at the workplace but also contributions from employees and the government. Therefore, as in other countries, various regulations have been implemented and will continue to be implemented in Turkey to continuously improve the working conditions, ensure effective participation of employees in OHS efforts, and plan national-level education on this subject. To measure the effectiveness of such regulations and see their impact, it is necessary to look at the indicators of countries' performance in this area. Statistical data on OHS and its changes and distribution over the years are essential elements of the business world.

Data on OADs in Turkey are recorded and published by the Social Security Institution (SSI). Until 2013, only accident data for incidents that occurred and were closed within the year were published. However, with the implementation of heavy sanctions for those who fail to report accidents, particularly following Law No. 6331, all reported accidents have been published since 2013. However, it is an issue to be considered that these statistical data only cover the reported OADs. According to Kurttekin and Taçgın (2019), it is known that many occupational accidents are not reported or recorded due to insufficient control and inspection. In addition, identifying occupational diseases is also quite difficult because most occupational diseases emerge either in advanced age or after the worker's retirement, so they are not considered as occupational diseases and are not recorded. Therefore, the accuracy of the data obtained from SSI statistics is accepted as a limitation of the research.

The furniture industry is one of the leading subsectors within the manufacturing industry. According to Magezi and Okan (2023), the Turkish furniture industry has made good use of the opportunities in the last ten years and has become competitive in EU. There are 21,758 businesses in the sector, with 154,829 insured employees (SSI, 2019). Furniture companies are mainly classified as dangerous and highly dangerous based on their risk group. The sector includes many hazardous tools such as cutting, drilling, carving, and crushing equipment, as well as many chemicals that threaten human health, such as wood dust and formaldehyde, paint, and thinner. In addition, there are many heavy tasks and workloads during production and assembly that can harm the musculoskeletal system of workers.

There are many different studies on the subject in the literature. Koç *et al*. (1998) examined OADs in the furniture industry and developed recommendations for their prevention. Akyüz *et al*. (2016) studied accident statistics in the forest products industry sector and made general evaluations regarding the issue. Gedik and İlhan (2014) analyzed OHS issues among furniture manufacturers in Sakarya province. Sevim Korkut and Gedik (2010) examined occupational safety in the Turkish forest products industry sector. Ulutaş (2016) evaluated provinces in Turkey in terms of OADs. Bilim and Bilim (2015) conducted a statistical study on OHS in Turkey and analyzed the issue. Lombardi *et al*. (2019) modeled a risk profile using the European Statistics on Accidents at Work (ESAW). Xu and Xu (2021) statistically revealed fatal accidents, accident numbers, accident locations, and accident areas in the construction sector in China. Nissi and Rapposelli (2012) evaluated occupational accidents in three economic sectors (manufacturing, construction and distribution trades) in Europe through VZA analysis. In this study, unlike the studies in the field, besides an activity evaluation, a model is also proposed that can be used in evaluating the activity.

This research is aimed to develop suggestions to minimize the work accidents (WA) or occupational diseases (OD) that occur in the provinces where the Turkish furniture industry is concentrated. The provinces determined for this purpose were evaluated in terms of the number of enterprises, employees, workplace physicians (WP) and other health personnel (OHP), and the number of occupational health and safety specialists (OHSS).

1.1 Data envelopment analysis (DEA)
1.1. Analiza omeđivanja podataka (DEA)

The data envelopment analysis (DEA) method has been chosen for the evaluation. It is one of the non-parametric evaluation methods. It allows efficiency evaluation with many inputs and outputs, and at the same time, it is easy to include undesirable outputs (UO) in efficiency evaluation with various models.

DEA was developed by Charnes *et al*. (1978) to measure and compare the technical efficiency of public institutions based on an article by Farrell (1957) on productivity measurement (Safak *et al*., 2014). DEA is a linear programming-based technique used to measure the performance efficiency of organizational units called Decision Making Units (DMUs). This technique measures how efficiently DMUs use available resources in producing a set of outputs (Charnes *et al*., 1978).

DEA gets its name from covering observations to set a cutoff value for evaluating investigations that represent the performance of all assets. It can be used to evaluate the performance not only of enterprises but also of public institutions and non-profit organizations such as schools, hospitals, and banks, and also of cities, regions, and countries in various fields (Cooper *et al*., 2006; Ulutaş, 2016; Depren, 2008). For this purpose, the definition of DMUs has been kept flexible to include any entity that uses similar inputs to produce similar outputs (Cooper *et al*., 2006).

1.2 Classic DEA models
1.2. Klasični DEA modeli

Charnes *et al*. (1978) designed a model 'that generalizes a single output rate efficiency measure for a single DMU in terms of a fractional linear programming formulation that converts multiple outputs into a single virtual output (Manzoni and Islam, 2012; Charnes *et al*., 1994). It is called the 'Charnes, Cooper and Rhodes (CCR) Model' in the literature. When this model is used to solve the problem for each DMU under investigation, it determines the best set of weights for each DMU (Pasupathy, 2002). Later, the model was developed by Banker *et al*. (1984) and the two models, BCC and CCR, have started to be used in the literature. Then, the DEA technique was used and improved by many people (Ulutaş, 2016; Depren, 2008; Charnes *et al*., 1978; Banker *et al*., 1984). While the CCR model used the assumption of constant returns to scale only in the measurement of technical efficiency, the BCC model developed the concept of efficient scale and rearranged the linear programming formula in the CCR model for the estimation of the return to scale (Depren, 2008; Besen, 1994).

DEA is used to determine the "most efficient" or effective decision-making units (DMUs) within a set of DMUs by using the least inputs to produce the most outputs. The accepted efficiency boundary is 1, and the efficiency levels of ineffective DMUs are measured using this boundary as a technical reference. DEA enables obtaining a single efficiency value for each DMU based on multiple input and output variables by using a linear programming model (Depren, 2008).

In a simple DEA model, the efficiency of a random DMU(i) can be defined as:

$$\text{Effectiveness of DMU(i)} = \frac{\text{Weighted sum of outputs for DMU(i)}}{\text{Weighted sum of inputs for DMU(i)}}$$

The solution of the formula is iterated with linear programming for each DMU under a set of predefined constraints. One of the constraints is the weighting factor. When reaching the solution, the unit is not allowed to choose weights that would cause it to achieve an efficiency greater than 100 %. The weighted sum of the DMUs outputs must be less than or equal to the weighted sum of its inputs. In addition, the weighted sum of the inputs is assumed to be 1 to prevent limitless solutions (Manzoni and Islam, 2009). The principal aim of this equation is the assumption of obtaining the maximum output with the minimum input.

One of the points to be considered for DEA is the number of DMUs. The number of DMUs is crucial as it affects the decision-making ability of the model. For the optimal sample size, the number of DMUs should be greater than the sum of the inputs and outputs. In addition, the sample size is acceptable if the number of fully productive DMUs is not more than one-third of the total number of DMUs in the sample (Cooper *et al*., 2006; Manzoni and Islam, 2009).

1.3 DEA models with undesirable outputs
1.3. DEA modeli s neželjenim izlazima

The DEA approach has a structure that aims to bring the DMU closer to the determined efficiency limit by reducing inputs or increasing outputs after an active limit value is determined in general. However, UO or inputs may also occur in this process (Zhou *et al*., 2008). UO are the unexpected results of manufacturing processes aimed at maximizing outputs while minimizing inputs. For this reason, the perspective of DEA to approach the effective limit by increasing the output should be handled differently in terms of UO (Seiford and Zhu, 2002). Therefore, many different DEA models have been developed, including UO. Seiford and Zhu (2002) or Faere *et al*. (1989) can be shown as the most widely known and used DEA models. Whereas Seiford and Zhu's (2002) model focuses on data transformation, Faere *et al*. (1989) show an approach that ignores UO (Zhou *et al*., 2008; Seiford and Zhu, 2002). Table 1 lists six specific models that are most frequently used and recommended in the literature.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

Sectoral data in the study were taken from SSI 2019 statistical annuals. First, the provinces where the furniture sector was intense were determined using these data. From the insurance and workplace statistics table in statistical annuals of SSI 2019, the data under the furniture manufacturing title with the code 31 in the NACE coding system have been compiled according to the provinces. In 2019, the number of workplaces with SSI registration in the furniture sector was 21.758, and the number of employees was 154.829. In the study, it was determined that approximately 92 % of the insured employees in the sector are concentrated in 30 provinces (Table 2). The number of enterprises in 30 cities constitutes about 88 % of the total number of enterprises (SSI, 2019). For this reason, the research was limited to 30 provinces, as the representativeness of the data is high.

Table 3 presents the number of WP, OHP in the provinces and the numbers of A, B and C class OHSS. These data were taken from the statistical table on the website of the Ministry of Labor and Social Security (MoLSS, 2021). The information on the Ministry webpage is irregular and does not include the distribution by year. That was assumed as a constraint of the research.

The data used in the study have been compiled from the tables in the SSI 2019 Occupational Accident and Occupational Disease Statistics. These data were the number of employees who have had WA and OD, the number of cases of WA and OD, Temporary Incapacity for Work Duration (TIWD), the number of right holders and the number of permanent incapacity income recipients. Table 4 shows the distribution of these data by province. These data include only data reported and officially registered cases. There was no information about the unreported and unregistered data. That is another limitation of the study.

In this study, the DEA Solver add-on included in the Excel program was used for the efficiency evaluation of the data compiled and tabulated for data envelopment analysis. DEA Solver is a very suitable program for the evaluation of data to a maximum of 30 DMUs and it does not need to be purchased separately as it is included in the Excel program. As a method in the analysis, six different model approaches shown in Table 1 were preferred.

Table 1 DEA models used with UO
Tablica 1. Primijenjeni DEA modeli s neželjenim izlazima

Item No	Method / *Metoda*	Source / *Izvor*
1	Ignoring undesirable factors / *ignoriranje nepoželjnih čimbenika*	Faere *et al*. (1989)
2	Treating unwanted outputs (inputs) as inputs (outputs) (U=-u) *tretiranje neželjenih izlaza (ulaza) kao ulaza (izlaza) (U=-u)*	Dyckhoff and Allen (2001)
3	Improvement of undesirable factors in nonlinear DEA model *poboljšanje nepoželjnih čimbenika u nelinearnom DEA modelu*	Faere *et al*. (1989)
4	Applying a nonlinear monotone decreasing transform (U=1/u) to undesirable factors *primjena nelinearne monotone padajuće transformacije (U=1/u) na nepoželjne čimbenike*	Dyckhoff and Allen (2001)
5	A linear monotone decreasing transform to deal with undesirable factors *linearna monotona padajuća transformacija za rješavanje nepoželjnih čimbenika*	Seiford and Zhu (2002)
6	Directional distance function approximation *aproksimacija funkcije usmjerene udaljenosti*	Hua and Bian (2007)

Table 2 Distribution by provinces with intense furniture industry, number of enterprises and employees by provinces
Tablica 2. Pregled pokrajina u kojima je industrija namještaja bila intenzivna te broj poduzeća i zaposlenika u njima

Item No	**Provinces** *Pokrajine*	**Enterprise (General)** *Poduzeća (općenito)*	**Employee (General)** *Zaposlenici (općenito)*	**Enterprise (Furniture)** *Poduzeća (namještaja)*	**Employee (Furniture)** *Zaposlenici (u proizvodnji namještaja)*
1	İstanbul	537,982	4,130,578	5,073	29,855
2	Bursa	78,554	682,103	2,388	25,918
3	Kayseri	32,423	220,267	1,206	23,511
4	Ankara	144,459	1,116,500	2,481	13,343
5	İzmir	131,243	889,856	1,668	10,867
6	Antalya	74,551	503,569	856	4,954
7	Kocaeli	46,580	500,326	360	2,981
8	Sakarya	23,059	179,564	314	2,472
9	Konya	46,424	306,968	455	2,336
10	Gaziantep	33,702	299,808	263	2,273
11	Adana	41,767	301,270	498	2,240
12	Samsun	26,118	166,688	426	1,981
13	Hatay	24,092	168,446	397	1,763
14	Düzce	8,127	70,727	126	1,578
15	Eskişehir	20,888	170,464	208	1,574
16	Mersin	37,791	248,307	374	1,546
17	Çanakkale	14,343	85,437	128	1,410
18	Manisa	27,748	238,198	185	1,333
19	Denizli	25,811	186,613	253	1,151
20	Sivas	10,015	72,591	133	1,102
21	Diyarbakır	17,017	151,506	118	1,087
22	Balıkesir	29,698	172,904	219	1,083
23	Ordu	13,494	83,907	182	1,077
24	Tekirdağ	24,019	268,967	126	1,073
25	Trabzon	19,753	116,044	227	1,061
26	Zonguldak	11,269	86,844	119	741
27	Kahramanmaraş	16,281	144,710	118	673
28	Aydın	27,380	149,941	145	612
29	Malatya	12,695	99,419	115	552
30	Muğla	36,301	182,747	170	523
Subtotal		1,593,584	11,995,269	19,331	142,670
Sum Total		1,891,512	14,314,313	21,758	154,829

Table 3 Distribution of workplace physicians, other healthcare personnel, occupational physicians and occupational safety centers and OHSSs by provinces
Tablica 3. Raspodjela liječnika medicine rada i drugoga medicinskog osoblja, liječnika medicine rada i centara za zaštitu na radu te OHSS-ova prema pokrajinama

Item No	**Provinces** / *Pokrajine*	**WP**	**OHP**	**WPOSC**	**OHSS (A)**	**OHSS (B)**	**OHSS (C)**
1	İstanbul	7,108	3,808	414	3,112	5,854	7,996
2	Bursa	1,290	659	85	688	846	1,739
3	Kayseri	430	203	29	221	278	629
4	Ankara	2,794	1,666	295	1,744	1,900	4,195
5	İzmir	2,138	1,304	164	1,146	1,319	2,743
6	Antalya	945	457	39	402	442	1,038
7	Kocaeli	766	873	53	483	1,017	1,785
8	Sakarya	350	208	34	148	249	437
9	Konya	722	354	38	285	400	816
10	Gaziantep	523	184	43	173	254	623
11	Adana	653	382	55	341	633	1,265
12	Samsun	398	312	16	183	232	600
13	Hatay	349	223	10	152	281	647
14	Düzce	142	70	5	50	83	143
15	Eskişehir	408	341	37	213	300	668
16	Mersin	513	303	28	202	391	926

Table 3 Continuation
Tablica 3. Nastavak

Item No	Provinces / *Pokrajine*	WP	OHP	WPOSC	OHSS (A)	OHSS (B)	OHSS (C)
17	Çanakkale	176	181	8	87	146	337
18	Manisa	354	322	18	185	258	628
19	Denizli	422	247	29	222	218	458
20	Sivas	177	129	8	78	140	284
21	Diyarbakır	443	217	39	108	265	595
22	Balıkesir	369	322	22	176	307	556
23	Ordu	134	130	9	56	90	272
24	Tekirdağ	426	264	10	253	364	608
25	Trabzon	274	224	26	129	209	513
26	Zonguldak	208	123	46	222	180	449
27	Kahramanmaraş	272	137	10	101	162	426
28	Aydın	364	218	31	171	189	395
29	Malatya	261	132	24	116	135	374
30	Muğla	388	250	14	182	214	425
TOTAL		28,822	17,267	1,639	13,289	20,453	40,092

*WP – Workplace physicians, OHP – Other health personnel, WPOSC – Workplace physician and occupational safety center
**WP – liječnici medicine rada, OHP – ostalo medicinsko osoblje, WPOSC – liječnici medicine rada i centri za zaštitu na radu*

Table 4 Distribution of data on work accidents and occupational diseases by provinces
Tablica 4. Pregled podataka o nezgodama na radu i profesionalnim bolestima prema pokrajinama

Item No	Provinces *Pokrajine*	Employee *Zaposlenici* (WA)*	Employee *Zaposlenici* (OD)*	Death *Smrt* (WA)	TIWD* (Total / *Ukupno*)	Number of right holders *Broj nositelja prava* (WA)	Number of permanent incapacity recipients *Broj osoba s trajnom nesposobnošću*
1	İstanbul	109,695	186	199	697,844	641	12,757
2	Bursa	23,075	29	48	227,941	175	3,715
3	Kayseri	10,274	9	25	87,551	83	1,850
4	Ankara	30,286	54	88	214,421	262	4,578
5	İzmir	34,618	115	55	338,322	213	5,026
6	Antalya	23,483	2	47	118,221	125	1,757
7	Kocaeli	25,944	133	41	292,383	176	3,570
8	Sakarya	7,555	30	6	64,564	50	1,087
9	Konya	7,413	6	36	72,975	162	1,662
10	Gaziantep	6,048	2	28	75,511	125	1,379
11	Adana	6,550	3	31	64,036	166	1,782
12	Samsun	3,082	0	5	25,265	67	1,490
13	Hatay	2,257	5	16	32,315	124	1,299
14	Düzce	2,567	11	4	28,355	34	467
15	Eskişehir	7,283	15	14	66,524	25	979
16	Mersin	4,073	0	31	49,179	111	1,316
17	Çanakkale	2,207	11	7	24,751	35	353
18	Manisa	14,128	25	13	153,599	59	1,393
19	Denizli	6,772	2	20	79,036	82	1,012
20	Sivas	1,606	2	12	11,853	41	580
21	Diyarbakır	1,880	0	20	18,201	180	1,022
22	Balıkesir	4,135	5	21	46,080	82	1,108
23	Ordu	1,401	0	7	10,810	68	1,075
24	Tekirdağ	12,188	19	14	128,777	86	1,324
25	Trabzon	1,640	1	15	16,047	63	663
26	Zonguldak	5,670	48	10	77,932	386	6,150
27	Kahramanmaraş	2,426	1	7	25,079	82	966
28	Aydın	4,500	4	18	50,040	67	1,005
29	Malatya	1,830	0	11	14,122	59	1,393
30	Muğla	7,322	6	23	55,606	35	616
TOTAL		371,908	724	872	3,167,340	3,864	63,374

*WA – Work accident, OD – Occupational diseases, TIWD – Temporary incapacity for work duration
**WA – nezgoda na radu, OD – profesionalne bolesti, TIWD – trajanje privremene nesposobnosti za rad*

Table 5 Models used in DEA analysis and their inputs and outputs
Tablica 5. Modeli primijenjeni u DEA analizi te ulazi i izlazi u tim modelima

Model No *Broj modela*	**Input** *Ulaz*	**Output** *Izlaz*	**Undesirable output** *Neželjeni izlaz*
Model 1	G1, G2	O1, O2, O3	Ignored / *zanemareno*
Model 2	G1, G2, U1, U2, U3, U4, U5, U6	O1, O2, O3	As an input / *kao ulaz*
Model 3	G1, G2	O1, O2, O3, U1, U2, U3, U4, U5, U6	As an improved output / *kao poboljšani izlaz*
Model 4	G1, G2	O1, O2, O3, U1, U2, U3, U4, U5, U6	As a transformed output (a nonlinear monotone decreasing) *kao transformirani izlaz (nelinearno monotono padanje)*
Model 5	G1, G2	O1, O2, O3, U1, U2, U3, U4, U5, U6	As a transformed output (a linear monotone decreasing) *kao transformirani izlaz (linearno monotono padanje)*
Model 6	G1, G2	O1, O2, O3, U1, U2, U3, U4, U5, U6	As a transformed output by (directional distance function approximation) *kao transformirani izlaz (aproksimacija funkcije usmjerene udaljenosti)*

The abbreviations in Table 5 can be listed as follows:

G1 (number of enterprises), G2 (number of employees insured), O1 (total number of WP and OHP), O2 (number of WPOSC), O3 (A, B, and C-class OHSSs), U1 (number of employees who experienced WA), U2 (number of employees who experienced OD), U3 (number of deaths resulting from WA and OD), U4 (total temporary incapacity for work duration), U5 (number of beneficiaries) and U6 (number of permanent incapacity income recipients).

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

Table 6 shows the distribution of the efficiency scores according to the provinces obtained as a result of DEA, in which UO are modeled with the six different models shown in Table 1.

First, the model of Faere *et al*. (1989) was used. In the model, UOs are ignored. In the classical DEA, the increase of the outcomes is assumed by keeping the inputs. As a result of the analysis, Aydın, Diyarbakır, Muğla and Zonguldak came to the fore as the most effective cities. Kayseri was the most ineffective province with an efficiency score of 0.1143. The average efficiency of the states is 0.6199.

In the second model, UOs were included in the analysis as inputs, as proposed by Dyckhoff and Allen (2001). In the DEA analysis, UOs were defined as inputs by inverting them according to the addition process (U=-u). As a result of the analysis, Aydın, Diyarbakır, Manisa, Muğla, Tekirdağ and Zonguldak were seen as the most effective provinces. With an efficiency score of 0.1443, Kayseri was again the most ineffective city. The average efficiency score is 0.6339.

Seiford and Zhu (2002) stated that including UO as input into DEA like in the second model does not reflect the actual production process. For this reason, in their research, they predicted that UO in Faere *et al*. (1989) nonlinear DEA model should be included in the analysis by improving them with data transformation. Therefore, in the third model, according to this approach, UOs were improved and included in DEA as output. According to the DEA result, Aydın, Diyarbakır, Kahramanmaraş, Malatya, Muğla and Zonguldak came to the fore as effective provinces. Kayseri was in the last place in the efficiency evaluation as the most ineffective city with an efficiency score of 0.1071. In this model, the average efficiency score is 0.69100.

In the fourth model, as suggested by Dyckhoff and Allen (2001), undesirable factors are transformed into desired outputs by applying a nonlinear monotonous decreasing conversion (U=1/u). Then, they are included in DEA as natural outputs. In Table 6, where the results of the analysis are shown, Aydın. Çanakkale, Diyarbakır, Kahramanmaraş, Malatya, Muğla, Tekirdağ and Zonguldak are seen as the most effective cities. With an efficiency score of 0.1443, Kayseri is in the last place as the most ineffective province. The average efficiency score was calculated as 0.7561.

A linear monotone decreasing transformed model was applied as the fifth model. This model is suggested by Seiford and Zhu (2002). The undesirable factors were improved and added in DEA with natural outputs. As seen in Table 6, Aydın, Çanakkale, Diyarbakır, Düzce, Eskişehir, Kahramanmaraş, Malatya, Muğla, Ordu, Sakarya, Samsun, Sivas, Trabzon and Zonguldak were the most efficient provinces. While the average efficiency score of the provinces was calculated as 0.7871, Kayseri was seen as the most inefficient city with an efficiency score of 0.1443.

According to Yang *et al*. (2008), UOs can be transformed into desired outcomes by curing them with the Shephard distance function. Hua and Bian (2007) stated in their study that undesired factors can be transformed into desired outputs by using the directional distance function and that DEA results can better

Table 6 Distribution of efficiency scores of six different models calculated with DEA by province
Tablica 6. Rezultati učinkovitosti šest različitih modela izračunanih uz pomoć DEA-e prema pokrajinama

Item No	Province *Pokrajina*	**Efficiency scores** / *Rezultati učinkovitosti*					
		Model 1	Model 2	Model 3	Model 4	Model 5	Model 6
1	İstanbul	0.4569	0.4569	0.4799	0.4569	0.4569	0.4569
2	Bursa	0.1660	0.1660	0.1554	0.1660	0.1660	0.1660
3	Kayseri	0.1443	0.1443	0.1071	0.1443	0.1443	0.1447
4	Ankara	0.4685	0.4685	0.4875	0.4685	0.4685	0.5391
5	İzmir	0.4840	0.4846	0.4426	0.4840	0.4840	0.4878
6	Antalya	0.3405	0.3405	0.3566	0.4466	0.3483	0.3432
7	Kocaeli	0.8905	0.9034	0.9911	0.8905	0.8905	0.9030
8	Sakarya	0.3655	0.3687	0.4010	0.7491	1	0.2488
9	Konya	0.5228	0.5228	0.5411	0.5228	0.5228	0.1861
10	Gaziantep	0.5026	0.5026	0.5054	0.8097	0.5073	0.5026
11	Adana	0.7013	0.7013	0.7496	0.7316	0.8148	0.8365
12	Samsun	0.3895	0.3895	0.3926	0.6386	1	1
13	Hatay	0.4056	0.4057	0.4492	0.5077	0.5582	0.4766
14	Düzce	0.2636	0.2719	0.9648	0.8700	1	0.2643
15	Eskişehir	0.7346	0.7346	0.7302	0.9681	1	1
16	Mersin	0.6504	0.6504	0.7098	0.6782	0.8925	0.8797
17	Çanakkale	0.7793	0.7835	0.9560	1	1	1
18	Manisa	0.7056	1	0.7280	0.8148	0.7766	0.7056
19	Denizli	0.6313	0.6313	0.6367	0.7420	0.7216	0.6493
20	Sivas	0.5439	0.5439	0.9079	0.9311	1	0.6106
21	Diyarbakır	1	1	1	1	1	1
22	Balıkesir	0.8515	0.8519	0.7515	0.8549	0.9343	0.8762
23	Ordu	0.3862	0.3863	0.6442	0.9193	1	0.4213
24	Tekirdağ	0.9054	1	1	1	0.9275	0.9073
25	Trabzon	0.6692	0.6692	0.6416	0.8876	1	1
26	Zonguldak	1	1	1	1	1	1
27	Kahramanmaraş	0.6876	0.6876	1	1	1	0.9259
28	Aydın	1	1	1	1	1	1
29	Malatya	0.9510	0.9510	1	1	1	1
30	Muğla	1	1	1	1	1	1
Average scores *Srednje vrijednosti rezultata*		0.6199	0.6340	0.6910	0.7561	0.7871	0.6844

reflect the actual production process. This approach model of Hua and Bian (2007) was used as the sixth model. According to the DEA result in Table 6, Aydın, Çanakkale, Diyarbakır, Eskişehir, Malatya, Muğla, Samsun, Trabzon and Zonguldak were the most influential cities. Kayseri took the last place again as the most ineffective province with an efficiency score of 0.1447. The average efficiency score is 0.6844.

Coping with UO is very important in DEA. In this study, the data with UO were improved and transformed according to six different models in Table 1. Then, they were included in DEA and subjected to an efficacy evaluation. Table 6 shows that Model 2, Model 3 and Model 6 produce similar results in terms of DMUs. When Table 6, Table 2, Table 3 and Table 4 are evaluated together, it can be observed that optimal results in terms of efficiency are obtained with the Model 6. Hua and Bian (2007) state that in this model, since the direction vector is strongly affected by the weights determined by the users, optimum results for DMUs can be achieved more effectively.

According to the OHS Law, an OHSS must be assigned to every 1000 employees for the less hazardous class, every 500 employees for the dangerous group, and every 250 employees for the very precarious class. Likewise, there is a requirement to appoint a workplace physician for every 2000 employees in the less dangerous business class, every 1000 employees in the hazardous class, and every 750 employees in the very precarious enterprise. According to the current law, these numbers in the sector are sufficient.

The 30 provinces within the scope of the study are about 88 % of the total number of enterprises or employees in the furniture sector. Also, they cover approximately 84 % of the total number of enterprises or employees in Turkey (Table 2). Istanbul is the city with the highest number of enterprises and employees countrywide. One out of every 38 employees in Istanbul had a work accident. One out of every 20757 employees died in a work accident (Table 2 and 4). Besides, the number of employees per one WP or OHP in Istanbul is approximately 380. The number of enterprises per workplace physician and occupational safety center is 1300. The number of enterprises per OHSS is 32, and the number of employees is 244 (Table 2 and 3).

As a result of DEA, Aydın, Diyarbakır, Muğla, and Zonguldak emerge as the most effective provinces in all models. Zonguldak is a significant city in both the furniture and mining sector. One of every 16 employees in Zonguldak has a work accident. One of every 8685 employees died in a work accident (Table 2 and 4). The employees' number per WP or OHP in Zonguldak is about 263. The number of enterprises per workplace physician and occupational safety center is determined as 245 (Table 2 and 3).

The death rate due to work accidents or occupational diseases in Turkey is 8.01 per 100,000 employees. The average of 30 provinces is 7.27. In the European Union (EU), the average rate is 1.77. In Romania, where the highest number of deaths occurred, this rate is 4.33, or in Luxembourg 4.22. When examining the ratio of work accidents, while it is 2951 per 100,000 in Turkey, the EU average is 1650. This rate is around 3450 in France. The average of 30 provinces within the scope of the research is 3100 (Eurostat, 2021), (Table 2 and 4).

4 CONCLUSIONS
4. ZAKLJUČAK

Previous studies show that the work environments of enterprises in the furniture sector are not suitable enough in terms of OHS. For example, according to Koç *et al*. (1998), 59 % of the enterprises were not adequately ventilated. Likewise, Karademir and Koç (2020) found in their study that 30 % of the enterprises were completely without air, while 40 % were not properly ventilated. Although 20 years elapsed between the two studies, it seems that the work environments of the enterprises have not been brought to a good point in terms of OHS. At this point, it is known that the sector enterprises should be effectively supervised and kept under control, especially in terms of employees' safety and health.

In the study, according to Model 6, 9 of 30 provinces were found to be effective and 21 of them were found to be ineffective. The most ineffective is Kayseri with an efficiency value of 0.1447. At this point, Kayseri should reduce the number of its employees by 10 % compared to Diyarbakır, for example, and 14 % compared to Zonguldak. Likewise, it should increase the number of WPOSC by 40 % compared to Diyarbakır and by 52 % compared to Zonguldak.

OHSSs' field tours, risk analyses, environmental measurements, periodic inspections and workups, and feedback from employees are very important for accelerating the detection and improvement of malfunctions in the workplace (Ulutaş. 2016). Reports and records kept by the occupational physician, other health personnel, occupational physician and occupational safety centers and OHSSs increase the accuracy and reliability of statistical data on occupational accidents and diseases.

Although the number of OHSSs seems sufficient according to the current laws, the data in the field shows that these numbers are insufficient to prevent OADs. Despite being the most efficient province in all analyses, the number of employees per WP is high even in Zonguldak. Herein, according to the hazard classes, work accidents that have occurred from the past to the present should be examined. According to accident frequency, causes of accident and death statistics, a sectoral status evaluation should be made in the enterprises. Optimal numbers should be determined according to assessment, and workloads should be recalculated. The relevant articles of the OHS law should be updated.

Increasing numbers and reducing workloads will play a significant role in preventing occupational accidents and deaths. However, it is not enough by itself. At this point, training programs with the participation of all stakeholders such as business owners, white and blue-collar employees, WP, OHP and OHSSs should be planned and periodically implemented.

Up till today, full cooperation between industry and universities has not been established in practice. For having a good collaboration, a legal basis must be established between the parties, and this legal ground must be a forcing factor. Universities must be involved as a partner in projects preparation, training, auditing and consultancy services.

According to Lombardi *et al*. (2019), learning from accidents is one of the steps that can be taken to prevent possible future accidents. The most substantial step for the learning approach is to create a detailed accident database on a sectoral basis across the country. The variables of all work accidents that occur should be determined, and the variables interrelationships should be calculated. In this way, the most probable accident causality models for each accident should be revealed and the accident proximal and distant specific features should be defined. As a result, risk profiling for each accident will provide foremost and practical information for the safety precautions.

As to the European Agency for Occupational Safety and Health (OSHA), the key to sustainable economic recovery is the employees' well-being. Moreover, OSH is seen as a significant aspect of responsible and sustainable development of rapidly growing new technologies (nanotechnologies and green jobs). Therefore, there is a need to establish an OHS culture, namely a cultural evolution, to increase the value of occupational health among an extensive range of stakeholders, from decision-makers to OHS experts, employers, workers and their representatives, and ultimately the whole society (Gagliardi. 2012).

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Thermal Modification Intensity of Heat-treated Poplar Wood. Part 2: Characterization and Predication from Outside to Core Layers

Intenzitet toplinske modifikacije topolovine. Dio 2: Karakterizacija i predviđanje od vanjskoga prema unutarnjim slojevima

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ABSTRACT • *Based on the previous study of the heat-treated wood at 0-3 mm surface layer, this study focuses on the transition of thermal modification intensity on 160-220 °C heat-treated poplar from surface to core layers. The color change was evaluated by CIELAB, and surface hardness was detected via Shore D (HD) and pressing ball method (H_R); furthermore, the FT-IR was applied to detect the thermal degradation of wood components. The results show that the degradation of cell wall components in the surface layer of heat-treated poplar wood is greater than that in the core layers, and the thermal degradation intensity of the surface layer of the heat-treated poplar wood is greater than that of the other inner layers. Surface color and hardness properties of the heat-treated wood between S_0 and S_1-S_5 test surfaces were significantly different under the same heat treatment conditions; the surface hardness showed an increasing trend, and the H_R value of the H_{220-2} core layer was 105.71 % higher than that of the surface layer. Heat treatment temperature is the main factor affecting the property of wood surface, while the effect of duration is smaller. The hemicellulose content change was mainly related to the degradation intensity on heat-treated wood at different locations. An accurate prediction model of surface color, hardness, and other properties of the heat-treated wood at different locations was established by Table Curve 3D software.*

KEYWORDS: *heat treatment; modification intensity; transition; hardness; color; surface layer; inner layers; poplar wood*

SAŽETAK • *Prethodno istraživanje toplinski modificiranog drva odnosilo se na površinski sloj od 0 do 3 mm, a ovo se istraživanje bavi prijelazom intenziteta toplinske modifikacije pri 160 – 220 °C s površinskoga prema*

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unutarnjim slojevima topolovine. Za evaluaciju promjene boje primijenjen je CIELab sustav, a tvrdoća površine utvrđena je Shore D (HD) metodom i metodom utiskivanja kuglice (HR). Osim toga, procjena toplinske razgradnje spojeva u drvu napravljena je FT-IR spektroskopijom. Rezultati pokazuju da je razgradnja stanične stijenke spojeva drva u površinskom sloju toplinski modificirane topolovine veća nego u središnjim slojevima te da je intenzitet toplinske razgradnje površinskog sloja topolovine veći od intenziteta razgradnje ostalih unutarnjih slojeva. Boja površine i svojstva tvrdoće toplinski modificiranog drva između S0 i S1-S5 ispitnih površina značajno su se razlikovali uz jednake uvjete toplinske modifikacije. Tvrdoća površine pokazala je rastući trend, a HR vrijednost H220-2 unutarnjeg sloja bila je 105,71 % vrijednosti površinskoga. Temperatura toplinske modifikacije glavni je čimbenik koji utječe na svojstva površine drva, dok je utjecaj trajanja modifikacije manji. Promjena sadržaja hemiceluloze uglavnom je povezana s intenzitetom razgradnje toplinski modificiranog drva na različitim lokacijama. Točan model predviđanja boje i tvrdoće površine te drugih svojstava toplinski modificiranog drva na različitim mjestima uspostavljen je uz pomoć Table Curve 3D softvera.

KLJUČNE RIJEČI: *toplinska modifikacija; intenzitet modifikacije; prijenos; tvrdoća; boja; površinski sloj; unutarnji slojevi; topolovina*

1 INTRODUCTION
1. UVOD

Wood, a renewable and green material, has unique material properties and excellent visual characteristics such as lightweight, good toughness, high plasticity, beautiful texture, rich color, and easy processing (Wang *et al*., 2019; Ansell, 2015). Wood is now widely used in the field of furniture and construction (Gurleyen *et al*., 2017; Yang *et al*., 2020). Due to the deepening conflict between the growing wood processing industry and the scarce natural forest resources, China has vigorously promoted the planting of fast-growing wood to alleviate it (Liu *et al*., 2019; Yang *et al*., 2020). According to the results of the Ninth National Forest Resources Inventory Report of the National Forestry and Grassland Administration (2018), the dominant tree species (groups) in China's artificial arbor forest area are *Cunninghamia lanceolata*, *Populus* spp., *Eucalytus* spp., *Larix* spp., *Pinus massoniana*, *Pinus tabuliformis*. In 2021, China's plantation area accounted for 73 % of the global plantation area (Wang and Shen, 2022). Poplar, as one of the major plantation woods in China, can provide a vast quantity of raw materials for processed products such as paper making and wood-based panels. Although poplar has a relatively short growth cycle and gives a high yield, it usually exhibits some dimensional instability and low-density problems, which limits its efficient utilization and large-scale application as a solid wood material (Pan *et al*., 2019; Yang *et al*., 2020).

Modification of fast-growing poplar wood can effectively improve its properties, and increase its economic benefits (Kamperidou *et al*., 2012; Shi and Li, 2014). The modification refers to the treatment of wood materials by chemical, physical or biological methods to change the internal composition or structure of the wood to enhance some aspects of wood performance (Hill, 2006), of which heat treatment is an environmentally friendly technology without any addition of chemicals. Heat-treated wood is popular for its excellent performance, green and safe process, and has developed remarkably (Gu *et al*., 2020).

Property changes of heat-treated wood and its modification mechanism have been widely studied. Most of the existing research is focused on the overall performance or surface properties of heat-treated wood. However, the process of thermal treatment is commonly applied to sawn wood of especially large dimensions, and the properties of the treated wood on different locations in the thickness direction vary with the progression of the heat treatment and mass transfer. Kadem *et al*. (2020) performed a transient analysis of heat and mass transfer processes during heat treatment and found that, as the sample thickness increased, there was a gradual difference in the temperature at the center of the sample, attributed to the lower thermal conductivity of the wood. Wang (2011) studied the thermal effects of vacuum heat treatment of rough-barked eucalyptus wood and found that the sample thickness has a significant effect on heat absorption per unit mass and that thickness differences lead to uneven heat transfer in thin slices. The modification intensity of wood is unequal at different locations or layers in the direction of thickness. Our previous study uncovered the surface properties change, including surface hardness, roughness, and abrasion of heat-treated poplar, and the discrepancy of those surface properties of heat-treated poplar at the 3 mm surface layer, showing that the most thermal degraded part located in the 1 mm outer surface layer, and the surface hardness, wet-ability, and bonding strength of the heat-treated wood could be remarkably enhanced by removing the outer surface layer (Chu *et al*., 2020).

The performance change of heat-treated wood from surface to core layers is still unknown. This study aims to reveal the variation of heat-treated poplar on different test layers located from the surface to the center core layers in the direction of thickness. Properties including surface color, Shore and pressing ball hardness, and cell wall component changes were taken into consideration. Besides, the FT-IR and XRD were applied to detect the degradation of wood components and explore the mechanism of the modification intensity variation from outside to core layers, which provided the basis for evaluating the performance of the heat-treated poplar wood.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Sample preparation
2.1. Priprema uzoraka

The 12-year-old Zhonglin 46 poplars were selected, and the straight part of the air-dried lumber was cut into 200 mm × 150 mm ×30 mm boards. The moisture content of all the samples was around 10 %.

The heat-treated poplar wood was prepared by a self-made high temperature experiment device. The poplar wood samples were evenly put in the device, and the temperature inside the box was raised to 130 °C at a heating rate of 20 °C·min^{-1} and maintained for 30 minutes, and then raised to the target temperature (160 °C, 190 °C, and 220 °C) at a heating rate of 10 °C·min^{-1} and maintained for 2 h or 4 h. After that, the samples were removed to the climate chamber to achieve an equilibrium moisture.

Then the modified poplar wood was cut with a 3 mm gradient using a precise sliding saw on the tangential section, forming 6 different testing surfaces (S_0, S_1, S_2, S_3, S_4, S_5), and 5 testing layers (T_1, T_2, T_3, T_4, T_5), as shown in Figure 1.

2.2 Measurement of surface color and hardness changes
2.2. Mjerenje promjene boje i tvrdoće površine

According to the CIE1976LAB standard colorimetric system, a precision colorimeter (HP-200, Shenzhen Hampoo) was used to obtain the surface color parameters of different heat-treated wood layers, namely S_0 to S_5 of wood in each group. Five sites were randomly selected for each sample, and each group was repeatedly tested three times, and then the average value of the results was taken. Before the measurement, all the samples were subjected to humidification balance treatment in a conditioning chamber with a temperature of 20 °C and relative humidity of 65 %. Color difference value ΔE^* was calculated according to Eqs. (1) to (4).

$$\Delta L = L_t - L \quad (1)$$

$$\Delta a = a_t - a \quad (2)$$

$$\Delta b = b_t - b \quad (3)$$

$$\Delta E^* = \left[(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2\right]^{\frac{1}{2}} \quad (4)$$

Where L_t, a_t, b_t are the chromatic parameters of the poplar wood after HT, and L, a, b are the chromatic parameters of wood before HT.

Press ball hardness test adopts a high-precision double-column universal testing machine (AG-X plus, Shimadzu, Japan), using a hemispherical steel indenter mold, referring to the national standard GB/T1941-2009 wood hardness test method. The hemispherical steel indenter was pressed into the surface of the balanced specimen at a uniform speed of 1 mm/min, the pressing load was marked as P, and each experimental group was repeated 15 times. Based on the indentation load of the untreated Poplar wood, the pressing ball hardness (H_R) was calculated with Eq. (5) (Yu *et al.,* 2020).

$$H_R = \frac{P}{P_0} \quad (5)$$

Where P is the load at 1 mm on the surface of the heat-treated wood (N) and P_0 is the load at 1 mm on the surface of the untreated wood (N).

A Shore D hardness tester (HS-D, Wenzhou Baoyi) was used to test the surface hardness of the balanced samples. Ten positions were randomly selected on the surface of each sample for testing, and each experimental group was repeated three times. The surface hardness value (HD) was calculated as shown in Eq. (6).

$$HD = \frac{100 - L}{0.025} \cdot 100\% \quad (6)$$

Where L is the displacement of the Shore D hardness tester needle tip relative to the pressure foot surface when the tester pressure foot is completely attached to the surface of the sample, unit: mm.

2.3 Chemical analysis
2.3. Kemijske analize

According to Figure 1, the parts T_1 to T_5 of each sample were smashed into 100-120 meshed wood powder, and the absolutely dried samples were selected

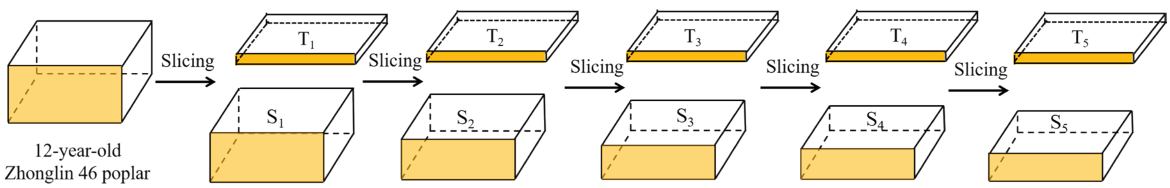

Figure 1 Flow chart of layering of heat-treated poplar board
Slika 1. Dijagram toka raslojavanja ploče od toplinski modificirane topolovine

for chemical analysis. The Fourier transform infrared spectra were collected in a Tensor-II (Bruck, Germany), the wave number interval was 400-4000 cm^{-1}, and the resolution was 4 cm^{-1}. Each sample was scanned and analyzed 32 times.

2.4 Data analysis and modeling of surface properties
2.4. Analiza podataka i modeliranje površinskih svojstava

For the surface color and hardness, multiple comparisons were first subjected to analysis of variance (ANOVA), and significant differences between the average value of untreated and heat-treated samples were determined using Duncan's multiple range test. Besides, differences between S_0~S_5 test surfaces of the wood treated under the same treatment condition were also determined, and then the Curve 3D software was used to establish the prediction models of ΔL^*, ΔE^*, H_R, and *HD* values.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Color and hardness of different test surfaces on heat-treated poplar wood
3.1. Boja i tvrdoća različitih ispitnih površina toplinski modificirane topolovine

The main purpose of heat treatment is to change the color of the wood, improve the single and light color of the fast-growing wood surface, and therefore enhance the decorative properties (Lei *et al.*, 2021). The L^*, a^*, and b^* values of the test surfaces (S_0-S_5) at the position of different thicknesses on 160-220 °C heat-treated poplar wood are shown in Figure 2.

From Figure 2, it can be seen that the lightness value L^* of the heat-treated wood tends to decline with the increase of heat treatment temperature and duration, while the red-green value a^* and yellow-blue value b^* increase first, and then decline. The changing trend of chromatic parameters is consistent with the

Table 1 ANOVA analysis of color and hardness of different test layers of heat-treated poplar wood
Tablica 1. ANOVA analiza boje i tvrdoće različitih ispitnih slojeva toplinski modificirane topolovine

Source of difference / *Izvor razlike*		Sum of squares of deviations (SS) / *Zbroj kvadrata odstupanja (SS)*	Degree of freedom (*df*) / *Stupanj slobode (df)*	Mean square (*MS*) / *Srednji kvadrat (MS)*	*F*-value / *F-vrijednost*	*P*-value / *P-vrijednost*
ΔL^*	HT temperature / *HT temperatura*	60035.16	2	30017.58	2814.75	1.40E-131
	HT duration / *HT trajanje*	603.13	1	603.13	56.56	2.95E-12
	Test layer location / *lokacija ispitnog sloja*	1179.14	5	235.83	22.11	4.66E-17
	Deviation / *odstupanje*	1823.61	171	10.66		
	Summation / *zbroj*	173782.04	180			
ΔE^*	HT temperature / *HT temperatura*	57552.64	2	28776.32	2934.01	4.45E-133
	HT duration / *HT trajanje*	601.92	1	601.92	61.37	4.78E-13
	Test layer location / *lokacija ispitnog sloja*	1038.93	5	207.79	21.19	1.86E-16
	Deviation / *odstupanje*	1677.14	171	9.81		
	Summation / *zbroj*	188136.76	180			
H_R	HT temperature / *HT temperatura*	1.17	2.00	0.59	36.53	8.13E-15
	HT duration / *HT trajanje*	0.67	1.00	0.67	41.70	4.70E-10
	Test layer location / *lokacija ispitnog sloja*	7.38	5.00	1.48	92.19	5.02E-57
	Deviation / *odstupanje*	4.47	279.00	0.02		
	Summation / *zbroj*	241.60	288.00			
HD	HT temperature / *HT temperatura*	79.32	2.00	39.66	11.88	1.12E-05
	HT duration / *HT trajanje*	56.45	1.00	56.45	16.91	5.15E-05
	Test layer location / *lokacija ispitnog sloja*	932.93	5.00	186.59	55.91	3.97E-40
	Deviation / *odstupanje*	931.09	279.00	3.34		
	Summation / *zbroj*	638614.85	288.00			

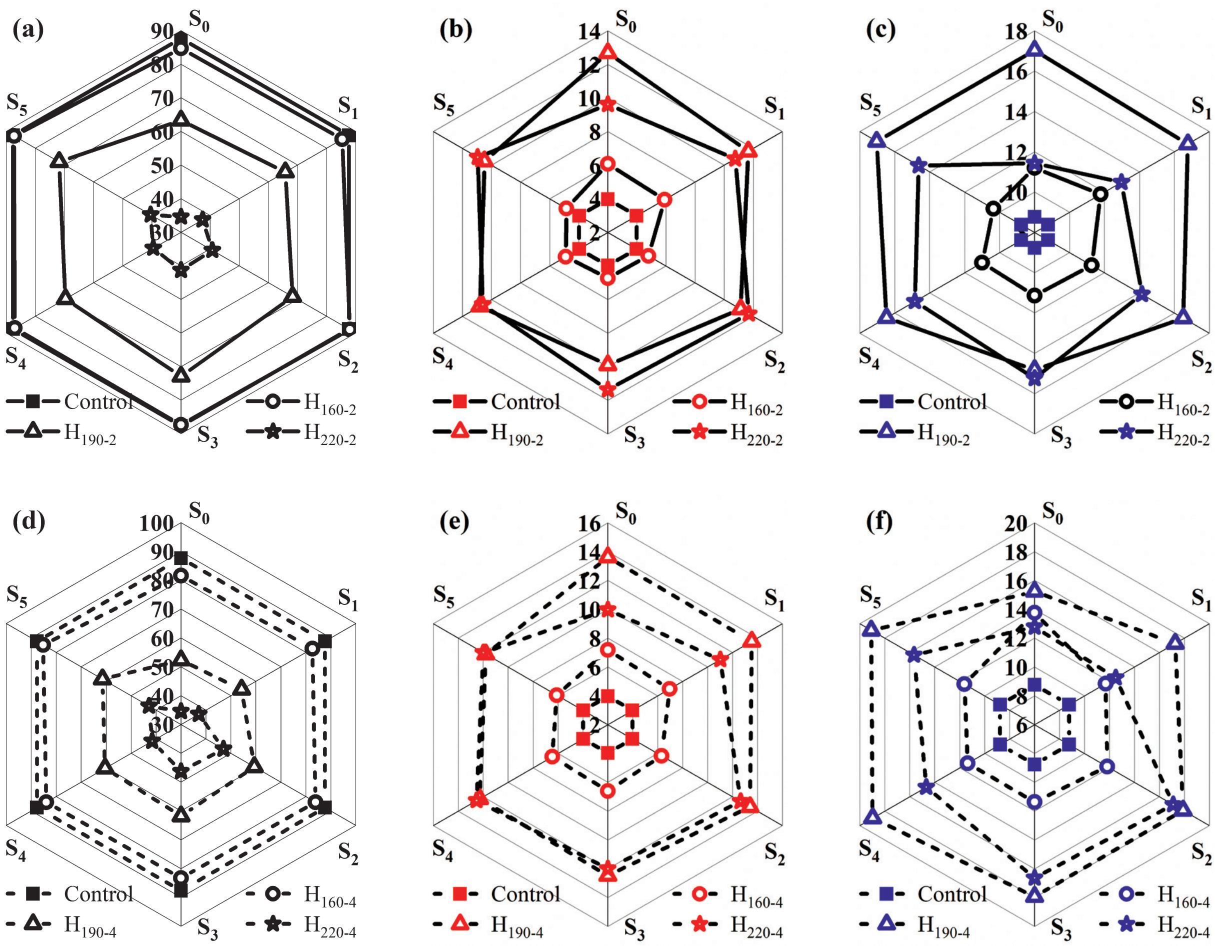

Figure 2 Chromatic parameters of different test surfaces on heat-treated poplar wood
Slika 2. Kromatski parametri različitih ispitnih površina toplinski modificirane topolovine

previous research (Hill, 2006; Chu *et al*., 2016; Gao *et al*., 2019). Interestingly, for all the heat-treated wood, the L^* value of the test surface increased as the position changed from S_0 to S_5, and this increment of the L^* value varies with the HT temperature and duration. For 160 °C and 190 °C heat-treated wood, the a^* value decreased as the position changed from S_0 to S_5, the greatest decreasing amplitude being 22.2 % in the H_{160-2}. The b^* value increased from S_0 to S_5 when the heat treatment temperature was 190 °C and 220 °C; the b^* value of the H_{190-2} on S_0 surface was 28 % higher than that of the S_5 surface. It could prove that the heat-treated wood was not uniform regarding surface color in the direction of thickness, and the properties of real processing surface after cutting or sanding needed to be further studied. The lightness difference ΔL^* between the S_0 and S_5 of heat-treated wood increased with the HT temperature, in which the lightness of the S_5 surface of the H_{220-4} increased by 24.1 % compared with that of the S_0 surface.

It could be clearer that the surface color and hardness changed as the position of different thickness ranged from S_0 to S_5, based on the lightness difference ΔL^* and the color difference ΔE^*. Two different methods, that is the surface pressing ball hardness and Shore hardness, were used to characterize the hardness of the test layers of heat-treated poplar wood at different positions, and the results are shown in Figure 3. ANOVA analysis of the chromatic parameters and surface hardness are shown in Table 1.

Figure 3 and Table 1 show that the HT temperature, duration, and test surface layer location all have significant effects on the surface hardness of the test layers. The change of the test surface position has a more significant effect on the surface hardness of the heat-treated wood, proving that the modification intensity of the heat-treated wood was different in the thickness direction. H_R and *HD* values of heat-treated poplar board decreased with increasing HT temperature, while it increased only slightly with increasing HT duration. As predicted, the H_R and *HD* values of the S_0 on the heat-treated poplar wood decreased significantly with increasing HT temperature in all groups. Interestingly, the other test surfaces S_1-S_5 also decreased but to a relatively lesser extent. There is a certain thickness of brittleness layer on the surface of the heat-treated wood, and its degree of thermal degradation and brittleness is higher than that of the internal material. The overall increasing trend of the surface hardness of the heat-treated wood from the surface to the core layer

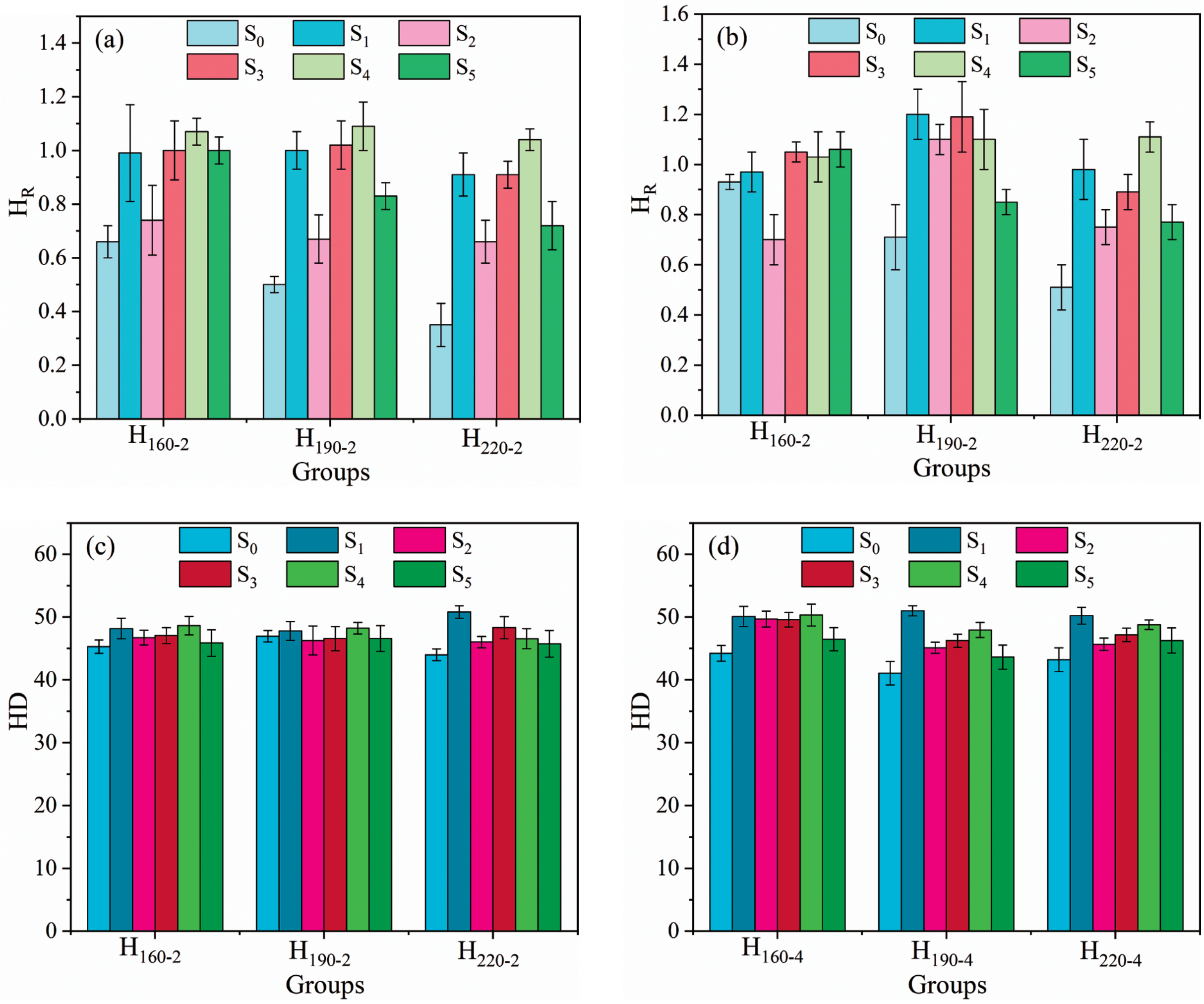

Figure 3 Surface pressure ball and Shore hardness of test layers in different positions of heat-treated poplar board
Slika 3. Tvrdoća površine izmjerena utiskivanjem kuglice i tvrdoća po Shroreu ispitnih slojeva na različitim mjestima toplinski modificirane topolovine

exhibited an "M" shape, and the difference between the surface and core layers increases with the increase of the HT temperature.

The ΔL^*, ΔE^*, H_R, and *HD* values of the test surfaces on S_0 to S_5 of the same experimental group were analyzed by SPSS. The S-N-K (Student Newman Keuls) method was selected for pairwise comparison, and the obtained data were marked with letters, as shown in Table 3. The HT temperature, duration, and test surface location all have significant effects on the color change of the heat-treated poplar boards, with the effect of HT temperature being the most significant. It is obvious that the ΔE^* values of the test surfaces at the same position gradually increase with the increase of the HT temperature. The extended HT duration increased the ΔE^* value, and the effect was less compared to the HT temperature, which is consistent with the findings of the ANOVA in Table 1.

The color change of heat-treated wood was closely related to treatment factors like treatment time, temperature, agent of treatment (Zhan *et al*., 2022). In addition, the effect of HT duration on wood color gradually decreases at higher HT temperatures. For example, the ΔE^* of H_{160-2}, H_{160-4} was 4.46 and 8.58, while the ΔE^* of H_{220-2}, H_{220-4} was 53.46 and 53.62, indicating that the color of poplar wood no longer changed significantly when the HT duration exceeded 2 h under the HT temperature of 220 °C. The ΔL^* and ΔE^* values, whose test surfaces of the wood board were treated by the same HT temperature and duration but located in a different position in the direction of thickness, were also significantly different. Except for H_{160-2}, there were significant differences regarding ΔL^* and ΔE^* values between the S_0 and S_1-S_5 surfaces of the heat-treated poplar boards at the 0.05 level. For H_{160-2}, the color change of S_0 and S_1 were statistically significantly different from that of S_2~S_5 surfaces.

Based on the result in Table 2, it could be concluded that the depth of the Shore hardness tester probe into the wood is about 1.3 mm, which is quite close to the depth of 1 mm pressure ball. The changing trend of H_R is basically consistent with that of *HD*. The H_R of the heat-treated wood S_0 and S_5 test surfaces showed significant differences at different HT conditions. As

Table 2 Color change and hardness of test surfaces at different positions of heat-treated poplar
Tablica 2. Promjena boje i tvrdoće ispitnih površina na različitim mjestima toplinski modificirane topolovine

Groups		ΔL^*	ΔE^*	H_R	HD	Groups		ΔL^*	ΔE^*	H_R	HD
H_{160-2}	S_0	-2.97^{a}	4.46^{a}	0.66^{b}	45.3^{b}	H_{160-4}	S_0	-6.05^{a}	8.58^{a}	0.93^{b}	46.96^{a}
	S_1	-2.32^{a}	4.34^{a}	0.99^{a}	48.18^{a}		S_1	-5.02^{b}	6.55^{b}	0.97^{ab}	47.80^{a}
	S_2	0.27^{b}	2.67^{b}	0.74^{b}	46.75^{ab}		S_2	-3.77^{c}	5.41^{cd}	0.70^{c}	46.28^{a}
	S_3	-0.32^{b}	2.57^{b}	1.00^{a}	47.05^{ab}		S_3	-4.38^{bc}	5.74^{bc}	1.05^{a}	46.59^{a}
	S_4	-0.60^{b}	2.61^{b}	1.07^{a}	48.65^{a}		S_4	-3.72^{c}	5.18^{cd}	1.03^{a}	48.25^{a}
	S_5	-0.41^{b}	1.94^{b}	1.00^{a}	45.88^{b}		S_5	-2.45^{d}	4.35^{d}	1.06^{a}	46.59^{a}
H_{190-2}	S_0	-24.37^{a}	27.20^{a}	0.50^{d}	43.99^{d}	H_{190-4}	S_0	-35.36^{a}	37.23^{a}	0.71^{c}	44.24^{c}
	S_1	-21.86^{b}	24.52^{b}	1.00^{a}	50.83^{a}		S_1	-33.39^{b}	35.76^{a}	1.20^{a}	50.09^{a}
	S_2	-19.33^{c}	22.02^{c}	0.67^{c}	46.03^{c}		S_2	-28.27^{c}	31.18^{b}	1.10^{a}	49.69^{a}
	S_3	-14.74^{e}	17.00^{e}	1.02^{a}	48.31^{b}		S_3	-25.89^{d}	28.76^{c}	1.19^{a}	49.60^{a}
	S_4	-17.99^{d}	20.73^{c}	1.09^{a}	46.56^{c}		S_4	-27.25^{cd}	30.27^{bc}	1.10^{a}	50.33^{a}
	S_5	-15.82^{e}	19.01^{d}	0.83^{b}	45.75^{c}		S_5	-26.18^{cd}	29.21^{bc}	0.85^{b}	46.46^{b}
H_{220-2}	S_0	-53.08^{a}	53.46^{a}	0.35^{d}	41.08^{e}	H_{220-4}	S_0	-53.13^{a}	53.6^{2}a	0.51^{e}	43.21^{c}
	S_1	-50.24^{b}	50.87^{b}	0.91^{b}	51.01^{a}		S_1	-50.52^{b}	51.15^{b}	0.98^{b}	50.23^{a}
	S_2	-46.88^{c}	47.84^{c}	0.66^{c}	45.11^{c}		S_2	-40.62^{e}	42.37^{e}	0.75^{d}	45.69^{b}
	S_3	-46.32^{c}	47.37^{c}	0.91^{b}	46.25^{c}		S_3	-41.37^{e}	42.88^{e}	0.89^{c}	47.19^{b}
	S_4	-48.14^{c}	48.98^{c}	1.04^{a}	47.96^{b}		S_4	-46.08^{c}	47.25^{c}	1.11^{a}	48.80^{a}
	S_5	-47.25^{c}	48.12^{c}	0.72^{c}	43.64^{d}		S_5	-44.82^{d}	46.05^{d}	0.77^{d}	46.28^{b}

Different superscript letters indicate significant differences at 0.05 statistical level.
Različita slova u eksponentu označavaju statistički značajne razlike pri razini od 0,05.

displayed in Table 2, the H_R and *HD* value of the heat-treated poplar wood has several levels with significant differences between the different positions of S_0~S_5 surfaces (except for H_{160-4}). When the HT temperature was relatively low, there were only two significantly different levels in the H_{160-2}, and differences between S_3-S_5 test surfaces were insignificant. As the HT temperature increased, the difference between the S_1 and core layers increased. For instance, the H_R value of the S_5 of H_{220-2} was 105.71 % higher than that of S_0, and four in five test surfaces were significantly different.

3.2 Chemical component change of different test surface layers

3.2. Kemijske promjene spojeva u različitim slojevima ispitne površine

The FT-IR spectrum of the changes in chemical functional groups of poplar wood before and after heat treatment are shown in Figure 4. The experiments in the spectral range of 4000-400 cm^{-1} mainly analyzed the changes of characteristic peaks in the range of 1800-1750 cm^{-1}. The shear vibration peak of cellulose at 1424 cm^{-1} changed to a lesser extent after heat treatment, so the peak here was selected for normalization of the spectrum (Wentzel *et al.,* 2019), and the ratios of the peaks are shown in Table 3.

The changes in the functional groups on the surface of the heat treatment were analyzed by combining Figure 4 and Table 3. The characteristic peak at 1735 cm^{-1} is the hemicellulose acetyl non-conjugated carbonyl group (Li *et al.,* 2009), and the intensity of this peak decreases with the increasing heat treatment temperature. This phenomenon is mainly caused by hemicellulose degradation, and the higher the heat treatment temperature, the more intense degradation.

The characteristic peak at 1163 cm^{-1} is attributed to the C-O-C stretching vibration of cellulose and hemicellulose. Moreover, the characteristic peak near 1058 cm^{-1} is the C-O stretching vibration of cellulose and hemicellulose, and the intensity of this peak increases during low temperature heat treatment, indicating the formation of alcoholic and aldehyde pyrolysis products during heat treatment (Gao, 2019). The characteristic peak near 1058 cm^{-1} reflects the C-O condensation vibration of cellulose and hemicellulose, and the intensity of this peak increases at low heat treatment temperatures, indicating the generation of alcoholic aldehydes pyrolysis products during heat treatment. However, its intensity decreases with increasing temperature, suggesting that the condensation reaction of alcohols and aldehydes occurs.

The carbonyl stretching vibration peak at 1650 cm^{-1} is attributed to lignin and is mainly present on the propyl-branched chain. A spectral peak at 1603 cm^{-1} corresponds to the stretching vibration peak of the benzene ring backbone of lignin conjugated to C=O, and 1264 cm^{-1} corresponds to the absorption peak of the C=O stretching vibration of the G ring and acyloxy bond in lignin (Gao *et al.,* 2020). The intensity of the

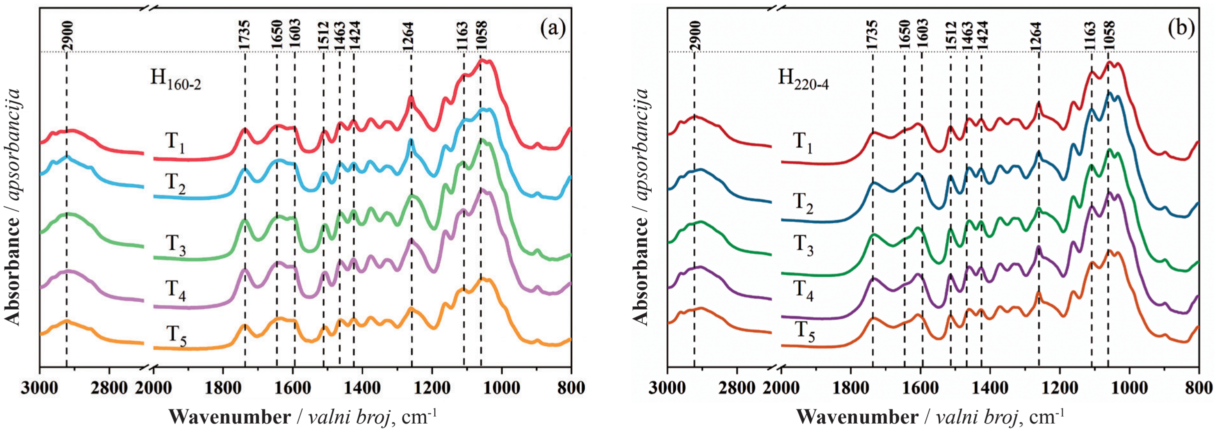

Figure 4 Infrared spectra of different surface layers of heat-treated poplar wood
Slika 4. Infracrveni spektri različitih površinskih slojeva toplinski modificirane topolovine

Table 3 Strength ratios of peaks normalized at 1424 cm^{-1}
Tablica 3. Omjeri intenziteta vrhova normalizirani na 1424 cm^{-1}

Groups		I1735	I1603	I1512	I1463	I1264	I1163	I1058
Control		0.865	0.801	0.737	0.974	1.840	0.904	2.560
H_{160-2}	T_1	0.782	0.824	0.725	0.944	1.620	1.577	2.542
	T_2	0.827	0.992	0.717	0.992	1.677	1.496	2.543
	T_3	0.808	0.859	0.712	0.955	1.311	1.537	2.446
	T_4	0.784	0.852	0.733	0.955	1.403	1.580	2.426
	T_5	0.780	0.954	0.721	0.963	1.358	1.505	2.339
H_{190-2}	T_1	0.773	0.864	0.733	0.962	1.553	1.538	2.659
	T_2	0.777	0.921	0.748	0.971	1.295	1.518	2.432
	T_3	0.787	0.806	0.755	0.968	1.387	1.535	2.626
	T_4	0.795	0.888	0.739	0.975	1.267	1.534	2.484
	T_5	0.855	0.796	0.763	0.980	1.434	1.546	2.579
H_{220-2}	T_1	0.709	0.847	0.810	0.985	1.518	1.358	2.234
	T_2	0.742	0.856	0.825	1.009	1.336	1.507	2.686
	T_3	0.770	0.867	0.819	1.000	1.566	1.588	2.942
	T_4	0.788	0.872	0.847	1.010	1.355	1.498	2.507
	T_5	0.819	0.896	0.850	1.016	1.383	1.466	2.425
H_{160-4}	T_1	0.816	0.810	0.741	0.966	1.425	1.598	2.534
	T_2	0.868	0.755	0.759	0.972	1.637	1.519	2.594
	T_3	0.801	0.768	0.755	0.960	1.470	1.536	2.377
	T_4	0.830	0.785	0.756	0.970	1.719	1.541	2.733
	T_5	0.741	0.856	0.759	0.966	1.345	1.517	2.236
H_{190-4}	T_1	0.846	0.787	0.757	0.978	1.375	1.551	2.603
	T_2	0.840	1.000	0.769	0.982	1.296	1.515	2.444
	T_3	0.852	1.000	0.723	0.992	1.287	1.525	2.451
	T_4	0.861	0.933	0.733	0.976	1.315	1.576	2.533
	T_5	0.864	0.950	0.721	0.979	1.443	1.600	2.721
H_{220-4}	T_1	0.707	0.900	0.833	1.013	1.360	1.387	2.280
	T_2	0.744	0.911	0.867	1.017	1.311	1.461	2.411
	T_3	0.766	0.886	0.864	1.011	1.250	1.457	2.310
	T_4	0.789	0.871	0.860	1.018	1.421	1.503	2.468
	T_5	0.771	0.866	0.843	1.000	1.465	1.449	2.543

peaks at 1650 cm^{-1} and 1264 cm^{-1} decreased with the increasing heat treatment temperature, while the intensity of the peak at 1603 cm^{-1} increased, indicating that the relative lignin content increased, while the branched-chain breakage occurred by thermal degradation and possibly cross-linked condensation reaction. The benzene ring carbon skeleton vibration peak of lignin at 1512 cm^{-1} and the C-H bending vibration peak in lignin and carbohydrates at 1463 cm^{-1} were enhanced with increasing heat treatment temperature and duration, which may be caused by the increase in relative lignin content due to hemicellulose degradation (Zang *et al.,* 2018).

As can be seen in Table 3, the characteristic peak at 1735 cm^{-1} showed a small difference between the surface and core layers at 160 °C treatment. With the increasing heat treatment temperature, the intensity of this peak showed an increasing trend from the surface layer to the core layer, and the characteristic peak at $H_{220\text{-}2}$ was 0.709 in the surface layer, which was 13.43 % lower than that in the core layer. Therefore, hemicellulose degradation was less at 160 °C treatment. The difference in the degree of hemicellulose degradation between the surface and core layers increased with the increase in temperature, while it decreased with increasing duration. At 160 °C treatment, the intensity of the characteristic peak at 1163 cm^{-1} in the surface layer was greater than that in the core layer. As the heat treatment temperature increased, the intensity of this absorption peak tended to enhance from the surface layer to the core layer, probably since the surface hydroxyl groups of cellulose and hemicellulose in the surface layer formed ether bonds earlier than those in the core layer at lower temperatures. However, it is also the ether bond in the surface layer that breaks first when the temperature increases, and the extension of the duration has minor effect on this peak. Only at the temperature of 220 °C, the absorption peak intensity of the core layer at 1058 cm^{-1} was significantly stronger than that of the surface layer. With the increase of heat treatment temperature, the hemicellulose and cellulose in the surface layer were the first to undergo ring-opening reactions to form alcohols and aldehydes. Then, with the further increase of temperature, the alcohols and aldehydes in the surface layer underwent condensation reactions and the core layer was less reactive than the surface layer, and the difference increased with the extension of the duration.

Under different heat treatment conditions, the variation of absorption peak intensity at 1512 cm^{-1} and 1463 cm^{-1} between the surface and core layers was light, indicating that the heat treatment had small effect on the lignin benzene ring skeleton. The intensity of the characteristic peak at 1264 cm^{-1} showed a decreasing trend from the surface layer to the core layer after 2 h duration, reflecting that the relative lignin content in the surface layer of the heat-treated wood was greater than that in the core layer; however, the intensity of the characteristic peak at 1264 cm^{-1} in the surface layer was gradually greater than that in the core layer with the extension of the duration, suggesting that the pyrolysis of lignin in the surface layer was greater than that in the core layer.

3.3 Prediction of surface properties on different test surface layers
3.3. Predviđanje površinskih svojstava na različitim ispitnim površinskim slojevima

Surface color and hardness of S_0 surface on the heat-treated wood are significantly different from those of the S_1~S_5 core layer surfaces, and the H_R of the S_0 surface is significantly different from all other test surfaces. Therefore, the S_0~S_5 test surfaces have a unique property variation that warrants a separate study. As the effect of HT temperature on the color of heat-treated wood is relatively small, this study mainly investigates the influence of HT temperature of the test surfaces at different positions. Table Curve 3D software was used to establish the prediction models of ΔL^* and ΔE^* values. The fitting surfaces are shown in Figure 5.

Table Curve 3D software was used to establish the prediction models of HT temperature (T) and test surfaces (F) with lightness change (ΔL^*) and color changes (ΔE^*). The fitting surfaces are shown in Figure 5.

The prediction models for ΔL^* and ΔE^* of the heat-treated poplar wood are:

$\Delta L^*=160.69-1.09*T-43.92*F-1.15\times10^{-4}*T^2+1.43*F^2+0.44*T*F+2.90\times10^{-6}*T^3+0.05*F^3-0.01*T*F^2-9.72\times10^{-4}*T^2*F$ (R^2=0.966);

$\Delta E^*=199.51-29876.22/T+13.35*F-1308772.50/T^2+2.80*F^2-(8820.77*F)/T+1.84\times10^{-8}/T^3-0.04*F^3-(386.87*F^2)/T+(1026586.80*F)/T^2$ (R^2=0.966).

The model fits well and the empirical prediction model could be used to predict the changes in the surface color of poplar boards at different positions under different HT conditions.

This color difference on heat-treated wood in the direction of thickness could be explained by the diversity of chemical components degradation intensity, as well as extractives migration from the core layer to the surface layer. Shi and Bao (2021) found that the extractives of heat-treated wood migrate under hydrothermal conditions, which may cause differences in the color of different layers. Under the HT conditions, polysaccharides, especially the hemicellulose, degrade and, numerous hydroxyl groups are oxidized to carboxyl groups and carbonyl groups. As the HT proceeds, these degradation products continue to react and generate furfural and phenolic compounds with color-emitting or color-assisting groups. At the same time, lignin undergoes condensation and oxidation reactions with

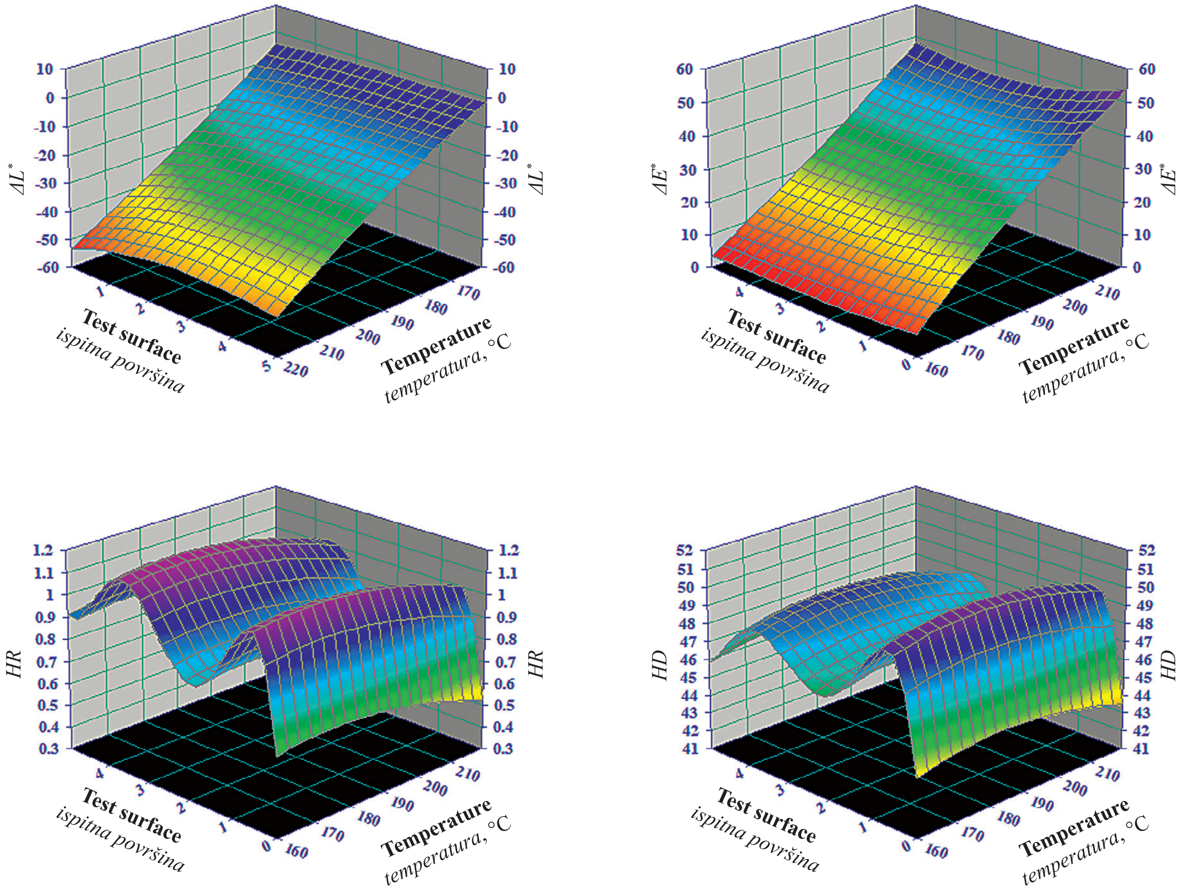

Figure 5 Fitting surface of ΔL^*, ΔE^*, H_R, HD of heat-treated poplar board
Slika 5. Fitanje površine ΔL^*, ΔE^*, H_R, HD za ploče od toplinski modificirane topolovine

degradation products under high temperature conditions, generating new color-emitting substances that change the color of heat-treated wood (Cao *et al.,* 2018; Konnerth *et al.,* 2010; Kamperidou, 2012).

Table Curve 3D software was used to establish the prediction models of HT temperature (T) and test surfaces (F) with the hardness of the heat-treated poplar board in terms of ball hardness (H_R) and Shore hardness (HD). The fitting surfaces are shown in Figure 5.

The prediction models for H_R and HD of the heat-treated poplar wood are:

H_R=-1.30-8.13*ln$T$+2.30*(ln$T$)^2+0.08*(ln$T$)^3-0.04*(ln$T$)^4+1.99*F-2.65*F^2+1.31*F^3-0.27*F^4+0.02*F^5 (R^2=0.729)

HD=-1015.94+407.34*ln$T$-39.11*(ln$T$)^2+21.68*F-25.39*F^2+11.24*F^3-2.11*F^4+0.14*F^5 (R^2=0.704)

The equations fit well, so the empirical prediction model can be used to predict the variation of hardness at different locations of poplar wood boards under different heat treatment conditions. Besides, the surface hardness of heat-treated wood is influenced by multiple factors. Konnerth *et al*. (2010) used hemicellulose for targeted hemicellulose removal and found that wood hardness is most sensitive to changes in cell wall hemicellulose content, so the reduction in wood hardness after heat treatment is mainly influenced by hemicellulose degradation. The difference in hardness between the surface and core layer increases with increasing temperature, probably because the hemicellulose, a cell wall filling material, decreased to a different extent. Altgen *et al*. (2018) found by infrared spectroscopy that wood undergoes cross-linking reactions in the cell matrix during heat treatment, and that differences in the degree of cross-linking between the surface layer and core layer may also contribute to their differences.

4 CONCLUSIONS
4. ZAKLJUČAK

The surface color and hardness of the heat-treated wood were not uniform from the surface layer to the core layer, and there were several levels with significant differences as the temperature increased. The surface color and hardness properties between S_0 and S_1~S_5 test surfaces were significantly different under different heat treatment conditions. The color difference of heat-treated poplar wood tended to decrease from the S_0 surface to the core layers, from 4.46 to 1.94 for H_{160-2} and from 53.46 to 48.12 for H_{220-2}; the surface hardness shows an increasing trend. H_{160-2} increases from 0.66 to 1.0, an increase of 34 %. The hardness of the H_{220-2} core surface layer increases by 105.71 % compared to the S_0 surface.

The degradation of cell wall components in the surface layer of heat-treated poplar wood was greater than that in the core layers. FT-IR showed that the thermal degradation of hemicellulose and lignin of the T_1 surface layer was greater than that in the core layers. The difference of the test surfaces could be predicted as it related to the location and heat treatment conditions. Table Curve 3D software was used to establish an accurate prediction model of the trend of surface color, hardness, and other properties at different locations of the heat-treated wood and different heat treatment temperatures.

In conclusion, this paper displayed the property difference of test surface from outside to core layers of the heat-treated wood and proved that the thermal modification intensity of the heat-treated poplar wood was different in the direction of thickness. Accurate models were established, which could be used to predict the surface color and hardness on each test surface of the heat-treated poplar wood in the direction of thickness.

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Water Permeability and Adhesion Strength of Bio-based Coating Applied on Wood

Vodopropusnost i adhezivna čvrstoća biopremaza nanesenoga na drvo

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ABSTRACT • *The main task of wood manufacturers is to ensure the supply of safe wood products. In this respect, the use of plants as raw material for the wood coatings production is a sustainable alternative to fossil fuels, especially since innovative materials minimize the health and environmental risks of the final product over its entire lifespan. This paper presents a study of the water permeability of a water-borne bio-based coating applied to spruce (Picea abies), a study of the adhesion strength of the same coating applied to spruce (Picea abies), beech (Fagus sylvatica L.) and beech plywood, as well as a study of the adhesion strength on spruce after the water permeability test. The tests were performed according to EN 927-5:2006 and EN ISO 4624:2016. The roughness parameters were measured before and after the water permeability test. The coating was found to be hydrophobic but also water permeable. The highest value of adhesion strength was observed for beech surface, the lowest for spruce. The changes in the surface profiles after the water permeability test are insignificant. According to the water absorption criteria, this coating system could only be applied on exterior wood intended for end-use categores such as overlapping cladding, fencing, garden sheds, open cladding, and ventilated rain screen.*

KEYWORDS: *water permeability; adhesion strength; bio-based coating; beech; spruce*

SAŽETAK • *Izrada sigurnih proizvoda od drva glavni je zadatak njihovih proizvođača. U tom je smislu upotreba biljaka kao sirovine za proizvodnju premaza za drvo održiva alternativa fosilnim gorivima, pogotovo zbog inovativnih materijala koji umanjuju zdravstvene i ekološke rizike finalnih proizvoda tijekom cijeloga njihova životnog vijeka. U članku je prikazano istraživanje vodopropusnosti biopremaza na bazi vode nanesenoga na smrekovinu (Picea abies), istraživanje adhezivne čvrstoće tog premaza na smrekovini (Picea abies), bukovini (Fagus sylvatica L.) i bukovoj furnirskoj ploči te istraživanje adhezivne čvrstoće na smrekovini nakon ispitivanja vodopropusnosti. Ispitivanja su provedena prema EN 927-5:2006 i EN ISO 4624:2016. Prije i nakon ispitivanja vodopropusnosti izmjereni su parametri hrapavosti površine drva. Utvrđeno je da je premaz hidrofoban, ali i vodopropustan. Najveće vrijednosti adhezivne čvrstoće zabilježene su za bukovinu, a najmanje za smrekovinu. Promjene profila površine nakon ispitivanja vodopropusnosti neznatne su. Prema kriterijima upijanja vode, taj se sustav premaza može nanositi samo na drvo u eksterijeru koje se upotrebljava za fasadne obloge koje se preklapaju, za ograde i vrtna spremišta, kao i za otvorene fasadne obloge i ventilirane zaslone protiv kiše.*

KLJUČNE RIJEČI: *vodopropusnost; adhezivna čvrstoća; biopremaz; bukovina; smrekovina*

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1 INTRODUCTION
1. UVOD

Wood lignocellulosic biopolymers (cellulose, lignin, hemicelluloses) are subjected to progressive oxidative degradation processes under the action of environmental factors (UV radiation, moisture, heat/cold variations, atmospheric oxygen), which affect wood native durability and cause the occurrence of significant structural and colour changes (discoloration), along with a progressive diminution of its resistance against biological agents (biodegradation or decay development) and its mechanical properties (Teacă *et al*., 2019). Furthermore, wood is a hygroscopic material that can adsorb or desorb water in response to the temperature and relative humidity of the surrounding atmosphere. Consequently, the moisture content of wood is one of the most important variables affecting its physical and mechanic properties (Hartley, 2001). In this context, wood products are easy to absorb water and steam when they are exposed to environmental conditions for a long time, which greatly affects the durability of wood products and degrades their properties. One effective way to prevent wood degradation processes is to apply coatings on the surface. The varying moisture content of wood results in dimensional and conformational instability, which can compromise the performance of other materials combined with wood, such as adhesives and surface coatings (Mantanis and Papadopoulos, 2010). For protective and decorative reasons, coatings (varnishes) are generally applied on wooden substrates as multi-layered systems that are composed of primer and topcoat. Their application on wood materials aims to enhance the biological, physical, and mechanical properties of wood.

The wood coatings industry represents an active field of research and development, driven by the necessity to produce high-performance materials able to respond to different environmental regulations and constraints. Recently, the attention is turning towards the development of coatings based on green materials, namely "bio-based coatings", obtained through sustainable processes that do not generate toxic emissions (Sarcinella and Frigione, 2023). Recent trends in this area include the use of bio-based natural products (such as wood and plant extractives, vegetable oils, natural waxes, different biopolymers, and biological control agents) and nano-based materials (Teacă *et al*., 2019). For all these products, the main criterion for the estimation may be represented by the protection provided by the physical and mechanical properties of coatings applied on the wood surface. The basic properties of a coating are determined by its main components, namely binders, pigments, solvents, fillers, and additives (Sandberg, 2016). Permeability of coatings for water and water vapour is a supreme factor in their wood protective function. Permeability is a measure of the ease with which fluids are transported through a porous solid under the influence of a pressure gradient (Siau, 2012). Liquid water and water vapour uptake are affected by coating film thickness, number of coats, and coating composition.

In order to perform satisfactorily, coatings must adhere to the substrates on which they are applied. Adhesion is a complex physico-chemical phenomenon for which, however, there is not a rigorous theoretical definition. Adhesion is difficult to define, and an entirely satisfactory definition has not yet been found (Silva *et al*., 2011, Ebnesajjad and Landrock, 2014). Therefore, a quantitative analysis of coating adhesion on wood is needed. One of the main factors used for the evaluation of the adhesion of coatings is the adhesion strength. Adhesion strength is an important feature to measure the durability of the coatings. Various methods are used to evaluate adhesion strength of the coats. The most objective and widespread method is the pull-off test. The main advantage of this method includes its practicality and simple application for different surfaces. Adhesion of a single coating or a multi-coat system of paint, varnish or related products is assessed by measuring the minimum tensile stress necessary to detach or rupture the coating in a direction perpendicular to the substrate.

In furniture production, the coating processes have great importance for technical, economic, aesthetical, and ecological evaluation of the wood materials. Different coating technologies provide different levels of protection. More than 95 % of exterior wood coatings are applied as liquid coatings, either solvent- or water-borne (e.g. acrylic, polyurethane, alkyd), but their use and subsequent emission of volatile organic compounds (VOCs) represents a dominant concern because of their significant contribution to global warming (Teacă *et al*., 2019). As the coatings industry seeks to improve its environmental impact, plant-based technologies present a major innovation opportunity. Natural extractives, vegetable and essential oils, natural resins and waxes, and various biopolymers can be included in coating formulations (primarily water-borne ones) to prevent or limit the harmful impact of these formulations on the environment due to their specific properties (e. g. non-toxicity, reversible character, resistance to moisture and solvents, compatibility). The aim of this study is to determine the water permeability and adhesion strength of bio-based coating for the protection of wood surfaces. Therefore, the axial pull-off test was used to determine the adhesion strength of samples manufactured from different wood species to provide an initial data for improving the quality of finished products manufactured from bio-based raw materials. The standard method described in EN 927-5:2006 was used to evaluate the water permeability of bio-based coating films.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

For the tests, spruce (*Picea abies*) with an average density of 440 kg/m^3, European beech (*Fagus sylvatica* L.) with an average density of 730 kg/m^3 and beech plywood with an average density of 750 kg/m^3 were used. The plywood surface was sanded with a P80-120 grain size sandpaper supplied by the manufacturer (S. C. Cildro Plywood, Romania).

Two sets of test specimens were prepared.

The samples from spruce with dimensions 150 mm × 70 mm × 20 mm (longitudinal, tangential, and radial directions, respectively) were prepared for the water permeability test. The test samples were manufactured in accordance with the specifications in EN 927-5:2006. The specimens were chosen for their lack of defects, such as knots, cracks, or resin spots.

For the adhesion strength test, samples of spruce and beech wood with dimensions 150 mm × 50 mm × 20 mm and beech plywood with dimensions 50 mm × 150 mm × 15 mm were prepared.

All samples were conditioned prior to coating application (temperature of (20±2) °C and relative humidity of (65±5) %).

For coating of samples, a bio-based wood stain with the colour "tobacco" for outdoor application, produced by Industrias Quimicas Masquelack, S. A. (Spain), was used (Figure 1). According to the information given by the manufacturer, it creates a water-resistant, breathable and flexible film with high outdoor durability. The coating system is water-borne with a bio-based content of 39 % and contains Decovery® SP - 7450 by Covestro. Decovery® SP - 7450 is an acrylic copolymer emulsion with elongation, blocking resistance, and early water resistance that is suitable for clear and opaque formulations in high-demand outdoor durability applications. The bio-based wood stain mixtures had a viscosity of 21 s when measured at 23 °C in a DIN 4 mm viscosity cup.

The test samples were sanded with P150 grain size sandpaper, and the first layer of the bio-based wood stain was applied by brush. After drying, the surfaces were treated with abrasive steel wool (Scotch-brite®) and a second layer was applied. The stain amounts Q for the two layers were Q_1 = 80 g/m^2 and Q_2 = 30 g/m^2, respectively. After the coating drying, the average dry residue content of hardened film is 17.34 %.

To establish the coating hydrophobicity, the behaviour of drops of distilled water on the surfaces of the various test specimens was observed. The drops are dosed with a micropipette and have a volume of 2.5 μm.

The European standard EN 927-5:2006 specifies a test method for assessing the liquid water permeability of coating systems for exterior wood by measuring the water absorption of coated wood panels. After the application of the coating, all the remaining sides were sealed with a two-component polyurethane (PU) system. The PU system has a predetermined water permeability of 16 g/m^2. For each set of six replicates, the arithmetic mean value of the weight increase and the standard deviation were calculated. The arithmetic mean value of the weight increase after 72 hours of floating is reported as the mass of absorbed water per test face area. The amount of water absorbed by each sample was measured as mass of absorbed water (*MWA*) per test face area relative to the weight of the conditioned specimen prior to the test, Eq. (1):

$$MWA_1 = (w_1 - w_0) / A \qquad (1)$$

Where MWA_1 – mass of absorbed water per area in g/m^2 at time 1; w_0 – weight in g at time 0; w_1 – weight in g at time 1; A – area of test face in m^2.

The adhesion strength of the coating is defined by a standardized pull-off method (ISO 4624:2016) with a glued stamp (dolly). The test is performed by securing a loading fixture (dolly) perpendicular to the surface of the coating with an adhesive. After the adhesive is cured, a testing apparatus is attached to the loading fixture and aligned to apply tension perpendicular to the test surface. The force applied is gradually increased and monitored until either a plug of coating material is detached, or a specified value is reached.

Figure 1 Wood surfaces coated with bio-based wood stain: A – spruce (*Picea abies*); B – European beech (*Fagus sylvatica* L.); C – beech plywood
Slika 1. Drvene površine premazane biolazurom: A – smrekovina (*Picea abies*); B – bukovina (*Fagus sylvatica* L.); C – bukova furnirska ploča

The Mitutoyo SJ-210 surface roughness measurer (Mitutoyo, Japan) was used to indicate the surface roughness at the following settings: profile – *R* (radius - 5 µm), filter - Gauss; number of segments $N = 6$; cut-off length λc – 2.5 mm; measuring speed – 0.25 mm/s. The measurements were made perpendicular to the wood grains. Surface parameters like arithmetic mean deviation of the assessed profile (*Ra*), maximum height of profile (*Rz*), maximum profile peak height (*Rp*), total height of profile (*Rt*), maximum profile valley depth (*Rv*) and mean width of the profile elements (*RSm*) were estimated according to ISO 4287:1997. Roughness depth (*Rk*) reduced peak height (*Rpk*), as well as reduced valley depths (*Rvk*) were estimated by ISO 13565-2:1996.

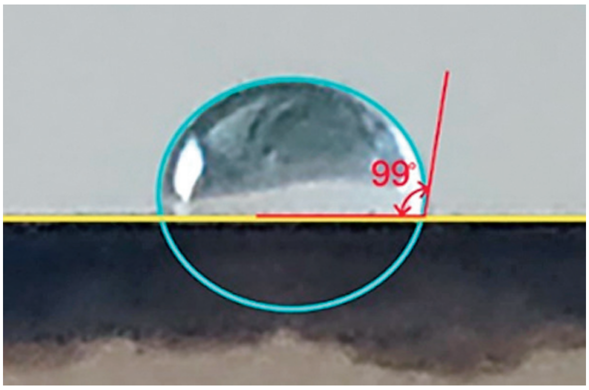

Figure 2 Behaviour of liquid (distilled water) in contact with bio-based coating surface (static water contact angle θ is 99°)
Slika 2. Ponašanje tekućine (destilirane vode) u dodiru s površinom biopremaza (statički kontaktni kut vode θ je 99°)

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Water permeability of bio-based coating
3.1. Vodopropusnost biopremaza

The obtained bio-based coating is hydrophobic because the static water contact angle θ is > 90° (Figure 2). The water permeability test, on the other hand, revealed that the bio-based coating applied on the spruce specimens showed extremely high water-permeability (the average value of 725 g/m^2).

The time-dependent mass of absorbed water (*MWA*) during the water permeability test of spruce (*Picea abies*) coated with bio-based stain is shown in Figure 3. Manufacturers mention that the used bio-based wood stain is water resistant, but the two coats applied were completely insufficient. According to the water absorption criteria in EN 927-2:2022, this coating system is non-stable. Mostly, this is due to the porous, breathable, and thin protective coating made by bio-based stain. It is also possible to free the movement of coated wooden products. By classification in EN 927-1:2013, this system could be used outdoor only for end-use categories such as overlapping cladding, fencing, garden sheds, open cladding, and ventilated rain screen.

After the water permeability test, the surface profiles of the investigated samples have changed. A quantitative assessment of these changes is given by the roughness parameters. The changes in the roughness parameters in the selected study phases indicate the redistribution mechanism of the remaining water in the test specimen.

Table 1 shows the average roughness parameters of a surface coated with bio-based stain before and after water permeability testing. The table also presents the percentage changes in the roughness parameters after 72 hours of drying the samples at room temperature (20 ±2 °C) and relative humidity (65 ±5 %). Measurements of roughness parameters were made on the

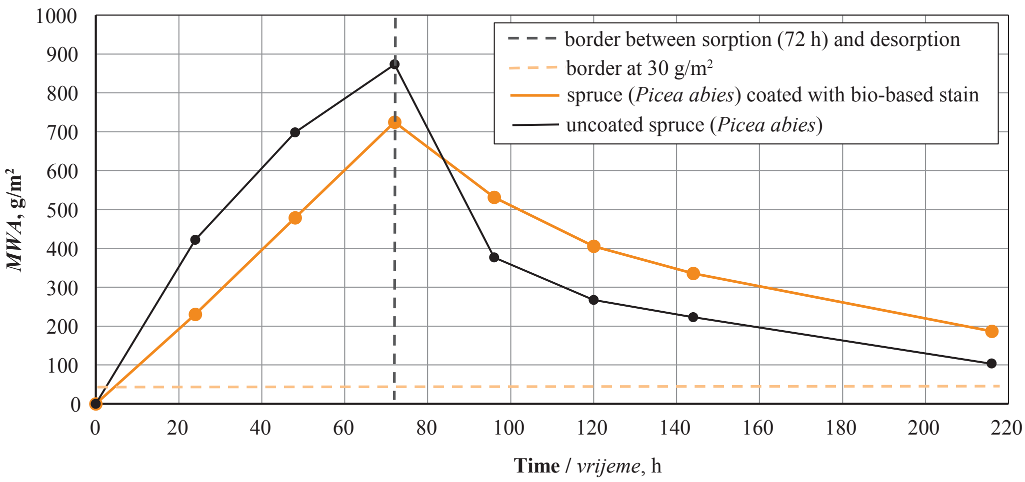

Figure 3 Time-dependent mass of absorbed water (*MWA*) during water permeability test of spruce (*Picea abies*) coated with bio-based stain
Slika 3. Vremenski ovisna masa apsorbirane vode (*MWA*) tijekom testa vodopropusnosti smrekovine (*Picea abies*) premazane biolazurom

Table 1 Roughness of a spruce surface coated with bio-based stain before and after water permeability testing, and 72-hour drying
Tablica 1. Hrapavost površine smrekovine premazane biolazurom prije i nakon ispitivanja vodopropusnosti te nakon 72-satnog sušenja

Roughness parameter *Parametri hrapavosti*	Average values, µm *Srednje vrijednosti, µm*			Accuracy index, % *Indeks preciznosti, %*			Change, % *Promjena, %*	
	Initial *Na početku*	After testing *Nakon ispitivanja*	72 h drying *Nakon 72-satnog sušenja*	Initial *Na početku*	After testing *Nakon ispitivanja*	72 h drying *Nakon 72-satnog sušenja*	After testing *Nakon ispitivanja*	72 h drying *Nakon 72-satnog sušenja*
$\overline{Ra}$	4.84	4.77	4.87	2.16	2.22	2.11	-1.57	2.22
$\overline{Rz}$	35.73	34.25	34.24	1.80	1.87	2.09	-4.13	-0.03
$\overline{Rp}$	16.81	15.51	15.48	1.90	2.06	2.23	-7.76	-0.17
$\overline{Rv}$	18.91	18.74	18.76	2.11	2.10	2.37	-0.91	0.09
$\overline{RSm}$	279.95	282.18	295.10	2.40	3.09	3.41	0.80	4.58
$\overline{Rt}$	46.14	44.30	44.62	2.26	2.50	2.67	-3.98	0.73
$\overline{Rk}$	15.22	14.99	15.69	2.50	2.49	2.32	-1.52	4.63
$\overline{Rpk}$	6.86	6.18	6.26	2.39	3.23	3.35	-9.84	1.29
$\overline{Rvk}$	7.52	7.67	7.35	2.31	2.72	3.00	1.95	-4.19

same sections of the same length. The values show a decrease in the roughness of the coating after the water permeability test, with the largest percentage decrease in the parameters *Rp* (maximum profile peak height) and *Rpk* (reduced peak height). This means that the coating is elastic enough to act as a barrier which limits grain raising.

Based on the high water-permeability of the studied coating, it can be concluded that using the bio-based stain by itself will not provide proper water protection of the wood products. It is advisable to apply an acrylic topcoat with proven hydrophobic properties.

3.2 Adhesion strength of bio-based coating
3.2. Adhezivna čvrstoća biopremaza

After the pull-off test according to ISO 4624:2016, (90-100) % adhesion destruction was observed between the substrate and the coating (Figure 4). According to the wood species (Figure 5), the adhesion strength of stain is higher on European beech (*Fagus sylvatica* L.) and on beech plywood than on spruce (*Picea abies*). These results confirm the general statement that the adhesion strength of coatings is higher on hardwood than on softwood (Kaygin and Akgun, 2008). There are a lot of factors that may cause this difference among the species, e.g. intensity, cell structure, basic and secondary compounds of wood, texture, extractive substances (Kaygin and Akgun, 2008).

The two-layer bio-based coating on wood surfaces has sufficiently good adhesion strength (Figure 5). According to the technological criteria for outdoor paints and varnishes (Decision, 2009), the critical value of the adhesion strength is 1.5 N/mm^2.

Adhesion of coatings on wood is most critical under wet conditions (de Meijer, 1999; Sönmez *et al.*, 2009). In this regard, the adhesion strength of the spruce specimens was measured after the water permeability test, i.e. after 72 h of sorption and 144 h of desorption. The average value obtained for the adhesion strength of the spruce specimens after the water permeability test is 2.58 N/mm^2, which is a decrease of 2.7 %. This means that the coating of bio-based stain

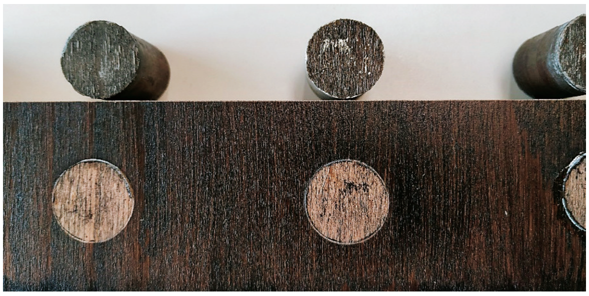

Figure 4 Typical destruction after axial pull-off test
Slika 4. Tipični lom nakon *pull-off* testa

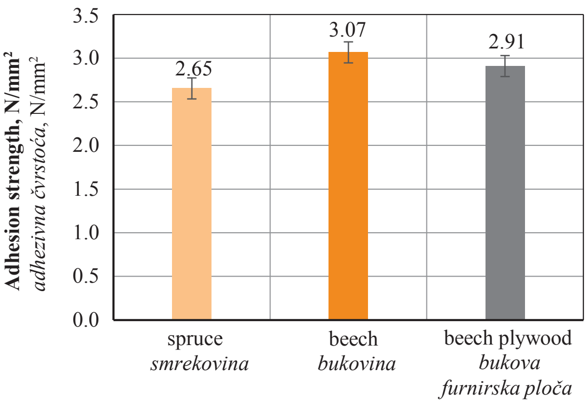

Figure 5 Adhesion strength of wood samples coated with two-layer bio-based stain
Slika 5. Adhezivna čvrstoća uzoraka drva premazanih dvama slojevima biolazure

applied to wood retains almost all its adhesion strength after contact with water.

4 CONCLUSIONS
4. ZAKLJUČAK

Under the conditions of this study and based on the results obtained, the following conclusions can be made specifically for the above-mentioned materials:

Good hydrophobicity of the studied bio-based stain applied to wood has been established;

The water permeability of the studied two-layer bio-based coating is 725 g/m^2. According to the water absorption criteria, this coating system is non-stable. This coating system could be used outdoor only for end-use categories such as overlapping cladding, fencing, garden sheds, open cladding, and ventilated rain screen;

The two-layer bio-based coating on wood surfaces has comparatively high adhesion strength and retains almost all of it after 72 hours of contact with water.

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Environmental Assessment/Evaluation of 3D Printing and 3D Printing with Wood-PLA Composites - Case Study

Ekološka procjena/evaluacija 3D printanja i 3D printanja s drvo-PLA kompozitima – studija slučaja

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ABSTRACT • *In recent years, additive manufacturing has become a regular process in various industries, and consequently there is an increasing need to evaluate the environmental aspects of this technology and its associated materials. In this paper, comparative cradle-to-grave life cycle assessments between a conventional product and a 3D-printed alternative made of polylactic acid (PLA) and PLA-wood material were investigated based on the standard ISO 14044:2006. The environmental impact of each product was quantified for 18 categories. The goal of life cycle assessment (LCA) was to determine whether the use of 3D printed PLA/PLA-wood products can be a sustainable alternative to traditional metal products. The paper presents a case study in which a comparative LCA was conducted. The results show that a metal part manufactured using conventional subtractive processes (milling, drilling, welding, etc.) has a higher environmental impact compared to 3D-printed alternatives made from renewable materials. However, there are many sub-issues that need to be adequately addressed.*

KEYWORDS: *life cycle assessment; 3D printing; environmental impact; carbon footprint; wood-PLA composite*

SAŽETAK • *Posljednjih je godina aditivna proizvodnja postala redoviti proces u raznim industrijama, a posljedično se pojavila sve veća potreba za procjenom ekoloških aspekata te tehnologije i s njom povezanih materijala. U ovom su radu ispitane i uspoređene procjene životnog vijeka konvencionalnog proizvoda „od kolijevke do groba" te 3D isprintane alternative izrađene od polilaktične kiseline (PLA) i PLA-drvnog materijala na temelju standarda ISO 14044:2006. Utjecaj svakog proizvoda na okoliš kvantificiran je unutar 18 kategorija. Cilj procjene životnog vijeka takvih proizvoda (LCA) bio je utvrditi može li uporaba 3D printanih PLA/PLA-drvnih proizvoda biti održiva alternativa tradicionalnim metalnim proizvodima. U radu je prikazana studija slučaja u kojoj je pro-*

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vedena komparativna procjena životnog vijeka – LCA. Rezultati pokazuju da metalni dio proizveden primjenom konvencionalnih subtraktivnih procesa (glodanja, bušenja, zavarivanja itd.) ima veći utjecaj na okoliš nego 3D isprintane alternative izrađene od obnovljivih materijala. Međutim, u vezi s tim postoje i mnoga potpitanja koja se moraju adekvatno riješiti.

KLJUČNE RIJEČI: *procjena životnog vijeka; 3D printanje; utjecaj na okoliš; ugljični otisak; drvo-PLA kompozit*

1 INTRODUCTION
1. UVOD

Three-dimensional printing (3DP) is a manufacturing process in which a product is built up layer-by-layer using a digital model. Since its introduction in the 1980s, additive manufacturing (AM) has been used primarily for rapid prototyping due to its ability to produce objects with complex geometries. Of these methods, FDM (Fused Deposition Modeling) or FFF (Fused Filament Fabrication) is the most researched and increasingly appreciated in the last decade and has become the manufacturing method for 3D printers. FDM 3D printers are now affordable and available to a community of do-it-yourself enthusiasts (Krapež Tomec and Kariž, 2022).

3D printing creates shifts in work patterns as the process is highly automated and human workers are only needed in pre- and post-processing (Lindemann *et al.*, 2012). Through 3D printing, chains are expected to become shorter by reducing the need for centralized manufacturing and tooling. Production-related energy requirements and CO_2 emissions are reduced by shortened processes and more direct manufacturing. This reduces the need for tooling, the need for handling, and it lowers indirect material-related energy through higher resource efficiency (Reeves, 2013).

Numerous perspective research articles on using wood in AM have already been published and are presented in (Krapež Tomec and Kariž, 2022). In this regard, it is also worth promoting life cycle assessment (LCA) analysis, a quantitative evaluation of the environmental impacts that occur during the life cycle of the product.

AM replicates biological processes by building products layer-by-layer. It is inherently less wasteful than traditional subtractive production methods and holds the potential to decouple social and economic value creation from the environmental impact of business activities. There are many potential sustainability benefits of this technology, from less material waste, energy efficiency, local production, carbon footprint reduction, to circular economy, optimized design and innovation driver; three of them stand out: (1) Improved resource efficiency - improvements can be realized in both the production and use phases, as manufacturing processes and products can be redesigned for AM; (2) Extended product life -achieved through technical approaches such as repair, remanufacture and refurbishment, and more sustainable socio-economic patterns such as stronger human-product affinities and closer producer-consumer relationships (Kohtala, 2015); (3) Redesigned value chains - shorter and simpler supply chains, more localized production, innovative distribution models and new collaborations (Ford and Despeisse, 2016).

Reeves (2012) has shown in a case study of a structural aircraft component that manufacturing-related energy demand and CO_2 emissions can be reduced by up to 75 %. 3D printing-induced light weighting also leads to usage savings, amounting to 63 % savings in energy and CO_2 emissions over the entire life cycle of the product. This shows that 3D printing has great environmental potential beyond just manufacturing of products.

3D printing enables a buy-to-fly ratio of nearly 1:1, leading to a significant reduction in resource requirements and manufacturing-related waste. Case studies show that up to 40 % of raw material-related waste can be avoided by 3D printing, while 95-98 % of non-melted raw material can be reused (Petrovic Filipovic *et al.*, 2011). Other indirect manufacturing inputs can be avoided as 3DP does not require auxiliary materials such as coolants, lubricants or other substances that are sometimes harmful to the environment.

In general, 3D printing methods have better environmental characteristics. In terms of low carbon emission, there are five primary environmental benefits: (1) Reduction of raw material requirements in the supply chain. This reduces the mining and processing of ores as primary materials. (2) Reduced need for energy-intensive manufacturing processes, such as casting, and wasteful/harmful materials, such as cutting fluids in CNC machining. (3) Flexibility in designing more efficient components with better operational performance. (4) Reduced weight of products, helping to improve carbon footprint when used in the vehicle they are integrated into, e.g. aircraft. (5) Parts could be manufactured closer to the point of consumption, reducing energy consumption in logistics (Peng, 2017). All five points speak in favor of our 3D-printed versions from PLA and PLA-wood.

Despite the potential increase in recycling rates, the materials used for AM are not necessarily more environmentally friendly than those used in traditional manufacturing. The only exception could be the biopolymer polylactic acid (PLA) (Faludi *et al.*, 2015).

Potential material savings may be partially offset by the relative toxicity of the material used for AM and the impact of energy consumption to produce the feedstock and the processing itself. Therefore, the full environmental performance of AM needs to consider the energy demand from a system perspective and not just the process itself (Faludi *et al.*, 2015).

Wood fiber/flour stands out as a premier choice among raw materials for manufacturing plant fiber-plastic composites. PLA, a compostable synthetic polymer made from a monomer feedstock derived from corn starch, is an acceptable substitute for petroleum-derived plastics. The integration of wood-based materials into AM has garnered considerable attention, primarily due to their dual advantages: a favorable ecological footprint and enhanced material attributes (Tao *et al.*, 2017).

However, little research has been done on the toxicity and environmental impact of AM processes and materials. Such effects may exist in the processing and disposal of materials used in AM processes (Ford and Despeisse, 2016).

The fact that there are not many papers studying environmental impact of 3D-printed PLA-wood products led us to perform a comparative LCA analysis for the existing conventional product (metal chair connector) and the 3D-printed alternatives made of biocompatible material PLA and PLA-wood blend material (according to manufacturer wood makes up to 40 %). This case study presents a comparative sequence LCA of a part produced by two different manufacturing processes - Conventional Manufacturing (with milling, drilling, welding) and 3D printing process (FDM – Fused Deposition Manufacturing). A specific part – a chair connector - made of metal is analyzed from cradle to gate. The LCA is analyzed to provide a framework to choose the most appropriate manufacturing process in terms of environmental impact.

Figure 1 Chair design – chair type: domestic seating for adults (Cvetko, 2020)
Slika 1. Dizajn stolice – tip stolice: kućni namještaj za sjedenje namijenjen odraslima (Cvetko, 2020.)

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 A case study: A chair connector
2.1. Studija slučaja: poveznik stolice

A metal connector from a modern chair for domestic use (Figure 1) was chosen for a case study. The chair consists of the following components:

- the shell of the seat and backrest are made of plastic composite material and represent the seat part of the

a) b) c)

Figure 2 a) 3D model of connector (SolidWorks), b) connector made of PLA material, c) connector made of metal (Cvetko, 2020)
Slika 2. a) 3D model poveznika (SolidWorks), b) poveznik od PLA materijala, c) poveznik od metala (Cvetko, 2020.)

Table 1 Three versions of connectors defined with material usage, printing time, material price and weight
Tablica 1. Tri verzije poveznika definirane utroškom materijala, vremenom printanja, cijenom materijala i masom

Version *Verzija*	**Material** *Materijal*	**Print time, h** *Vrijeme printanja,* h	**Material used, g** *Utrošak materijala,* g	**Price of material, €/kg** *Cijena materijala,* €/kg	**Weight, g** *Masa,* g
Metal connector / *metalni poveznik*	metal	/	no data	no data	1340
Optimized connector 2-PLA *optimizirani poveznik 2-PLA*	PLA	13 h 13 min	387	19.52 (7.03 per item)	360
Optimized connector 2-WPC *optimizirani poveznik 2-WPC*	PLA-W	14 h 58 min	444	30.50 (15.12 per item)	347

chair. The seat part, in which there are four upholstery nuts, is fixed to the metal connector with four M6×14 screws.

- another component of the chair is a metal connector used to attach four oak legs and the seat part. The four oak legs are screwed to the metal connector with eight M6×45 screws and nuts.

The materials included in this case study are an original metal-based, alternative 3D- printed part from polylactic acid and a 3D-printed part from a filament mixed with PLA and wood.

2.2 3D printing of PLA and wood-PLA connectors

2.2. 3D printanje PLA i drvo-PLA poveznika

The digital models of 3D parts were modeled in SolidWorks software (SolidWorks Corp., Waltham, MA, USA) and exported to STL format. The STL models were sliced and prepared for 3D printing in Z-Suite software (Zorttrax, Olsztyn, Poland).

Parts used for the LCA comparison with original metal connector (from Maxxim, Dipo, reference unknown) were printed on Zorttrax M-200 (Olsztyn, Po-

Table 2 Life cycle inventory database used for different versions of connector (SimaPro references written in both bold and italic letters)
Tablica 2. Baza podataka inventara životnog ciklusa koja se primjenjivala za različite verzije poveznika (SimaPro reference napisane su podebljanim i kurzivnim slovima)

	Life cycle inventory database / *Baza podataka inventara životnog ciklusa*		
Life cycle stage *Faza životnog ciklusa*	**Metal connector** *Metalni poveznik*	**3D-printed PLA connector** *3D isprintani PLA poveznik*	**3D-printed PLA-wood connector** *3D isprintani PLA-drveni poveznik*
Premanufacturing or raw material extraction *pretproizvodnja ili ekstrakcija sirovina*	- materials processing - input from nature: ***iron ore*** */ obrada materijala - ulaz iz prirode: željezna ruda* - transformation input: *electricity, medium voltage, SI / transformacijski ulaz: električna energija, srednji napon, SI*	***- polylactide, granulate*** / *polilaktid, granulat* - filament production – input: ***electricity, medium voltage, SI /*** *proizvodnja filamenta – ulaz: električna energija, srednji napon, SI*	***- polylactide, granulate*** / *polilaktid, granulat* ***- wood wool*** (as replacement for wood flour) / *drvena vuna (kao zamjena za drvno brašno)* - filament production – compounding – *electricity**, medium voltage, SI*** / *proizvodnja filamenata – kompaundiranje – električna energija, srednji napon, SI*
Manufacturing and processing *proizvodnja i prerada*	- product manufacturing – processes (***sheet rolling, milling, drilling, welding***) / *izrada proizvoda – procesi (valjanje lima, glodanje, bušenje, zavarivanje)* *- transport from processing site to manufacturing site:* ***transport, freight, lorry 16-31 metric ton, euro5*** / *prijevoz od mjesta obrade do mjesta proizvodnje: prijevoz, teret, kamion 16-31 metrička tona, euro5* ***- electricity, medium voltage, SI*** / *električna energija, srednji napon, SI*	- product manufacturing – 3D printing; input is ***electricity, medium voltage, SI /*** *izrada proizvoda – 3D ispis; ulaz je električna energija, srednji napon, SI*	- product manufacturing – 3D printing; input is ***electricity, medium voltage, SI /*** *izrada proizvoda – 3D ispis; ulaz je električna energija, srednji napon, SI*
Transportation *transport*	- transport from manufacturing site to distribution site ***transport, freight, lorry 16-31 metric ton, euro5*** / *prijevoz od mjesta proizvodnje do mjesta distribucije: prijevoz, teret, kamion 16-31 metrička tona, euro5*	- no transport – 3D printing takes place at manufacturing site / *nema transporta – 3D ispis se obavlja na mjestu proizvodnje* - distribution to individual consumer is excluded / *isključena je distribucija pojedinačnom potrošaču*	- no transport – 3D printing takes place at manufacturing site / *nema transporta – 3D ispis se obavlja na mjestu proizvodnje* - distribution to individual consumer is excluded / *isključena je distribucija pojedinačnom potrošaču*
Usage / *uporaba*	- all 3 products have the same use phase / *sva tri proizvoda imaju istu fazu uporabe*		
End-of-life or waste disposal *kraj životnog vijeka ili odlaganje otpada*	- wasted metal is melted down for reuse / *otpadni se metal tali za ponovnu upotrebu*	- wasted PLA is recycled for reuse (or abiotically degraded) / *otpadni PLA se reciklira za ponovnu upotrebu (ili se abiotički razgrađuje)*	- wasted PLA is recycled for reuse (or abiotically degraded) / *otpadni PLA se reciklira za ponovnu upotrebu (ili se abiotički razgrađuje)*

land). Pure PLA filament and PLA-wood filament (with up to 40 % wood flour content), both commercially available, were used. The diameter of the filament was 1.75 mm, the diameter of the print nozzle was 0.6 mm, the layer thickness was set to 0.4 mm and the infill to 40 %.

2.3 LCA methodology
2.3. LCA metodologija

The study applied the LCA methodology based on the standard ISO 14044:2006 following four major steps to quantify the difference in environmental impact between conventional metal connector and two 3D printed alternatives. A "cradle-to-gate" evaluation was conducted within the SimaPro 9.0 software, developed by PRé Sustainability, Amersfoort, 2019. Additionally, all phases to the end of the life cycle were also taken into consideration, based on data from scientific papers. SimaPro 3D printing is not supported by SimaPro, as it was not yet included in the library at the time of this study. The geography for the manufacturing and distribution phases were set in Slovenia at time horizon 2021. The process trees are presented in Figure 4 and the input information of the Life Cycle Impact Assessment (LCIA) in Table 2.

For consistency, it was assumed that the input mass of all three versions of chair connector is 1 kg of raw material.

It was also assumed that all transport is carried out by road with a truck (SimaPro reference: Transport, freight, lorry 16-32 metric ton, EURO5 {GLO}). The geography for the manufacturing and distribution is set in Slovenia, where distances are short (typically less than 50 km) and no specific locations were determined (of manufacturing companies, etc.). For this study, it was assumed that the metal is processed by manufacturer A, while the metal part is formed by a nearby subcontractor and later distributed to a warehouse B (presumably in Kranj; halfway between Jesenice and Ljubljana - to negate the impact of distances between production sites, as they are fictitious). On the other hand, filaments are extruded by manufacturer B in Ljubljana and 3D parts are printed and assembled in warehouse B. In this sense, the 3D printing variants eliminate some manufacturing and transportation phases – considering that 3D printing can be produced at the same site where it is later assembled.

The classification and characterization processes were carried out according to the standard ISO 14040:2006. For the assessment of impacts, ReCiPe 2016 Midpoint (Hierachist) was applied to calculate the environmental impacts, and 18 impact categories were included in the LCA. Midpoint characterization factors are calculated based on a consistent environmental cause-effect chain, except for land-use and resources. Its regional validity is Europe; it is global for climate change, ozone layer depletion and resources. Its temporal validity is present time.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Life cycle impact assessment of three versions of chair connector
3.1. Procjena utjecaja na okoliš životnog vijeka triju verzija poveznika za stolicu

The results of the cradle-to-gate comparative LCA between the three types of chair connectors are shown in Figure 3, 4 and 5.

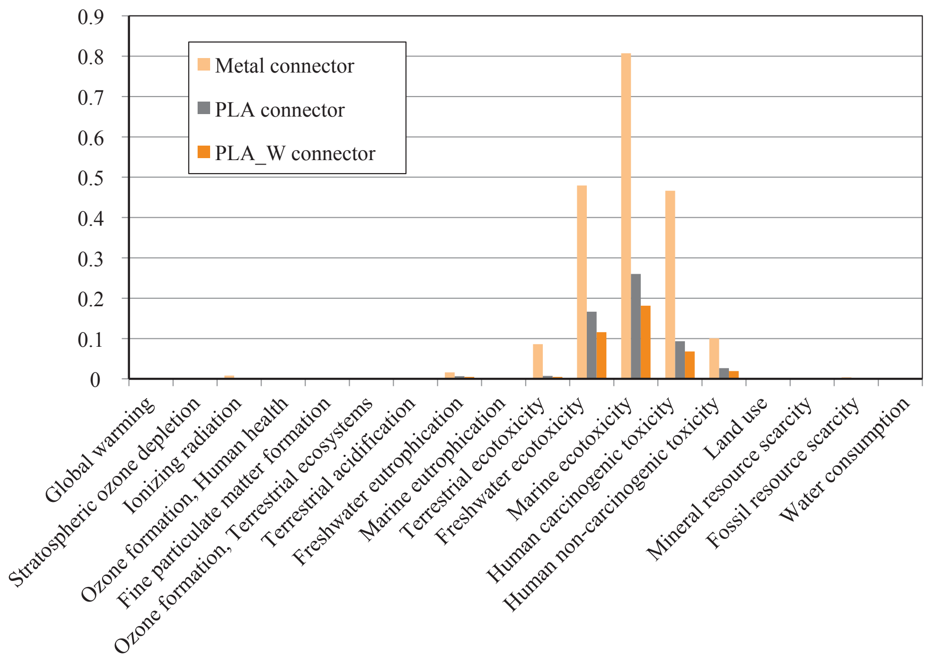

Figure 3 Impact assessment of 3 versions of chair connectors using ReCiPe 2016 Midpoint (H) method with normalization of results
Slika 3. Procjena utjecaja na okoliš triju verzija poveznika za stolicu primjenom metode ReCiPe 2016 Midpoint (H) s normalizacijom rezultata

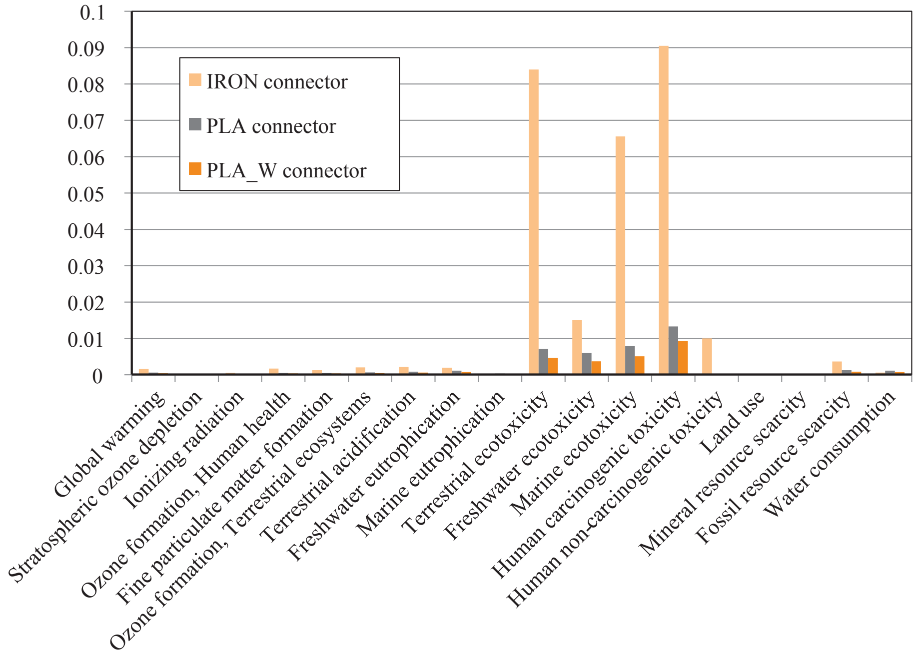

Figure 4 Impact assessment of 3 versions of chair connectors using ReCiPe 2016 Midpoint (H) method with normalization of results and excluded long-term emissions
Slika 4. Procjena utjecaja na okoliš triju verzija poveznika za stolicu primjenom metode ReCiPe 2016 Midpoint (H) s normalizacijom rezultata i isključenim dugoročnim emisijama

Normalization results can be used to compare different categories of impacts, as these impacts are individually converted by a multiplication factor to have all impacts in a single unit or ratio form.

Considering all three versions of connector, Freshwater and Marine ecotoxicity, and Human carcinogenic toxicity are the major environmental impacts. In all three impact categories, the metal connector has by far the highest values (70-80 %). As shown in Figure 5, this is due to operations related to processing of iron, part milling operation, high electricity usage and transport. When comparing the two 3D-printed versions, it is evident that the values of PLA-wood connector are 20 to 30 % lower than those of pure PLA connector.

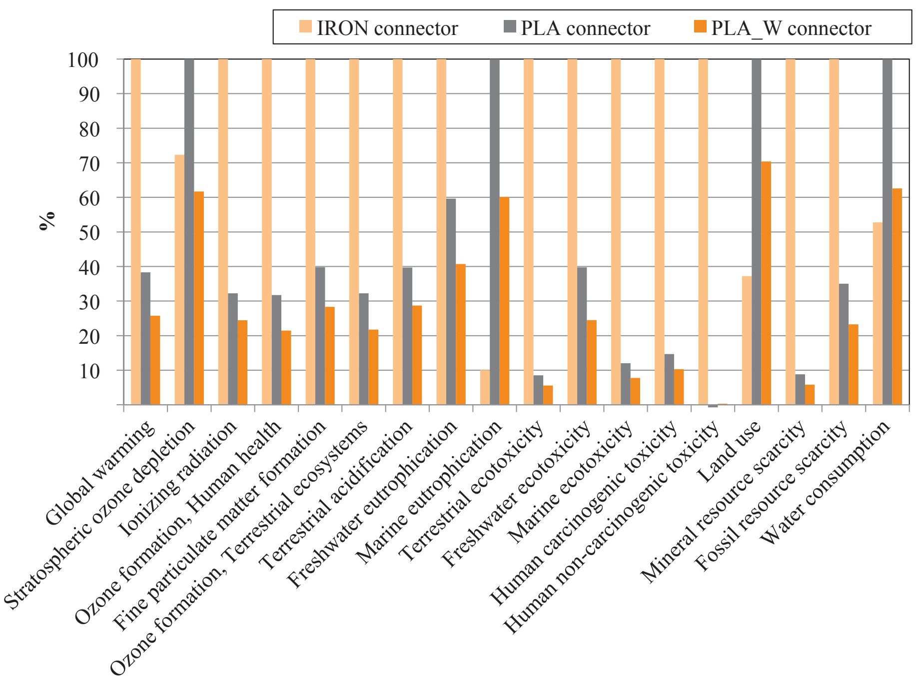

Figure 5 Impact assessment of 3 versions of chair connectors using ReCiPe 2016 Midpoint (H) method with characterization of results, excluding long-term emissions
Slika 5. Procjena utjecaja na okoliš triju verzija poveznika za stolicu primjenom metode ReCiPe 2016 Midpoint (H) s karakterizacijom rezultata i isključenim dugoročnim emisijama

Comparing all three versions of connector, when excluding long-term emissions, Terrestrial and Marine ecotoxicity, and Human carcinogenic toxicity are the major environmental impacts. In all three impact categories metal connector has by far (above 80 %) the highest values. Comparing the two 3D-printed versions, PLA-wood connector has around 30 % lower values than pure PLA connector. All three impact categories are defined by the use of electricity in 3D printing and also by the raw material used, i.e. PLA.

In Characterization, all results are plotted on a percentage scale.

The carbon footprint is the sum of greenhouse gas emissions caused directly or indirectly by an organization, product, service, or other activity that causes or contributes to greenhouse gas emissions over a period of time. It is defined in units of CO_2 equivalents (CO_2e) (Le Treut *et al.*, 2007). Impacts are calculated per unit of CO_2e of the six major greenhouse gasses (GHGs). The average of all these gasses causing global warming is known as Global Warming Potential (GWP) and is usually given in the time frame of 100 years.

The 3D-printed alternatives showed (Figure 5) 62 % (PLA) and 73 % (PLA-wood) lower GWP than the conventional metal part. However, the results of the cradle-to-gate life cycle assessments suggest that the 3D-printed PLA alternative may cause greater environmental impacts than the conventional products in some impact categories – Stratospheric ozone depletion, Marine eutrophication, Land use and Water consumption.

In terms of LCA, 3D-printed PLA-wood and PLA alternatives would be much more environmentally friendly compared to conventional products, although the environmental benefits might be insignificant from the manufacturer's point of view.

3.2 Life cycle interpretation (LCI)
3.2. Tumačenje životnog vijeka (LCI)

The aim of our case study was to determine with a quantitative analysis whether a 3D-printed product can be a sustainable alternative to the conventional connector from the manufacturing and material point of view.

3DP induces CO_2 emission reduction potentials over the entire lifecycle of a product. The life cycle of the connector component includes several phases, from raw material production/acquisition to the end-of-life phase of the connector. For each phase, the data from literature is described to estimate the environmental impacts throughout the connector life cycle.

The transportation of each material to the manufacturing site (which is assumed to be Slovenia) has also been considered (and described previously in subchapter "LCA analysis" of chapter Method and Materials).

3.2.1 Raw material phase
3.2.1. Faza sirovine

In iron ore processing, substantial solid waste, mainly slag, could be repurposed as secondary raw materials, aligning with circular economy principles. This also applies to energy waste from steelmaking. The most environmentally impactful stages are blast furnace and coke oven operations, driven by energy consumption and emission toxicity (Renzulli *et al.*, 2016). Raw materials (coal, pulverized coal, iron ore) are largely imported (Australia, South Africa, USA, Canada, Venezuela, Brazil, Mauritania).

Despite high initial production impacts, metal benefits from usage and end-of-life recycling, offsetting compared to non-metallic alternatives. A cradle-to-gate study is limited for LCAs involving metals, meanwhile cradle-to-grave provides comprehensive insights (ISO 14040/14044) (Santero and Hendry, 2016).

Metal production averages 20-25 MJ (5550-6950 Wh)/kg (Low-tech magazine, 2023), 6 kWh in SimaPro. Furthermore, crude PLA synthesis and filament conversion are considered, assuming 15 kg/hour production with 1 kWh/kg consumption and no extrusion waste.

For PLA-wood chair connectors, wood benefits (cost, renewability, recyclability, non-toxicity, reduced plastic use) attract manufacturers as a filler. Wood, cost-effective vs. petroleum/bioplastics, is combined with polymers for diverse performance (Ayrilmis *et al.*, 2019).

In raw material stage, metal connectors show higher environmental impact due to primary sourcing, meanwhile PLA and PLA-wood use renewable sources (plant-derived starch, trees).

3.2.2 Manufacturing phase
3.2.2. Faza proizvodnje

Observed sustainability challenges for the case-studied part include: (a) material and process standardization, (b) limited speed and reliability of AM technologies, (c) constrained quality and aesthetics of products, (d) cost efficiency and energy efficacy enhancement at higher production volumes.

From a sustainability standpoint, AM's additive nature reduces waste compared to subtractive techniques, despite potential higher energy intensity per unit. AM's make-to-order capacity aligns with better overall performance and dematerialization due to increased raw material utilization (Chen *et al.*, 2015).

AM's direct production from 3D CAD models eliminates tooling costs, promotes design sharing, customization, and faster prototyping. Energy intake involves electrical and material-based energy. AM's main environmental benefits over CNC machining are less waste and lower pollution, especially from metalworking fluids (Huang *et al.*, 2013).

While AM's energy consumption can be higher than conventional methods, benefits emerge from utilization rates. Sharing machines reduces environmental impact. Fused Deposition Modeling (FDM) shows lowest environmental impact per part with both high and low utilization (Ford and Despeisse, 2016).

3D printing's energy consumption surpasses that of injection molding, but it's relatively low in FDM. Material waste is minimized, but print time affects energy consumption. Lightweight 3D-printed designs reduce fuel costs during use (Gebler *et al.*, 2014).

In terms of production costs, 3D printing introduces shifts in the cost structure with a focus on machine costs. Material costs are case-specific but often comprise a smaller portion, although 3D printing materials are costlier, they pay off due to higher material efficiency (Reeves, 2008).

Comparatively, FDM 3D printing is cost-efficient below a break-even point, making it optimal for specific production volumes (Hopkinson *et al.*, 2006). 3D printing generates less waste than conventional methods, but supports and failed prints contribute to post-processing waste.

Hybrid manufacturing processes offer advantages such as improved surface quality and shorter production times. Integrating AM with traditional processes enhances these benefits (Ford and Despeisse, 2016).

3.2.3 Use phase
3.2.3. Faza uporabe

Concerning product use, the lightweight of a 3D-printed part must be highlighted. Both 3D-printed parts are approximately 4 times lighter than original metal part.

Optimized 3D-printed connectors were printed from polylactic acid (PLA) and wood-plastic composite and tested in the Furniture Testing Laboratory according to the requirements of the SIST EN 12520:2010 standard. The optimized 3D-printed connector made of PLA material met the requirements of the standard, and the connector made of wood-plastic composite did not, as a fracture occurred.

Observed sustainability challenges for the case-studied part are: (a) uncertain performance of product and component due to low maturity of technology and (b) uncertain performance of product and component over a longer lifetime.

3.2.4 Repair and remanufacturing phase
3.2.4. Faza popravka i ponovne proizvodnje

Another important segment is that of spare parts. If a part is broken and the replacement part is no longer manufactured by the industry, the entire object needs to be thrown away, resulting in various environmental impacts. However, if the spare part can be printed, the object will last longer and the process time for repair is reduced. This unequivocally contributes to sustainability objectives by mitigating waste generation and consequently reducing the associated carbon footprint.

Companies are beginning to discover the impact of using AM technologies to extend product lifecycles and close the loop (Ford and Despeisse, 2016).

3.2.5 End-of-life phase
3.2.5. Faza kraja života

In the end-of-life phase, metal's negative environmental impact turns positive due to its recyclability advantage. The highest value recovery occurs locally during manufacturing when unused AM material (powder or resin) is reclaimed. One the other hand, approximately 95-98% of metal powders can be reused (Petrovic Filipovic *et al.*, 2011).

Ford and Despeisse (Ford and Despeisse, 2016) noted that AM can integrate in situ recycling to divert waste to new applications. Simplified recycling systems are feasible with increased PLA use and reduced plastic variety. PLA's recyclability without quality loss enables closed material loops (Chen *et al.*, 2015).

'Biodegradable' materials decompose based on conditions. Composting is a controlled process. According to EN 13432, 'compostable' means 90 % conversion in industrial composting within 6 months. PLA degrades rapidly under these conditions, but takes decades in the wild, contributing to pollution (3Dnatives, 2023).

PLA degradation mechanisms include hydrolysis, thermal degradation, and photodegradation. Slow ambient degradation is observed, and PLA persists in certain environments (Bagheri *et al.*, 2017; Karamanlioglu and Robson, 2013).

AM enhances material recycling efficiency, raises awareness, and promotes recycled material acceptance.

4 CONCLUSIONS
4. ZAKLJUČAK

Industrial sustainability is a persistent priority, with growing emphasis on enhancing production efficiency and environmental harmony. Sustainable development seeks to reduce the ecological impact of manufacturing, a pursuit achievable through AM.

The focal point of this case study revolves around the comprehensive evaluation of diverse materials and manufacturing methodologies employed in the fabrication of a chair connector, uniting four oak legs with the seat component. This project aimed to assess the environmental life cycle of 3D-printed parts comparing the result with conventional metal part.

It was found that a metal part, manufactured with conventional technologies, has a higher environmental impact compared to 3D-printed alternatives from renewable materials.

These observations are due to the fact that the 3D printing uses significantly smaller amount of material as it is an additive manufacturing- in other words, it generates less waste during manufacturing, it is possible to optimize geometries and create lightweight components that reduce material consumption during manufacture and energy consumption during use, it reduces transportation in the supply chain and an inventory waste due to the ability to manufacture spare parts on demand.

In addition, it was found that the material used can strongly influence the environmental footprint in other impact categories, leading to important tradeoffs. Challenges in AM with biodegradable materials, such as wood composites, include processing issues during extrusion and part fabrication, particularly with respect to part dimensional stability and material brittleness depending on the degree of stress on the wood components, as well as effects on polymer crystallization behaviour during processing (Gardner and Wang, n.d.). In certain cases, the mechanical and physical properties of the printed parts can approach the property range of conventional wood composites such as particleboard, fiberboard, and wood-thermoplastic composites. With the proper incorporation of wood fibers, nanocellulose and continuous fiber printing, this could even be improved.

However, there is still an open question of PLA material environmental impact. It is, nevertheless, a renewable material, derived from natural, plant-based starch. Furthermore, it is advertized as biodegradable although studies (Bagheri *et al.*, 2022; Castro-Aguirre *et al.*, 2016; Karamanlioglu and Robson, 2013; Rezvani Ghomi *et al.*, 2021) show that its degradability is either abiotic (with hydrolysis, thermal- or photo-degradation) or very slow.

As for AM in general, it is still in its early stages and requires further research to reduce material and machine costs, create faster and more accurate printing processes, and operate autonomously (Gopal *et al.*, 2023).

The case study of a wooden chair with three different connectors is based on several assumptions, and future work (additional LCA analysis and comparison) is needed to better understand the environmental impact of 3D-printed products. This approach aligns with the principles of the European Bauhaus framework, a paradigm endeavouring to synthesize sustainability, aesthetic considerations, and innovative approaches.

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Assessment of Condition of Wooden Mill in Kovačevići Area in Bosnia and Herzegovina

Procjena stanja drvenog mlina na području Kovačevića u Bosni i Hercegovini

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ABSTRACT • *Wood is one of the most important materials that has been used for several millennia. It is therefore not surprising that wood plays an important role in the cultural and technical heritage of several European countries and beyond. An excellent example of cultural and technical heritage is a wooden mill, almost 100-year-old, near Cazin in Bosnia and Herzegovina. These mills played an important role, especially in times of Bosnian war (1992-95), when this region was cut off from electricity. The microscopic analysis of the wood materials used in the mills revealed that the mills were made of chestnut (Castanea sativa) and oak (Quercus sp.) wood. Sufficient durability of these wood species resulted in good structural integrity of the mills. The surface of the wood materials in the mills showed partial degradation patterns caused by weathering over the years. However, the interior parts of the wood materials were intact probably due to smoke deposits from the open fireplace. It is suggested that the roofing in the mills should be maintained regularly to prevent possible leaks to protect this heritage for future generations.*

KEYWORDS: *cultural heritage; technical heritage; wood; non-destructive assessment; decay*

SAŽETAK • *Drvo je jedan od najvažnijih materijala koji se u industriji i graditeljstvu upotrebljava već nekoliko tisućljeća. Stoga ne čudi da drvo ima važnu ulogu u kulturnoj i tehničkoj baštini više europskih zemalja. Izvrstan primjer kulturnoga i tehničkog nasljeđa drveni je mlin u blizini Cazina, u Bosni i Hercegovini. Takvi su drveni mlinovi imali izrazito važnu ulogu u ratnim vremenima, kada je na ovom području bila prekinuta opskrba električnom energijom. Mikroskopskom analizom utvrđeno je da su mlinovi najčešće izgrađeni od drva pitomog kestena (Castanea sativa) i drva hrasta (Quercus sp.). Dobra trajnost pojedinih vrsta drva od kojih su mlinovi izgrađeni rezultirala je njihovom dobrom strukturnom cjelovitošću. Površina drva promatranog mlina djelomično je oštećena vremenskim utjecajima. Interijer je zaštićen od biološke razgradnje zbog naslaga dima koji je dolazio s otvorenog kamina. Važno je naglasiti da krovište drvenih mlinova treba redovito održavati kako bi se spriječilo potencijalno prokišnjavanje te baština zaštitila za buduće generacije.*

KLJUČNE RIJEČI: *kulturna baština; tehnička baština; drvo; nerazorna procjena; propadanje*

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1 INTRODUCTION
1. UVOD

Wood is subjected to biotic and abiotic degradation processes when used in outdoor applications (Eaton and Hale, 1993; Hasan *et al*., 2008). In nature, these processes are desirable and necessary as they contribute to the carbon cycle and soil formation. However, when wood is used for technical purposes (constructions, bridges, buildings), the degradation processes should be slowed down as much as possible. Under favourable conditions, wood can become a nutrient for a variety of organisms that degrade one or more components of the wood to the point where they can metabolise them. Among biotic decomposers, fungi cause the most damage, while insects cause somewhat less damage in temperate climates (Schmidt, 2006; Humar *et al*., 2020).

The intensity of decomposition of wood has increased in the last decade due to climate change. Wood, in similar applications, is decaying faster today than it did decades ago. This difference is most pronounced in Alpine valleys (Humar *et al*., 2021; Van Niekerk *et al*., 2022). The main reasons for the increased fungal decay intensity are higher temperatures, milder winters and more precipitation in the winter months (Humar *et al*., 2021).

Wood has been one of the most widely used materials by mankind in the course of its long evolutionary history. Wood has been used for structural applications and for decorative, ritual and religious purposes. People's close association with wood over thousands of years has been embedded in the cultural heritage made of wood and represents people's lives and values (Kim and Singh, 2016). Many of the historic objects made of wood reflect the availability of wood and the processing tradition as well as its natural origin and beauty (Lo Monaco *et al*., 2018).

The water mills in the Kovačevići and Cazin area are a testimony to the ingenuity and resourcefulness of the local people in using the natural resources at their disposal. These mills were used to grind wheat and maize and provided the local population with flour and other essentials. All the mills had been built by the local communities of Ćoralići and Liskovac. The importance of these water mills cannot be overestimated, as they had played a crucial role in the survival of local communities, especially in times of war during WWII and the Bosnian war (1992-95). However, the need for water mills declined over time, and many were abandoned (Čekić, 2021). In Kovačevići, there are five wooden mills, while in the Cazin area, there are approximately 50 such water mills.

They remain important for the cultural and technical heritage of Bosnia and Herzegovina. Each of these mills had its own name, reflecting local traditions and culture. For example, "Vilenica", one of the older water mills in the area, was built by the local Kovačević family. Other mills were named after their location or owners, such as Novak, Čardak, Mali mlin and Mlin Slap. The construction of the water mills was crucial for their functioning, as the water flow had to be regulated to ensure enough energy to drive the millstones. Most of the mills were built from local, durable wood species such as sweet chestnut (*Castanea sativa* Mill) and oak (*Quercus sp*.) (Čekić, 2021).

The chestnut and oak forests in Bosnian Krajina are well known. They are widespread in the wider surroundings of Cazin, Velika Kladuša, Bužim and part of Bosanska Krupa. According to the forest inventory in Bosnia, oak grows on more than 8000 ha and sweet chestnut on 6000 ha of the forests in Una-Sana Canton, providing natural material for a wide range of uses (Federalna uprava za šumarstvo, 2012). Oak and sweet chestnut are ring-porous species with coloured heartwood. The sapwood is usually narrow. Large vessels in the early wood are typical for oak (diameter over 200 µm) and somewhat smaller for the sweet chestnut, and radially oriented groups of small vessels in latewood. In addition, rays are clearly visible in oak species, while in sweet chestnut wood rays are visible only under magnification (Wagenführ, 2014). Oak wood is traditionally used for the production of wooden structures, bridges, railway slippers, barrels and furniture (Dogu *et al*., 2017). Oak wood is characterised by high variability in durability and is therefore classified in durability classes from DC 2 (durable) to DC 4 (less durable) according to EN 350 (Brischke *et al*., 2013; Meyer *et al*., 2014). The reason for the classification of oak wood in low durability classes is related to the high variability of the chemical and anatomical structure of wood (Meyer *et al*., 2014). Part of the variability can be related to the ring width. It is reported that the durability of oak with a ring width of less than 1 mm is comparable to that of beech. The vessels dominate the narrower sapwood with large lumens, which reduces the effectiveness of water exclusion and durability (Humar *et al*., 2008). On the other hand, sweet chestnut is the most durable wood species in Europe, along with black locust (*Robinia pseudoacacia* L.), with much less variation in durability (Diamandis, 2010; Eichhorn *et al*., 2017).

Oak and sweet chestnut wood is decomposed by both white rot fungi (e.g. *Donkioporia expansa* (Desm.) Kotlába & Pouzar*, Laetiporus sulphureus* (Bull.) Murrill*, Trametes versicolor* (L.) Lloyd*, Stereum hirsutum* (Willd.) Pers) and brown rot fungi (*Daedalea quercina* (L.) Pers.*, Antrodia sp*.) (Schmidt 2006). Insects that attack older oak wood include *Bostrychus capucinus* L. and *Anobium punctatum* De Geer. Ants (*Formica sp*.)

have recently become more common on wood including oak wood (Unger *et al*., 2001; Humar *et al*., 2022).

After the end of the Bosnian war (1992-1995), the need for water mills was no longer present and they were abandoned. However, these mills have recently become a symbol of Gračanica and the local communities. As they were not properly maintained after the war, they started to deteriorate. Therefore, efforts have been made to preserve and restore them for future generations. Thus, it is necessary to determine their condition in order to prepare recommendations for conservation and protection for future generations. Moreover, the mills are an excellent example to study the performance of wood in harsh environments.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

The analysis was carried out at one single mill (Figure 1) founded at the entrance to the gorge of the Gračarica River near the village of Kovačevići (GPS coordinates: 45.020898, 15.871152), whilst there are several other mills at this location. The investigated mill is a representative example of traditional architecture and construction. It was powered by water as the region was cut off from the leading electricity grid during the war in the 1990s. The wooden mill in question was built in the early 1930s and rebuilt after the floods of 1962. The majority of the construction is almost 100 y old. In the 60s the roofing, water wheel and water channels were rebuilt. The analysis of the mill described in this paper was carried out on November 16, 2022.

Fungal spore concentrations found in the air were determined at four sites: two indoors and two outdoors. The control was outdoor air. PDA (Potato Dextrose Agar) medium (Difco) was used as the medium. Namely, 100 L air samples were taken. MAS-100 VF sampler from Merck was used. After sampling, the media plates were sealed with parafilm and incubated at 25 °C in a dark climate chamber. The plates were inspected daily and after 72 hours the colonies grown were identified and the number of colony forming units (CFU) per m^3 (CFU/m^3) was calculated. The presence of spores was determined, and only the crucial individual species were identified.

Figure 1 Water mill in the area of Kovačevići
Slika 1. Vodeni mlin u naselju Kovačevići

The moisture content of the wood in the watermill was determined using an electrical resistance metre from GANN (Gann GmbH, Gerlingen, Germany), which allows measurement of wood moisture content between 6 % and 60 % (Otten *et al*., 2017). The moisture content of the wood was determined as usual in wood technology (the ratio between the weight of water and the weight of the wood in an absolutely dry state, expressed in %).

Wood degradation was assessed using an IML PD500 resistograph (IML Instrumenta Mechanik Labour System GmbH, Wiesloch, Germany) based on the recording of the drilling resistance. A hole was drilled into the wood with a tiny drill with a diameter of 2 mm and the resistance of the material to drilling was recorded. The method is based on the fact that less energy is needed to drill a hole in decayed wood than in sound wood. If the device does not record the resistance, the wood is heavily decomposed (Sharapov *et al*., 2019). Alternatively, non-recording of the drilling resistance could also be the result of cracks, holes or other voids.

The mechanical properties of the wood were determined non-destructively. The speed of sound was determined using a device manufactured by Fakopp Microsecond timber. Two electrodes were driven into the wood at a distance of 500 mm. Then the emitting electrode was tapped with a 100 g steel hammer and the transit time of the sound was determined. From the distance and time, the sound transmission was determined. Based on the wood density and sound transmission data, the dynamic modulus of elasticity was calculated according to Eq. 1.

$$dMoE = \rho \times v^2 \quad (1)$$

Where:
dMoE – dynamic modulus of elasticity, MPa,
v – sound velocity, m/s
ρ – density, kg/m^3.

It should be noted that the dynamic modulus of elasticity is, as a rule, consistently higher than the static modulus. Dynamic determination of the modulus of elasticity, compared to static determination, usually gives about 10 % to 20 % higher values (Chauhan and Sethy, 2016). Measurements were performed on the areas without cracks, knots and growth anomalies with parallel fibre orientation.

At selected sites, density was determined indirectly by the screw withdrawal technique. A screw with a diameter of 3 mm and a depth of 15 mm was screwed into the wood with a screwdriver. The maximum force required to extract the screw was determined using a Screw Withdrawal Resistance Meter (Fakopp Enterprise Bt). The screw extraction force correlates well with the mechanical properties and is a good indicator of the initial, invisible stages of decay (Xue *et al*., 2019).

In addition, four samples were isolated from the mill at representative locations, as presented in Table 1. These samples were examined in detail in the laboratory, as described below. Microscopic analysis was performed using an Olympus DSX1000 digital microscope (Olympus, Tokyo, Japan). The surface area and cross-section of the selected samples were analysed. Before the microscopic analysis, the transverse xylotome plane was prepared using a GSL 1 sliding microtome (Künten, Switzerland). The analysis was performed under mixed illumination (light and dark field).

Wood density was determined using a GeoPyc device (Micrometics, Norcross, USA), which allows accurate volume measurement with a dry, flowable medium made of a mixture of very small graphite particles and ceramic microspheres. The oven-dry mass of the samples was determined before the measurement. The measurements were performed on absolutely dry wood, as the system is best suited for determining the density of wood in the absolutely dry state, where the moisture content of the sample does not change (Arnič *et al*., 2021; Humar *et al*., 2022).

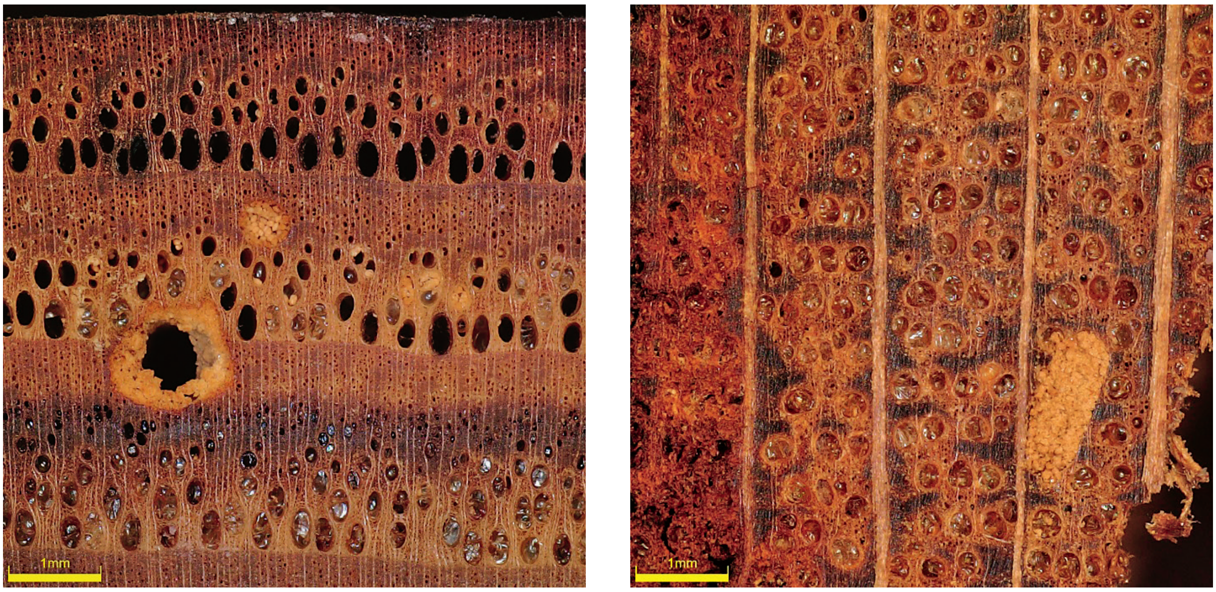
Quantitative elemental analysis was also performed on isolated samples. The inorganic elemental content of the samples was determined using an XRF TwinX X-ray fluorescence spectrometer manufactured by Oxford instruments (Abingdon, UK). Measurements were performed with a PIN detector (U = 26 kV, I = 112 µA, t = 360 s).

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

Microscopic analysis revealed that the mill in question consists predominantly of sweet chestnut (*C. sativa*) and European white oak (*Quercus sp.*) (Figure 2). The ring-porous structure is clearly visible in the images (Figure 2). The presence of the wide parenchyma rays distinguishes the oak from the sweet chestnut (Wagenführ, 2014).

The use of chestnut and oak wood in the construction of the mills indicates that part of the local population was aware of the importance of the use of durable wood species in harsh conditions with high relative humidity. Chestnut and oak wood are known for their durability and resistance to fungal decay (Brischke *et al*., 2013). This made them an ideal material for building the mills and ensured that the structure would last for many decades.

The assessment of the timber in the mill was carried out in November 2022. The outside temperature at the time of the visit was 13 °C, and the relative humidity was between 75 % and 85 %. It should be noted that the mill is located near running water, so high relative humidity is to be expected. The high relative humidity resulted in a relatively high moisture content (*MC*) of the wood (Figure 3). The *MC* of the object varied be-



A) B)

Figure 2 Cross-section of sweet chestnut (*C. sativa*) (A) and oak (*Quercus* spp.) wood sample (B); cross-section holes are caused by insect damage
Slika 2. Poprečni presjek uzorka pitomog kestena (*C. sativa*) (A) i hrasta (*Quercus* spp.) (B). Rupe na poprečnom presjeku oštećenja su nastala djelovanjem insekata.

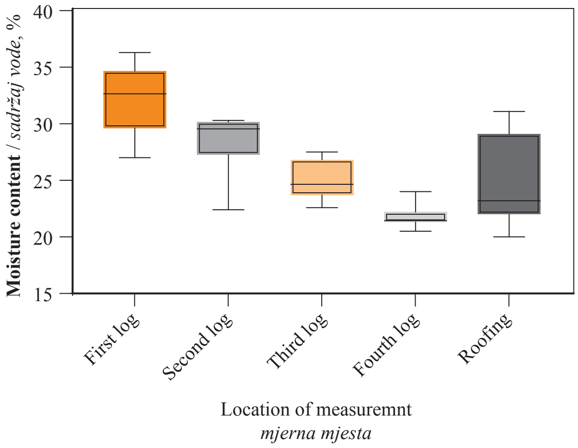


Figure 3 Wood moisture content at various locations in water mill (n = 10)
Slika 3. Sadržaj vode u drvu na različitim mjestima u vodenome mlinu (n = 10)

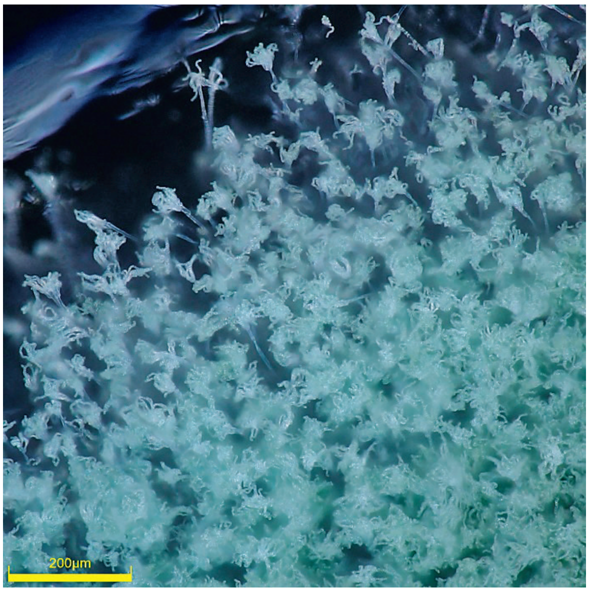


Figure 4 Fungal conidia that developed from collected spores belonging to genus *Penicillium*
Slika 4. Konidije gljivica koje su se razvile iz nakupina spora roda *Penicillium*

tween 20.0 % and 36.3 %. The highest individual and average moisture content was determined on the log that was adjacent to the ground and thus exposed to splash water. The average *MC* decreased with increasing distance from the ground. Relatively high *MC* was determined on the roof elements caused by roof leakage. Although the roofing was still not degraded.

The key question in connection with *MC* is: What is the limiting *MC* for fungal decay? Fungi can decompose wood if the *MC* is above a certain limit. A wide range of data is available in the literature. The first set of data states that the limits of *MC* depend primarily on the fungal species. For example, Schmidt (Schmidt, 2006) reported that the minimum *MC* of wood varies between (30 %) (*Fibriporia vaillantii* (DC.) Parmasto and *Gloeophyllum trabeum* (Pers.) Murrill) and 25 % (*Coniophora puteana* (Shum.: Fr.) P. Karst and *Serpula lacrymans* (Wulfen) P.Karst). However, new data indicate that *MC* limits for fungal growth also depend on the fungal species and wood species. For example, the limit *MC* for the decay of *G. trabeum* on Scoots pine sapwood is 16 %. However, it should be borne in mind that such low-limit values are only possible if the water source is nearby. On the other hand, the limiting moisture content of oak wood varies between 23.0 % (*Coniophora puteana*) and 33.8 % (*Trametes versicolor*) (Meyer and Brischke, 2015). Unfortunately, limit *MC* data are not available for sweet chestnut wood. As can be seen in Figure 3, the *MC* in the respective mill is above the threshold value in several places. It was found that the critical dose (number of days with *MC* suitable for wood decay) corresponding to incipient decay can be seen as more or less independent of the wood species. The critical dose was found to be around 325 days with favourable conditions for fungal decay (Isaksson *et al*., 2013).

One of the prerequisites for fungal infestation is the presence of fungal spores. Therefore, the spore concentration in the mill (1625 CFU/m^3) and before the mill (3480 CFU/m^3) was determined. Usually, between 360 and 1230 CFU/m^3 are reported in the air (Flores *et al*., 2014). As higher values are reported in the vicinity of the mill, this indicates the potential of the fungus in the environment. From the microscopic analysis, it can be concluded that the majority of the colonies identified belong to the genus *Penicillium* (Figure 4). *Penicillium* is a genus of ascomycetous fungi that is part of the mycobiome of several species and is of great importance in the natural environment.

However, in addition to moulds, wood-destroying fungi have also been identified. Secondary mycelium with clamps was found in the vessels of oak wood (Figure 5A). This type of mycelium is characteristic of basidiomycetes, which usually cause decay of wood (Schmidt, 2006). In addition, pink slime moulds were present on the wood surface (Figure 5B). These fungi typically occur on surfaces in high relative humidity environments (Eaton and Hale, 1993).

The outer part of the logs exposed to weathering was grey (Figure 1). Loose cellulose fibres can be seen on the microscopic image. The surface of the wood absorbs light due to the presence of lignin. As a result of UV irradiation, the lignin is degraded and leached from the surface layers. Loose cellulose fibres remain on the surface resulting in the silver-grey colour of weathered wood (Kropat *et al*., 2020). No blue colouration was observed in oak and sweet chestnut wood. The presence of extractives seems to limit the growth of blue stain fungi on the wood surface (Vek *et al*., 2019).

The density of the wood corresponded to the information found in literature. The average density of

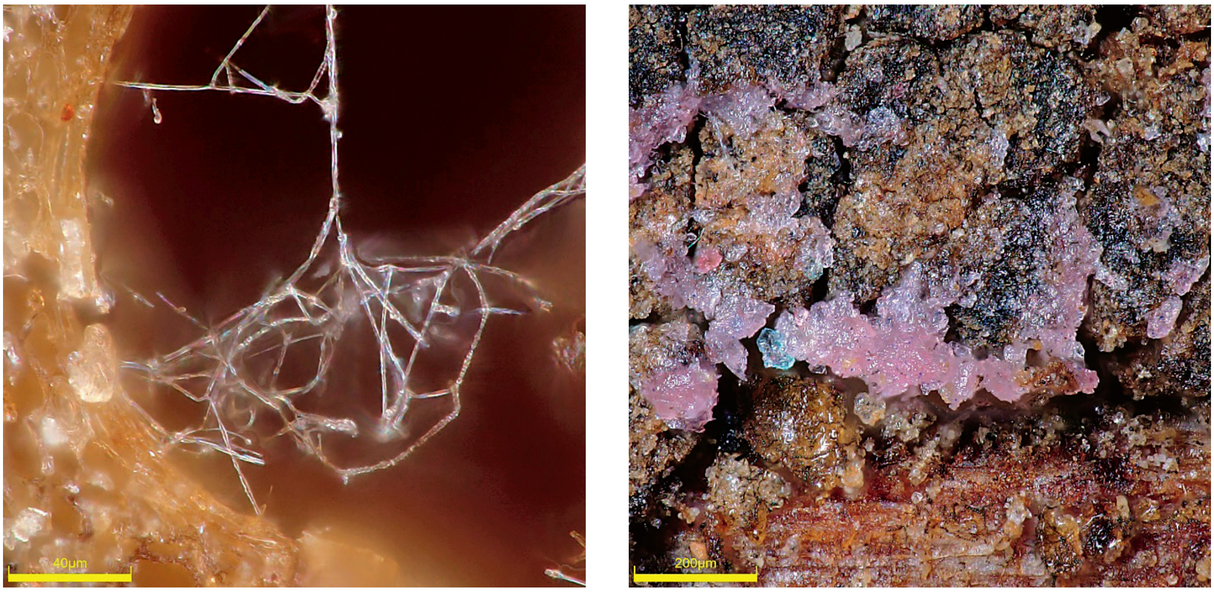


Figure 5 Secondary, clamp fungal mycelium in oak wood vessel (A) and slime fungus growing on wood surface (B)
Slika 5. Sekundarni micelij stezaljke u traheidi hrastovine (A) i sluzava gljiva koja raste na površini drva (B)

Figure 6 Presence of loose cellulose fibres on weathered wood surface
Slika 6. Gubitak celuloznih vlakana na površini drva izloženoga vremenskim utjecajima

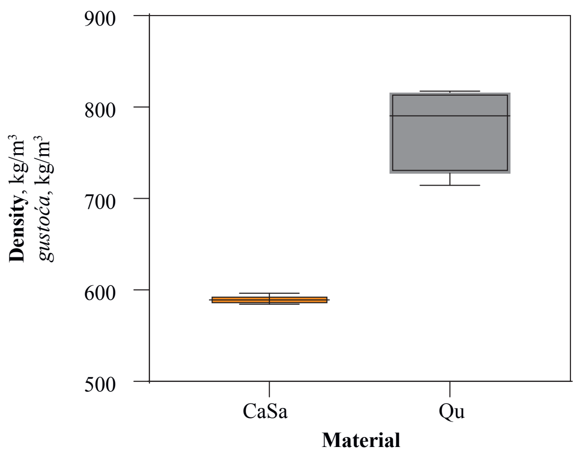


Figure 7 Density of mill sweet chestnut (CaSa) and oak (Qu) wood determined in laboratory (n = 5)
Slika 7. Laboratorijski određena gustoća drva pitomog kestena (CaSa) i drva hrasta (Qu) od kojih je mlin izgrađen (n = 5)

oak logs was 778 kg/m^3, while the density of sweet chestnut was slightly lower (590 kg/m^3). These values are in agreement with the literature data for both species. The literature shows that the density of oak varies between 390 kg/m^3 and 930 kg/m^3 (Wagenführ, 2014). A high density indicates that the wood has not been degraded. Furthermore, it is generally assumed that density has a positive influence on durability within the same ring-porous species. Denser wood is more durable than wood with lower density (Brischke *et al.*, 2013). The density determined in the laboratory agrees with the density determined during screw withdrawal (787 kg/m^3). This evaluation proves that only high-quality material was used for the respective construction. The high quality of the wood is reflected in the dynamic modulus of elasticity (*dMoE*). The dynamic modulus of elasticity was only determined on oak logs. The average *dMoE* of oak logs was 14.0 GPa. This is consistent with literature data for the static *MoE* of oak logs, which range from 10.0 GPa to 13.2 GPa. As mentioned earlier, the *dMoE* is always higher than the static *MoE* (Chauhan and Sethy, 2016). This is evidence of good structural integrity of the logs in the studied work.

The analysis of the wood with the resistograph confirmed previous studies. Twenty-six resistograph measurements were taken. Signs of rot were only found

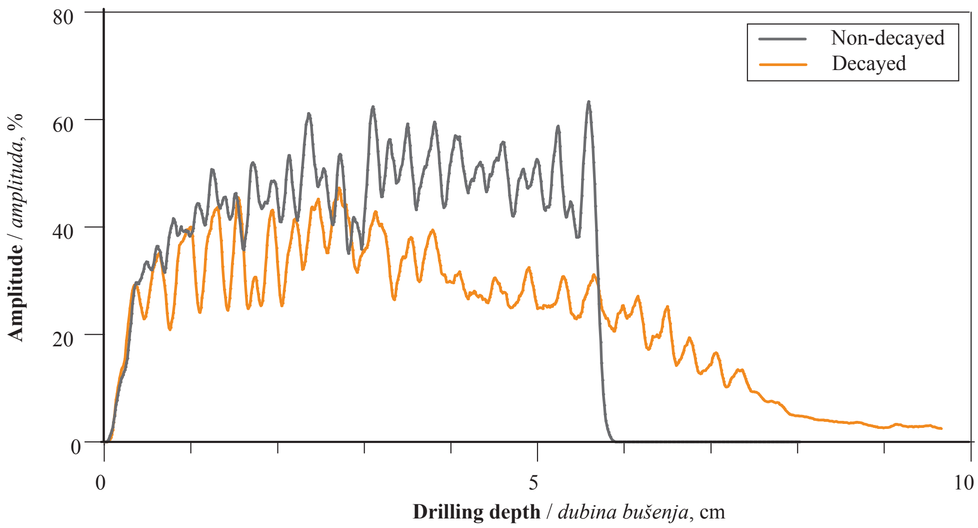


Figure 8 Resistograph profiles of non-decayed and decayed beam in the analysed mill
Slika 8. Profili rezistografa neraspadnute i raspadnute grede u analiziranome mlinu

at one point in the roof structure. This is consistent with the measurements of *MC* and is probably a result of roof leakage. The logs were not decayed. Only the outer surface was partially softened, probably due to weathering (Figure 8). The outer part of the logs was partially degraded due to the presence of the less durable sapwood, which was partially degraded by beetles of the genus *Anobium* (Figure 9). *Anobium* beetles are common in old constructions of wet hardwood (Unger *et al*., 2001).

Insect galleries were mainly found on the outside of the structure. The interior was at least partially protected by smoke deposits. The chimney without a flue was located inside the mill. The smoke could therefore easily spread over the entire volume of the building and escape through the opening in the roof. It should be remembered that the mills also operated at night, so the fire was the source of light and heat. Rahmat and coworkers (2020) reported that condensed gases from wood burning have fungicidal effect. As can be seen from the XRF spectra of the corresponding deposits, they contain high concentrations of iron (Fe), sulphur (S), chlorine (Cl), cadmium (Cd) and manganese (Mn). These elements are assumed to have originated from the corrosion of metal roof and smoke (Humar, 2010). However, it should be noted that these deposits were present only on the interior side of the beams.

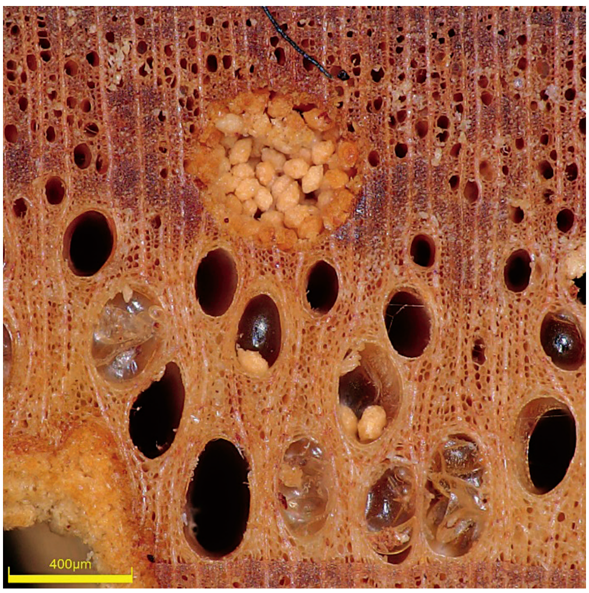


Figure 9 Insect hole in sweet chestnut wood filled with debris typical for beetles from genus *Anobium*
Slika 9. Rupe od kukaca u drvu pitomog kestena ispunjene ostatcima tipičnima za kornjaše iz roda *Anobium*

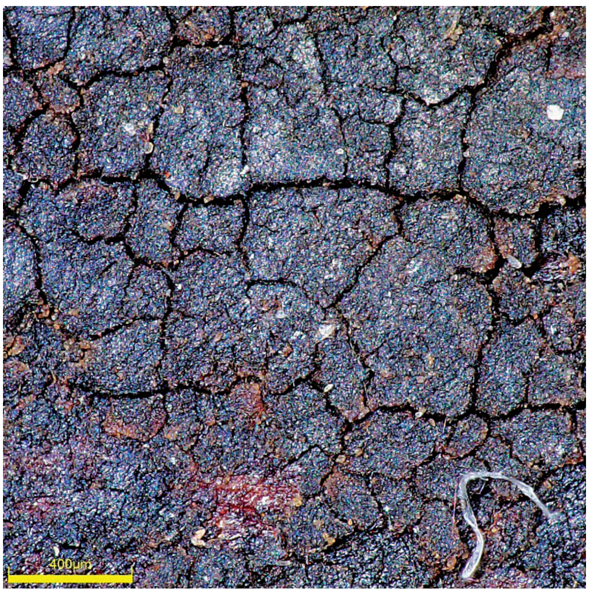


Figure 10 Deposits on oak beams surface in mill interior
Slika 10. Naslage na površini hrastovih greda u unutrašnjosti mlina

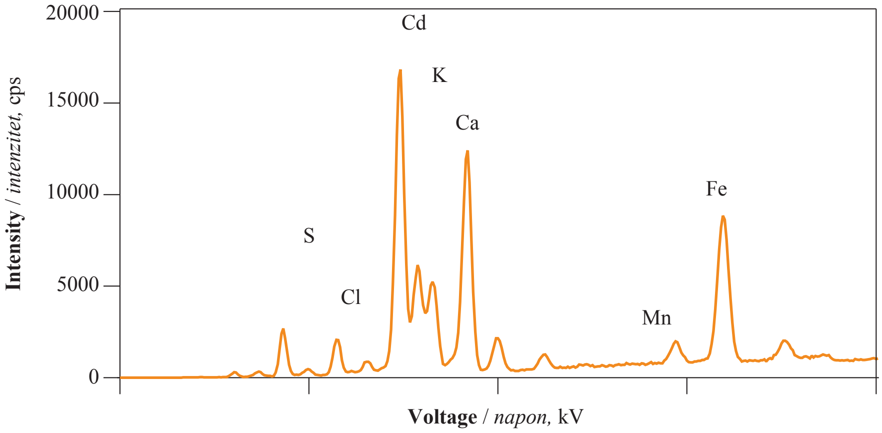


Figure 11 Representative XRF spectra of wood from mill interior
Slika 11. Reprezentativni XRF spektri drva iz unutrašnjosti mlina

4 CONCLUSIONS
4. ZAKLJUČAK

The oak and sweet chestnut wood in the mill examined is in quite good condition, as revealed by microscopic and mechanical analysis. The decay is limited to the sapwood and some other exposed elements. The main reason for the good condition of the respective building is the use of durable wood species and good construction. Respective mills offer a great opportunity to monitor the performance of wood in a realistic environment. To protect the respective mills for future generations, the roof should be maintained regularly to avoid possible leaks. The respective mill confirms that wooden structures can last for decades if proper wood is selected for the application, and protection by design measures is fully considered.

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Comparative Study on Dimensional Stability and Biological Durability of Poplar Wood Modified by Chemical and Heat Treatment

Usporedna studija dimenzijske stabilnosti i biološke trajnosti kemijski i toplinski modificirane topolovine

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ABSTRACT • *The fast-growing species of Populus deltoides is of significant commercial importance to the Indian wood merchants. Despite its abundant availability and better treatability, the use of P. deltoides is limited due to its perishable nature. Heat treated and chemically modified Populus deltoides L. was thus explored for dimensional stability and biological durability to commercialise its use. Chemical modification was performed using a combination of citric acid and sodium hypophosphite. Heat treatment was done in a laboratory oven in air at 140 °C for 8h. Both chemically modified and heat-treated samples exhibited improvements in dimensional stability and biological durability in comparison to the untreated control sets. Chemical modification resulted in better dimensional stability. Anti-swelling efficiency of Populus deltoides was 2-2.5 times more for the chemically modified set compared to heat treatment in both radial and tangential directions. Chemically modified set became significantly more resistant to biodeterioration showing 5 times less mass loss by termites, whereas controls and heat-treated sets returned loss percentages which were comparable. Soil block bioassay with Trametes versicolor (TV) exhibited mass loss of 9.46 % for chemically modified samples, 40.26 % for heat treated samples and 50.02 % for the untreated controls, respectively. Rhodonia placenta (OP) followed a similar trend with mass loss of chemically modified set being 5.72 % and heat-treated set being 37 %, respectively, with the controls showing 43.85 % mass loss. Mass loss exhibited by the heat-treated samples were less than the controls, but the values suggested that heat treatment at 140 °C for 8 h failed to impart any substantial resistance against rotting fungi and termites.*

KEYWORDS: *biological durability; chemical modification; dimensional stability; heat treatment*

SAŽETAK • *Brzorastuća vrsta Populus deltoides ima veliku komercijalnu važnost za indijske trgovce drvom. Unatoč velikoj dostupnosti i dobroj obradivosti, upotreba drva P. deltoides ograničena je zbog njegove slabe trajnosti. Stoga je radi bolje komercijalizacije drva Populus deltoides L. istražen utjecaj toplinske i kemijske modifikacije na njegovu dimenzijsku stabilnost i biološku trajnost. Kemijska modifikacija provedena je kombinacijom*

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limunske kiseline i natrijeva hipofosfita. Toplinska modifikacija u trajanju od osam sati obavljena je u laboratorijskom sušioniku pri 140 °C, uz prisutnost zraka. Kemijski i toplinski modificirani uzorci drva pokazali su bolju dimenzijsku stabilnost i biološku trajnost od nemodificiranih kontrolnih uzoraka. Kemijska modifikacija rezultirala je i većom dimenzijskom stabilnošću. Učinkovitost u sprečavanju bubrenja drva Populus deltoides bila je 2 – 2,5 puta veća u kemijski modificiranih uzoraka nego u toplinski modificiranih uzoraka, i to u radijalnome i tangencijalnom smjeru. Kemijski modificirani uzorci drva postali su znatno otporniji na biološko propadanje i pokazali su pet puta manji gubitak mase pri djelovanju termita nego toplinski modificirani i nemodificirani uzorci. Test s Trametes versicolor (TV) pokazao je gubitak mase od 9,46 % za kemijski modificirane uzorke, 40,26 % za toplinski modificirane uzorke i 50,02 % za nemodificirane uzorke. Za test s Rhodonia placenta (OP) uočen je sličan trend gubitka mase, koji je za kemijski modificirane uzorke iznosio 5,72 %, za toplinski modificirane uzorke 37 %, a za nemodificirane uzorke 43,85 %. Gubitak mase toplinski modificiranih uzoraka drva bio je manji od gubitka mase nemodificiranih uzoraka, ali su vrijednosti pokazale da se toplinskom modifikacijom pri 140 °C tijekom osam sati nije uspjela postići značajna otpornost protiv gljiva truležnica i termita.

KLJUČNE RIJEČI: *biološka trajnost; kemijska modifikacija; dimenzijska stabilnost; toplinska modifikacija*

1 INTRODUCTION
1. UVOD

The use of wood is abundant in modern life and is often promoted due to its natural origin, aesthetic appeal, and ability to sequester carbon in service. Despite being preferred over other construction materials, the use of wood is often limited due to its inherent properties like bio deterioration, poor dimensional stability (DS) and fire susceptibility, which are highly undesirable in service (Hom *et al*., 2020a). Treating wood with several chemicals can make its performance more acceptable and enhance its durability. However, these may contain some biocides often associated with environmental concerns and health hazards. With the level of environmental pollution from other sources reaching extremes, restrictions on the use of such chemicals have been proposed by the environmentalists. Many countries have already banned the use of formulations containing chromium, arsenic and pentachlorophenol, which has resulted in intensified research on more eco-friendly practices (Hill 2006; Ganguly, 2018; Ganguly *et al*., 2020)

Wood modification is a new approach to overcome one or more such disadvantages associated with wood by means of altering the physical and chemical properties of the material to enhance its performance and durability in service (Rowell, 2006; Sandberg *et al*., 2017; Samani *et al*., 2019; 2020; Ganguly *et al*., 2021). Chemical modification (CM) of wood offers a viable good alternative to conventional wood preservation. Chemically modified wood is expected to be more dimensionally stable, more durable against blue stain and rot fungi, and more resistant to UV radiation than unmodified wood (Hom *et al*., 2020b). Chemical modification of wood is an efficient way to improve dimensional stability (DS), Biological Durability (BD) and UV resistance, which is particularly interesting in service. This has been established through various studies using different chemicals, and their negative impacts on wood and the environment were also discussed at length (Singh *et al*., 1979; Rowell, 1982; Kumar and Agarwal, 1983; Kumar and Kohli, 1985; Matsuda, 1987; Kumar *et al*., 1991; Donath *et al*., 2004; Hansmann *et al*., 2005; Hom *et al*., 2020b). Acetic Anhydride was found to be the most suitable reagent for chemical wood modification through acetylation (Singh *et al*., 1992; Mantanis, 2017), although most of the researchers reported a net loss of more than 50 % of the reagent due to formation of acetic acid, which may affect the strength of wood and increase corrosiveness to metal fittings. Chemical modification by DMDHEU (1, 3-dimethylol-4, 5-dihydroxyethyleneurea) had reportedly increased dimensional stability of wood up to 50-60 % (Despot *et al*., 2008) and increased durability of modified wood (Videlov, 1989; Ashaari *et al*., 1990; Militz, 1993; Yusuf *et al*., 1994), although formaldehyde release is an associated problem (Katović and Soljačić, 1988). Heat Treatment (HT) is also known to impart dimensional stability to wood but varies from species to species (Militz, 2002) and is different in different directions. The dimensional stability, thus induced, is primarily due to the reduced hygroscopicity and subsequent decreased Equilibrium Moisture Content (EMC) of heat treated wood (Esteves and Pereira, 2009). Tjeerdsma *et al*. (1998) mentioned a possibility of the loss of methyl radicals of some guiacylic and syringylic units of lignin leading to the increase of phenolic groups and proportion of free ortho positions, which results in the increased dimensional stability of heat-treated wood.

Inhibiting wood affinity towards water should be the fundamental step in protecting it against rapid decay and subsequent distortion. This is highlighted in the research on wood modification, which sheds light on non-biocidal modes of action, such as water exclusion efficacy (Winandy and Morrell, 2017), anti-swelling efficiency (ASE) and dimensional stability (Hom *et al*., 2020b). CM is known to improve decay resistance of wood (Hill *et al*., 2005; Hill, 2006; Rowell *et al*., 2008). The use of citric acid (CA) for wood preservation and chemical modification is well known and can be traced back to the first part of the last decade. Wood modified by CA and cured by temperature or by microwaves showed improved dimensional stability. As a raw material, CA is easily affordable and adheres to strict ecological and environmental requirements. Water is used as a solvent during CM of wood with CA, which also makes it a cost-effective alternative to wood preservatives. CA modified wood was reported for its enhanced BD (Hasan *et al*., 2006, 2007; Despot *et al*., 2008; Šefc *et al*., 2009; Treu *et al*., 2020). Heat treatment of wood at higher temperatures has also been reported to become significantly durable by Dirol and Guyonnet (1993). Kim *et al*. (1998) further concluded that heat treatment at higher temperatures and longer duration imparts more resistance to fungal decay than it does at lower temperatures and exposure durations.

The fast-growing species of *Populus deltoides* (PD) is of significant commercial importance to the Indian wood merchants due to its rapid use for various purposes and is an integral part of the agroforestry system in the country. Previous studies of this species ascertained that the wood of poplar is of non-durable nature and is susceptible to the attack of *Trametes versicolor* (Casado-Sanz *et al*., 2019).

Keeping in mind the above cited literatures, the present study was conducted on highly perishable poplar wood in order to assess and evaluate the impact of chemical modification and heat treatment on its dimensional stability and biological durability, which may give an indication to its performance in service in both indoor and outdoor conditions. The primary aim of this study was to commercialise the use of poplar, a secondary timber species in Indian wood sector by making it more durable and less prone to damage due to moisture induced movements in wood.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Sample preparation and treatments
2.1. Priprema uzoraka i tretmani

Logs of *Populus deltoids* of 183 cm in length were procured from the Forest Range office of Forest Research Institute, Dehradun, India. Logs were converted into planks of full length. The dimensions of the planks were 182 cm × 25.4 cm × 9 cm. Samples of dimensions of 50 mm × 50 mm × 50 mm for dimensional stability test and 19 mm × 19 mm × 19 mm for soil block assay were prepared from seasoned planks. For termite mound test (TeMoT), the sample size was maintained as 100 mm × 25 mm × 6 mm. Defect free samples were chosen for the study and samples were prepared parallelly from the same part of the board. In total 3 sets of samples were prepared, with each set having 6 replicates for DS, SBA and TeMoT (Table 1). The first set (T1) was treated with CA (6.9 %) and SHP (6.5 %). The samples were treated by giving an initial vacuum of 30 mm of Hg (4kPa) for 10 min, and then the vessel was filled with the treating solution. Samples were maintained under the same vacuum for 3 h. Vacuum was released after 3h and samples were allowed to remain soaked in the solution for 18 h under atmospheric pressure (Despot *et al*., 2008). After completion of impregnation cycle, samples were removed from treating solution, air dried for 48h at room condition and kept in the laboratory oven at 140 °C for 8 h. The second set (T2) was kept in the oven at 140 °C for 8 h to see the effect of heat treatment. This was done to explore a cost-effective potential solution to an otherwise cumbersome method of heat treatment. The third set (T3) was made of control samples without any treatment (Table 1).

2.2 Weight percent gain (*WPG*)
2.2. Povećanje mase (*WPG*)

The weight percent gain (*WPG*) of the specimens after chemical modification and heat treatment (Loss) was calculated by:

$$WPG = (W_t - W_o) / W_o \times 100 \quad (1)$$

Where, W_o and W_t are oven dried (OD) weights of unmodified and chemically modified and heat-treated samples, respectively.

2.3 Water immersion
2.3. Uranjanje u vodu

2.3.1 Dimensional stability (DS)
2.3.1. Dimenzijska stabilnost (DS)

DS was determined by comparing the total volumetric swelling (*VS*) of treated and control samples. The

Table 1 Chemical treatments and sample distribution
Tablica 1. Kemijski tretmani i distribucija uzoraka

S. No.	Treatment *Tretman*	Chemical reagents *Kemijski reagensi*	Species: PD / *Vrste: PD*						Total *Ukupno*
			DS	SBA	TMT	RS	TS	VS	
1	T1 (CA 6.9+SHP 6.5)	Citric acid (6.9 %) and sodium hypophosphite (6.5 %) *limunska kiselina (6,9 %) i natrijev hipofosfit (6,5 %)*	6	6	6	6	6	6	36
2	T2 (Heat)	Heat treatment 140 °C for 8 h *toplinska modifikacija pri 140 °C tijekom 8 h*	6	6	6	6	6	6	36
3	T3 (Control)	Control / *kontrolni uzorak*	6	6	6	6	6	6	36
Total / *ukupno*			18	18	18	18	18	18	108

R – Radial, T – Tangential, V – Volumetric, S – Swelling coefficient (e.g., RS – radial swelling coefficient)
R – radijalno, T – tangencijalno, V – volumetrijski, S – koeficijent bubrenja (npr. RS – radijalni koeficijent bubrenja)

samples of all sets were oven dried at (103±2) °C for 24 h, and oven dried weight and initial dimensions were recorded. Swelling was determined by a water immersion test. All samples were submerged in distilled water and kept under vacuum (30 mm Hg/ 4 kPa) for 30 minutes and allowed to soak for 24 h under atmospheric pressure (Šefc *et al*., 2009). Weight and dimensions of saturated samples were recorded. Swelling coefficient (S) was calculated using the following Eq.

$$S\ (\%) = (V_s - V_o)/V_o \times 100 \quad (2)$$

Where, V_s is the volume of soaked sample and V_o is the volume of oven dried sample.

$$ASE\ (\%) = (S_u - S_m) / S_u \times 100 \quad (3)$$

Where, S_u and S_m are swelling coefficients of unmodified and modified wood samples, respectively.

2.3.2 Water exclusion efficiency (*WEE* %)
2.3.2. Učinkovitost odbojnosti drva prema vodi (*WEE* %)

WEE was calculated using the following method:

$$WEE\ (\%) = (W_c - W_t)/ W_c \times 100 \quad (4)$$

Where, W_c is water absorbed by untreated controls in g and W_t is water absorbed by treated samples in g.

2.4 Soil block bioassay (SBA)
2.4. Ispitivanje biološke trajnosti (SBA)

2.4.1 Test fungi and specimens
2.4.1. Ispitne gljive i uzorci

One brown rot, *Rhodonia placenta* (Fr.) Niemelä, K.H. Larss. & Schigel (OP) (FRI Culture No. 180) and one white rot, *Trametes versicolor* (L.) Lloyd (TV) (FRI Culture No. 651) fungi were selected for the SBA study (IS 4873:2008). Fresh cultures of the fungi were obtained from the collection of Forest Pathology Discipline of Forest Research Institute, India for research purposes. The test blocks of size 19 mm ×19 mm ×19 mm and the feeder blocks of size 4 mm ×19 mm ×35 mm were prepared along the length of grain. The feeder blocks were prepared from sapwood of *Bombax ceiba*. The test blocks were oven dried at (103±2) °C for 24 h and then conditioned at 75 % *RH* till constant weights (W_1) were achieved. W_1 was considered as the weight before incubation.

2.4.2 Preparation of soil culture bottles
2.4.2. Priprema posuda s hranjivom podlogom

Sieved, air-dried garden soil (125 g), having pH 5.0-7.0, was filled (compacted by tapping) in screw capped bottles. Distilled water (44 ml) was added to the bottles to obtain 130 % of water holding capacity of soil in test bottles. Two feeder blocks of size 4 mm × 19 mm × 35 mm were placed directly on the surface of the soil. The prepared bottles with caps loosened were sterilized in an autoclave at a pressure of 98066.5 Pa for 30 min prior incubation.

2.4.3 Preparation of test culture
2.4.3. Priprema ispitne kulture

Sterilized culture bottles were cooled and the fungus inoculum from freshly grown culture, approximately 8-10 mm in diameter, was placed on the edge of the feeder blocks in culture bottles. The inoculated bottles were incubated in B.O.D. (Biochemical oxygen demand) with slightly loosened lids at (25±2) °C and (70±4) % relative humidity (*RH*) for approximately 3 weeks, till the feeder blocks were completely covered by the test fungi.

2.4.4 Incubation of test blocks in culture bottles
2.4.4. Inkubacija ispitnih blokova u posudama s kulturama

Two blocks were placed on feeder blocks in contact with mycelium along the cross-section face in each culture bottle. The bottles containing the test blocks were incubated for a period of 12 weeks in the incubator maintained at (25±2) °C and a *RH* of about (70±4) %. At the end of the incubation period the blocks were removed from the culture bottles, cleaned off from the adhering mycelium with a soft tissue and dried at room temperature for 3-4 days. Subsequently the blocks were again dried in a hot air oven at (103±2) °C for 24 h and then conditioned at 75 % *RH* till the constant weights (W_2) were obtained.

2.5 Calculation of mass loss (*ML*)
2.5. Izračun gubitka mase (*ML*)

ML (%) was calculated from the conditioned weight of the blocks before and after SBA.

$$ML\ (\%) = (W_1 - W_2)/W_1 \times 100 \quad (5)$$

Where, W_1 is the conditioned weight of the blocks before test, and W_2 is the conditioned weight of the blocks after test.

2.6 Termite mound test (TeMoT)
2.6. Ispitivanje otpornosti drva na djelovanje termita (TeMoT)

TeMoT was conducted as per Shukla (1977). CM, HT and control blocks were buried at different places inside a termite mound of *Odontotermes obesus* (rambur) at the beginning of the month of May. Blocks were removed from the mound in November when the activity of termites almost ceased due to fall in temperature. The blocks were examined for termite attack and reinstalled in next May to have exposure to termites for two successive termite seasons. Blocks were cleaned of mud and debris and evaluated visually to ascertain and quantify damage by termites. Efficacy of treatments in terms of protection against termites was evaluated by *ML* % (wood consumed) as reported by previous researchers (Shukla, 1977; Kumar and Dev, 1993) (Table 2).

Table 2 Classification of wood into various resistance classes after TeMoT
Tablica 2. Klasifikacija drva u različite klase otpornosti prema TeMoT-u

***ML*, %**	**Termite resistance class** *Klasa otpornosti na termite*
0-6	Very resistant (Class I) / *vrlo otporno (klasa I)*
7-16	Resistant (Class II) / *otporno (klasa II)*
17-30	Moderately resistant (Class III) *srednje otporno (klasa III)*
31-50	Poorly resistant (Class IV±) *slabo otporno (klasa IV+)*
51 and above	Perishable (Class IV-) / *neotporno (klasa IV-)*

2.7 Statistical analysis
2.7. Statistička analiza

Statistical analysis was done using SPSS (Version 16) software package (IBM, Chicago, USA). The mean, standard deviation and standard error of mean (SE) were calculated, and ANOVA was performed. Duncan Analysis was performed further to compare the means statistically at 95 % significance level.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

The results of the overall study are presented in Table 3 and 4 and Figures 1, 2 and 3. Chemical modification (CM) showed better dimensional stability and biological durability over heat-treated (HT) and control samples. *WPG* of chemically modified samples were satisfactory and comparable with previous studies and provided enough resistance to fungi and termites (Larnøy *et al.*, 2018) The treatment methods chosen might have added to the modification as CA cured better when it was followed by curing at high temperatures (Larnøy *et al.*, 2018). For HT samples, a cheap alternative was explored by trying to modify the samples in oven at a relatively lower temperature. Heat treatment (HT) by this method was fairly successful and HT sets performed marginally better, but the performance was not up to the mark and further research should be carried out.

3.1 Dimensional stability (*DS*)
3.1. Dimenzijska stabilnost (*DS*)

A 12 % increase in weight observed by CM was due to substitution of OH groups of the cell wall by citric acid cross linking (Lee *et al.*, 2020), while HT causes a loss in weight. Similar weight loss (1.5 % at 180 °C for 4 h) was observed in previous studies (Alén *et al.*, 2002). Stamm, (1956) showed that wood heated in the presence of oxygen causes degradation of wood more rapidly than wood heated in an oxygen-free atmosphere, which was also reported by Mitchell (1988). Weight loss occurs to a higher extent in hardwoods as compared to softwoods (MacLean, 1951, 1953; Hills, 1975; Hill *et al.*, 2021). Mass loss was 2-3 % higher in hardwoods than softwoods treated under identical conditions (Zaman *et al.*, 2000).

The *WEE* observed for CM was higher as compared to HT. Citric acid modification resulted in 24 % while HT caused 16 % *WEE*. Previous studies have stated that the molecular volume of the substituted group along with a degree of substitution of the hydroxyl groups influences *WEE* of modified wood (Chang and Chang, 2002). The data of *WEE* for CM was similar to that observed by Rowell and Banks, (1985) of 20 % and 25 % for Acetic Anhydride and Butylene Oxide.

From Table 3 and Figure 1, it can be clearly deduced that both CM and HT have significantly reduced the swelling of PD wood as compared to control. It was observed that the swelling coefficient was lowest for CM (CA 6.9+SHP 6.5) as compared to T2 or T3 for radial, tangential and volumetric samples. The swell-

Table 3 Mean of weight percent gain/loss due to treatment and swelling in water immersion test of PD (Standard Error (*SE*) Values are in parenthesis)
Tablica 3. Srednja vrijednost povećanja/gubitka mase zbog tretmana i rezultati testa bubrenja uranjanjem uzoraka drva *Populus deltoides* u vodu (vrijednosti standardne pogreške *SE* navedene su u zagradama)

Treatment *Tretman*	WPG	**Radial swelling coefficient** *Koeficijent radijalnog bubrenja*	**Tangential swelling coefficient** *Koeficijent tangentnog bubrenja*	**Volumetric swelling coefficient** *Koeficijent volumnog bubrenja*	***WEE*, %**
T1 (CA 6.9+SHP 6.5)	11.76 (±0.82)	2.11 (±0.07)a	4.50 (±0.07)m	7.13 (±0.40)x	24.34 (±1.17)p
T2 (Heat)	-0.31 (±0.16)	3.25 (±0.07)b	6.23 (±0.15)n	10.20 (±0.61)y	16.12 (±0.79)q
T3 (Control)	-	4.18 (±0.15)c	7.35 (±0.11)o	13.36 (±0.45)z	-

Note: Different alphabets denote different homogeneous groups as per Duncan Analysis. Duncan Analyses were performed separately for all above parameters and reported accordingly. a, b, c denote different groups for radial swelling; m, n, o denote different groups for tangential swelling and x, y, z denote different groups for volumetric swelling, p and q represent the same for *WEE*.
Napomena: Različita slova označavaju različite homogene skupine prema Duncanovoj analizi, koja je provedena za svaki parametar naveden u tablici zasebno, a rezultati su prikazani u skladu s tim. Pritom a, b, c označavaju različite skupine za radijalno bubrenje; m, n, o obilježavaju različite skupine za tangentno bubrenje; x, y, z predočuju različite skupine za volumno bubrenje, a slova p i q predstavljaju isto to za WEE.

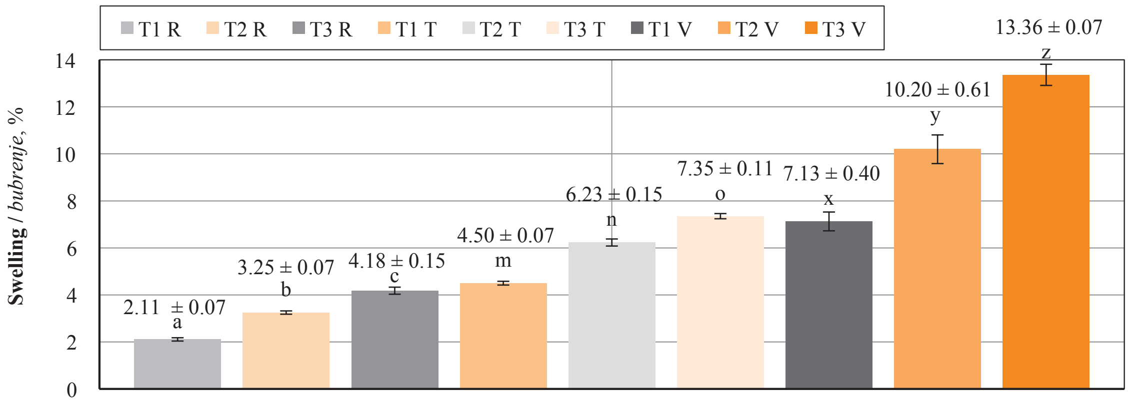

Figure 1 Swelling coefficient (*S*) after different treatments in radial (R) and tangential (T) direction and volumetric swelling coefficient (*V*) (T1 – chemical modification, T2 – heat treatment, T3 – control)
Slika 1. Koeficijent bubrenja (*S*) nakon različitih tretmana u radijalnome (R) i tangencijalnom (T) smjeru te koeficijent volumnog bubrenja (*V*) (T1 – kemijska modifikacija, T2 – toplinska modifikacija, T3 – kontrola)

Table 4 Anti swelling efficiency (*ASE*) of modified PD wood (*SE* values in parenthesis)
Tablica 4. Učinkovitost smanjenja bubrenja (*ASE*) modificiranog drva *Populus deltoides* (*SE* vrijednosti su u zagradi)

Treatment / *Tretman*	***ASE* radial**	***ASE* tangential**	***ASE* volumetric**
T1 (CA 6.9+SHP 6.5)	49.44 (±0.84)	38.79 (±1.02)	45.10 (±0.63)
T2 (Heat)	22.16 (±0.76)	15.26 (±0.95)	20.23 (±1.4)

ing for non-modified samples ranged between 4.18-13.36, while the *S* observed for T1 was between 2.11-7.12, which was significantly lower than T2 having *S* ranging between 3.25-10.20. The *S* obtained from T2 treatment was similar to that obtained by CM of radiata pine using acetic anhydride by combination of 689 kPa pressure for 30 mins followed by 137 kPa pressure and curing at 120 °C for 30 mins (Hom *et al*., 2016). Heat treated wood is hydrophobic in nature and hence reduces shrinkage and swelling due to decrease in the hydroxyl groups and decomposition of a large portion of the hemicelluloses in the wood cell wall (Boonstra, 2008; Korkut and Kocaefe, 2009).

The *ASE,* calculated for CM from *S*, was observed to be higher as compared to HT for all the samples. Radial samples showed highest *ASE* followed by volumetric and minimum for tangential samples. The *ASE* was between 39-49 % for CM, while HT resulted in 15-22 %. The *ASE* for CM was similar to that of *Fagus sylvatica* when modified with 7.0 % water solution of CA with 6.5 % SHP as a catalyst when cured at 140 °C causing *ASE* of 43 % (unleached) and 30 % (leached) (Šefc *et al.,* 2009). Similarly, the *ASE* observed by CM using CA is comparable to the values reported by previous researchers with maleic anhydride with *ASE* of 35-45 % (Essoua Essoua *et al*; 2016).

3.2 Soil block bioassay (SBA)
3.2. Ispitivanje biološke trajnosti (SBA)

Figure 2 shows that *ML* reduced significantly after both CM and HT, with CM returning excellent biological durability against both brown rot and white rot

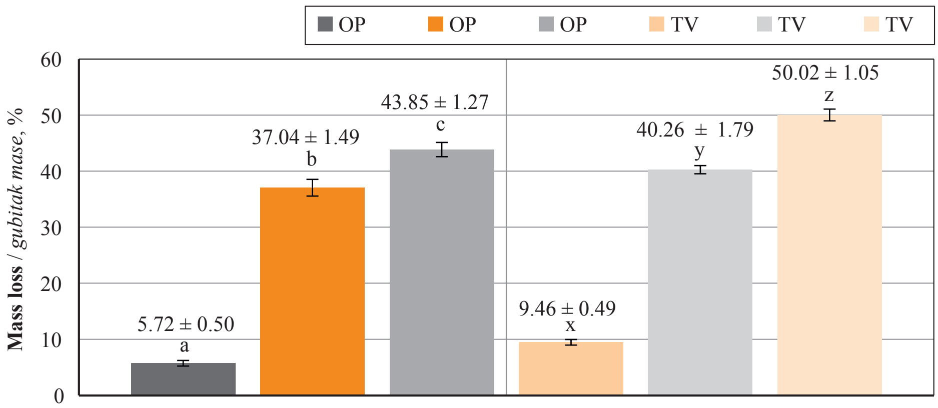

Figure 2 Mean mass loss (*ML*) percentage in PD after SBA against OP and TV (*SE* values are mentioned after ± and different alphabets (a, b, c for OP and x, y, z for TV) denote different homogeneous subsets as per Duncan analysis)
Slika 2. Srednji postotni gubitak mase (*ML*) uzoraka drva *Populus deltoides* nakon SBA s obzirom na OP i TV; vrijednosti *SE* prikazane su nakon znaka ±, a različita slova (a, b, c za OP i x, y, z za TV) označavaju različite homogene podskupove prema Duncanovoj analizi

fungi. Several authors reported higher rot resistance of HT wood when not in direct contact with ground (Esteves and Pereira, 2009). After HT, several wood polymers, like hemicellulose, degrade and release acetic acid, which may result in some chemical modification of wood compounds (Esteves and Pereira, 2009; Hill *et al*., 2021). The primary reason for increased durability against rotting fungi was reported to be the formation of several molecules after HT, like furfural, which reticulates with lignin in wood and becomes unrecognisable by the decaying fungi (Weiland and Guyonnet, 2003). Additionally, reduced *EMC* after HT and lower FSP also adds to protection of wood against decay. In the present study, HT reduced biodeterioration but not to a greater extent. A mere 7 % reduction in *ML* % against brown rot OP was observed in the HT sets with respect to the untreated control sets. For white rot, HT set performed slightly better as it resulted in *ML* % of 40.26 %, which was about 10 % less than *ML* observed in the control set (50.02 %). These findings are in conformity with the results of previous studies, where 35 % to 65 % rot resistance was reported for heat treated wood at different temperatures and time durations (Kim *et al.,* 1998), and the researchers highlighted that BD increased with increasing temperature and treatment time after HT. It can be said that, for highly perishable species like PD, HT at such low temperature level might not provide the required resistance to wood, and that wood may remain somewhat susceptible to fungal attack if no additional treatment is applied. CM however showed remarkable improvement in BD against both white and brown rot fungi and exhibited 6-8 times less reduction in mass against untreated samples exposed to TV and OP, respectively. This enhanced durability can be attributed to the cross linking of CA on -OH groups of cell wall components (Larnøy *et al*., 2018) of PD wood, which made it quite unrecognisable as a food source to the decaying fungi. Another possible reason could be the leaching of treating solution in the culture jars with higher *MC,* which might have reduced the virulence of test fungi with time.

3.3 Termite mound test (TeMoT)
3.3. Ispitivanje otpornosti drva na termite (TeMoT)

From Figure 3 it can be inferred that CM using CA and SHP has imparted a very high level of biological resistance to an otherwise perishable poplar (PD) wood. After two successive termite seasons, the control samples were almost destroyed, which is evident as their *ML* was 69.27 %. The biological durability (BD) is often attributed to the presence of toxic chemicals in wood, but the *ML* observed in case of PD in the present study indicates that PD is poor in terms of inherent natural durability, which is obvious as it has a very limited range of use in the Indian woodworking sector. Susceptibility of several Poplar species to biodeterioration, especially fungi, was previously reported (Karimi *et al*., 2013; Casado-Sanz *et al*., 2019). The chemically modified set of PD exhibited 6 times less ML and elevated the perishable PD species, which normally belonged to class IV, to class II and established it as resistant to termite decay. Decay resistance by CA treated wood were also reported before (Lee *et al.*, 2020; Treu *et al.*, 2020). Treu *et al.* (2020) highlighted in their study that the primary reason for such results was the potential leaching of the chemicals (CA+Sorbitol) due to changes in wood-water relations after modification, along with higher mortality rate of the termites. Another possible explanation of such enhanced BD against termites can be the decreased level of protozoa in certain termite species after being exposed to acetylated wood, which leads to starvation after one or two weeks as highlighted by Duarte *et al.* (2017). BD of the HT sets of PD was comparable with the control set as the HT set exhibited a ML of 65.46 %, which was marginally less than the control set. Esteves and Pereira (2009) highlighted the possibilities of

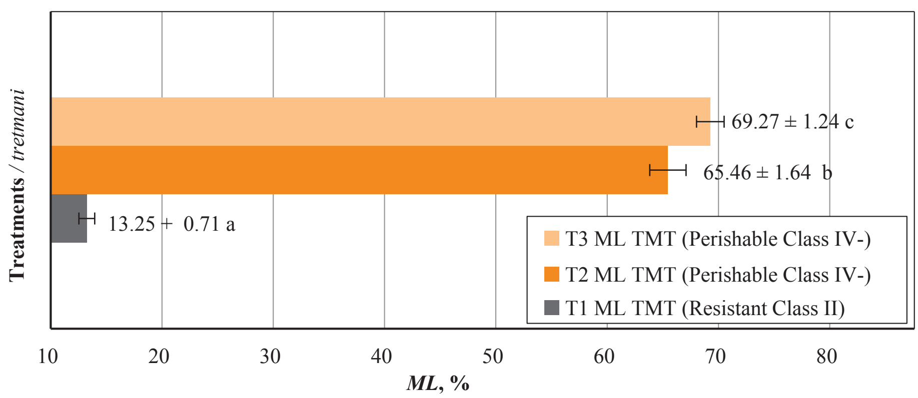

Figure 3 *ML* % and corresponding durability class for different treatments with TeMoT (*SE* values are mentioned after ± and different alphabets denote different homogeneous subsets)
Slika 3. Postotak *ML*-a i odgovarajuća klasa trajnosti za različite tretmane s TeMoT-om (vrijednosti *SE* prikazane su nakon znaka ±, a različita slova označavaju različite homogene podskupove)

an esterification of cellulose after degradation of hemicelluloses as the reason for enhanced biological durability of heat-treated wood, which produces substances that are not recognisable as food materials to decaying agents. Additionally, the *FSP* of HT wood is lower than untreated wood, which is a desirable property to impart better resistance against biological degradation. Further change of hemicelluloses from hydrophilic and easily digestible to hydrophobic molecules may also play some part (Esteves and Pereira, 2009). From the study, it can be concluded that HT at such lower modification temperature is not enough to ascertain sufficient BD to PD. The findings are in line with previous work by Nunes *et al.* (2004), where the researchers also reported insignificant improvement in biological durability against termites for heat treated wood.

4 CONCLUSIONS
4. ZAKLJUČAK

From the present study, it can be concluded that chemical modification of wood using Citric Acid with Sodium Hypophosphite as catalyst, is an eco-friendly modification process and resulted in better overall performance of modified wood in comparison to the heat-treated wood. Both the modification methods exhibited enhanced properties of dimensional stability and improved resistance to bio deterioration with respect to the untreated controls and improved durability class of treated wood against termites. Chemically modified wood with CA and SHP showed excellent resistance to both white and brown rot fungi as well as termites. This should further be investigated for threshold concentrations including additional test sites with varying climate and more decaying agents to further elucidate on the mode of action. The concentrations of the chemicals may be varied in order to establish an optimum combination for best output. These studies may provide valuable inputs to establish this mode of chemical modification as a cost effective alternative and further establish its use in the niche domain of wood science.

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Influence of Feed Speed and Cutting Depth During Planing on Surface Roughness of Fir, Poplar and Beech Wood

Utjecaj posmične brzine i dodatka za obradu drva na hrapavost površine jelovine, topolovine i bukovine obrađenih blanjanjem

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ABSTRACT • *The roughness of the machined surface is a crucial factor in the woodworking process because it influences the quality of future operations like gluing, sanding, pressing, surface treatment and protection, and assembly. The quality of the machined surface is determined by a number of machining process parameters as well as material properties, and their proper selection and optimization will yield the best results. The purpose of this article is to demonstrate how specific parameters and wood species affect surface roughness. In the experiment, three species of wood: beech (Fagus sylvatica), fir (Abies alba), and poplar (Populus alba) with the same moisture content were used, and combinations of feed speed (5 and 8 m/min) and cutting depth (2 and 4 mm) were created. The processing was done on a wood planer (thickener) machine of the SD-B-510 series manufactured by Robland Machines Belgium. Following that, roughness measurements of Ra, Rz, Rt, and Rq were taken with a focus on the mean deviation of the profile Ra, and an analysis of the results was presented, revealing that different roughness values are obtained with the same processing parameters depending on the wood species. The difference between the greatest (5.36 µm) and lowest (2.41µm) roughness values (Ra) for beech is 2.95 µm, 1.25 µm for poplar, and 1.34 µm for fir.*

KEYWORDS: *surface roughness; planing; feed speed; depth of cut; quality*

SAŽETAK • *Hrapavost obrađene površine ključni je čimbenik u procesu obrade drva jer utječe na kvalitetu naknadnih postupaka poput lijepljenja, brušenja, prešanja, površinske obrade, zaštite i montaže. Kvaliteta obrađene površine određena je brojnim parametrima obrade i svojstvima materijala, a njihovim pravilnim odabirom i optimizacijom postižu se najbolji rezultati. Svrha ovog članka jest pokazati kako specifični parametri i vrsta drva utječu na hrapavost površine obrađene blanjanjem. Istraživanje je provedeno na uzorcima od bukovine (Fagus sylvatica), jelovine (Abies alba) i topolovine (Populus alba) te uz kombinaciju dviju posmičnih brzina (5 i 8 m/min) te dvaju dodataka za obradu blanjanjem (2 i 4 mm). Obrada je provedena na blanjalici (debljači) proizvođača Robland Machines (serija*

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SD-B-510) iz Belgije. Nakon toga izmjereni su parametri hrapavosti Ra, Rz, Rt i Rq, s fokusom na srednjem odstupanju profila Ra. Analiza rezultata pokazala je da se različite vrijednosti hrapavosti dobivaju pri jednakim parametrima obrade za različite vrste drva. Razlika između najveće (5,36 μm) i najmanje (2,42 μm) vrijednosti hrapavosti (Ra) za bukovinu je iznosila 2,95 μm, za topolovinu 1,95 μm, a za jelovinu 1,34 μm.

KLJUČNE RIJEČI: *hrapavost površine; blanjanje; posmična brzina; veličina dodatka za obradu blanjanjem; kvaliteta*

1 INTRODUCTION
1. UVOD

The roughness of the machined surface is one of the parameters by which the quality of the processing itself is evaluated. In order for the product to be economical and competitive on the market, one of the important factors is the quality of the processing, which includes the precision of the processing and the roughness of the processed surface. The roughness of the machined surface directly and indirectly affects the quality of the product itself because further treatments such as surface treatment and gluing depend on the roughness of the machined surface.

In the final processing of wood, roughness represents defects on the surface of the wood that, among others, occur due to the action of the tool on the workpiece. Controlling the surface roughness of the workpiece is crucial in maintaining the quality of the product, and it is essential to constantly implement the flow of the entire production. This depends on the type of processing (sawing, planing, milling, etc.), but with each processing, even with the same processing parameters, a change in roughness can occur on the same sample of wood. This can be affected by the dullness of the tool, a change in moisture content in the wood, or the occurrence of certain defects in the wood (Karahasanović, 1988; Bjelić, 2022).

It is known that the quality of processing is influenced by the anatomical, physical, and mechanical properties of wood. They differ not only depending on the species of wood but also within the same species. In addition, the quality is affected by factors such as blade geometry, cutting direction, cutting depth, feed rate, cutting speed, etc. (Škaljić *et al.*, 2009).

Roughness is a microcosmic geometric shape on the surface of a measuring piece composed of depressions and peaks with a small space between them. There are a number of parameters that describe the roughness of the surface; the basic problem is the correct choice of parameters that correctly describe the surface being measured. Roughness parameters (according to ISO 4289/11) can be divided into sizes related to: height characteristics of irregularities (amplitude parameters), characteristics of irregularities in the length direction (distance parameters), and profile deviation form (hybrid parameters) (Sofuoğlu and Kurtoğlu, 2015; Bojović and Janjić, 2013).

Due to the effort to provide the most precise roughness values, two different measuring systems have appeared: M (Medium) system and E (Envelope) system. System M is a system of the middle line, while system E is a system of a separate line, and they differ only in the rate of occurrence of the roughness problem.

The basic difference between these two systems is in the way roughness is separated from other types of deviation. The basic parameters of roughness are determined by the standard BAS EN ISO 4287:2000, which refers to terms, definitions, and parameters of roughness that are currently in use (Bjelić, 2022).

The mean deviation of the profile (*Ra*) is the arithmetic mean value of the deviation of the profile within the sampling length, approximately determined according to Eq. 1:

$$R_a = \frac{\sum_1^n |Y_i|}{n} \tag{1}$$

Where:

Yi – distance of a single point of the effective profile from the middle line in absolute value,

n – total number of measured points of the effective profile. (Sofuoğlu and Kurtoğlu, 2015)

The medium deviation of the profile (*Ra*) shown in Figure 1 is the generally recognized and most widely used international parameter for roughness. In addition to *Ra* (the arithmetic mean value of profile deviation within the length of sampling), Figure 1 also shows the parameters *Rz* (maximum height of irregularity), *Rq* (root mean square profile deviation), and *Rt* (total height of peak and valley).

The aim of this study is to show how the type of wood, *i.e.* the anatomical structure of wood and various processing parameters, influence the surface quality of the processing and which are the optimum processing parameters to obtain the best surface quality.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Samples preparation
2.1. Priprema uzoraka

Samples of solid beech (*Fagus sylvatica*), fir (*Abies alba*), and poplar (*Populus alba*) wood with dimensions of 35 mm × 15 mm × 500 mm were used for testing. The wood from which the samples were pre-

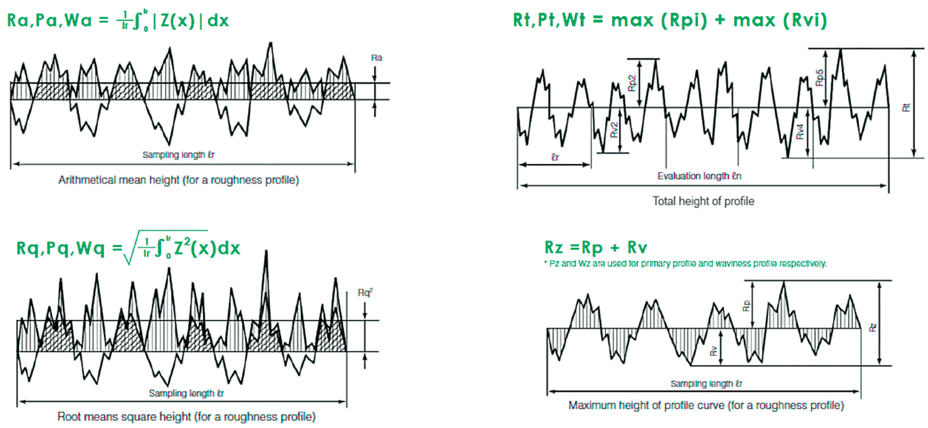

Figure 1 Graphics of roughness parameters evaluated included *Ra*, *Rq*, *Rt* , and *Rz* (Nicolas-Silvente *et al*., 2020)
Slika 1. Grafički prikaz istraživanih parametara hrapavosti *Ra*, *Rq*, *Rt* i *Rz* (Nicolas-Silvente *et al*., 2020.)

pared underwent a drying process where the moisture content of the wood was brought to (8±1) %. Wood samples were machined on a planer (thickener) SD-B-510-serie manufactured by Robland machines Belgium.

Figure 2 presents a machine for planing samples and their appearance. It has a knife holder with a diameter of 100 mm on which 3 knives are placed. The rotational frequency of the tool is 6000 min^{-1}, and the engine power is 5.5 kW. The specifications of the machine are automatic movement of the workpiece feed speed of 5, 8, 10 and 16 m/min and adjustable cutting depth.

The planing of samples for each wood species (beech, fir, and poplar) was done at a feed speed of 5 and 8 m/min, and cutting depth of 2 and 4 mm. Thus, two samples were planed for each of the mentioned combinations.

Table 1 Processing parameters for tested samples
Tablica 1. Parametri obrade ispitivanih uzoraka

Test mark *Oznaka uzorka*	**Wood species** *Vrsta drva*	**Wood density, g/cm³** *Gustoća drva,* g/cm³	**Feed speed, m/min** *Posmična brzina,* m/min	**Cutting depth, mm** *Dodatak za obradu blanjanjem,* mm
1B-52	Beech	0.664	5	2
2B-54	Beech	0.664	5	4
3B-82	Beech	0.664	8	2
4B-84	Beech	0.664	8	4
5T-52	Poplar	0.495	5	2
6T-54	Poplar	0.495	5	4
7T-82	Poplar	0.495	8	2
8T-84	Poplar	0.495	8	4
9J-52	Fir	0.432	5	2
10J-54	Fir	0.432	5	4
11J-82	Fir	0.432	8	2
12J-84	Fir	0.432	8	4

Figure 2 Machine for planing samples and their appearance
Slika 2. Stroj za blanjanje uzoraka i izgled uzoraka

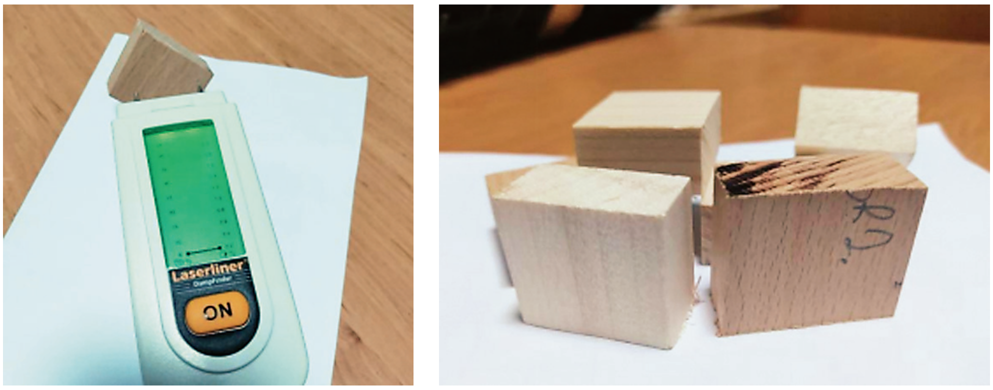

Figure 3 Measurement of moisture content and determination of density of samples
Slika 3. Mjerenje sadržaja vode i određivanje gustoće uzoraka

2.2 Measurement procedures
2.1. Postupci mjerenja

Before measuring the roughness, two tests were performed: measurement of wood density of the above samples and their moisture content. The moisture content of the samples was measured in accordance with the ASTM D4442 standard using the Laserliner 082.011A Damp Finder Plus wood moisture meter. The calibration of the equipment according to the manufacturer's instructions is part of the test method. The hygrometer probes are then pressed against the selected sample, and the moisture values for the given sample are displayed on the display. For every trial made, this method was repeated.

The ISO 3131 standard was used to calculate wood density. The test was performed using previously measured samples with dimensions of 30 mm × 30 mm × 15 mm and moisture content of (8±1) %. Six samples of each wood species were taken, and the arithmetic mean was determined following the measurements.

The measurement of the surface roughness of the machined samples was done using the TMR 120 TM Teck Instrument Co. roughness meter. Ltd. The basic characteristics of this device for measuring the roughness are the measurement parameters (µm): *Ra*, *Rz*, *Rt*, and *Rq*, stroke length of 6 mm, sampling length (*Lr*) values of 0.25, 0.8, and 2.5 mm, access length of 1.25 and 4.0 mm, and indication error ±15 %. This device complies with EN, ISO, DIN, ANSI, JIS, and ASME B46.1 national standards. The difference in the measurement parameters is reflected in the accuracy of the results and the reduction of errors. This measurement was made at a sampling length of 2.5 mm and an access length of 4.0 mm. The test was carried out in the direction of fibre stretching of processing at three arbitrarily determined points (Figure 4), where the results and the average value were recorded.

This test was done in the Wood Testing Laboratory at the Technical Faculty in Bihać, following all the recommendations for roughness testing according to the EN ISO 4287:2010 standard.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

The results of the roughness measurement are shown in Table 2; more precisely, the mean value of the measurement for each sample is shown. In addition to *Ra*, data are given for *Rz*, *Rq*, and *Rt*.

The following diagram shows that the measurement parameters (*Ra*, *Rz*, *Rt*, and *Rq*) change depending on the processing parameters for feed speed 5 and 8 (m/min) and cutting depth 2 and 4 mm. It is also shown for all three examined wood species (beech, fir, and poplar).

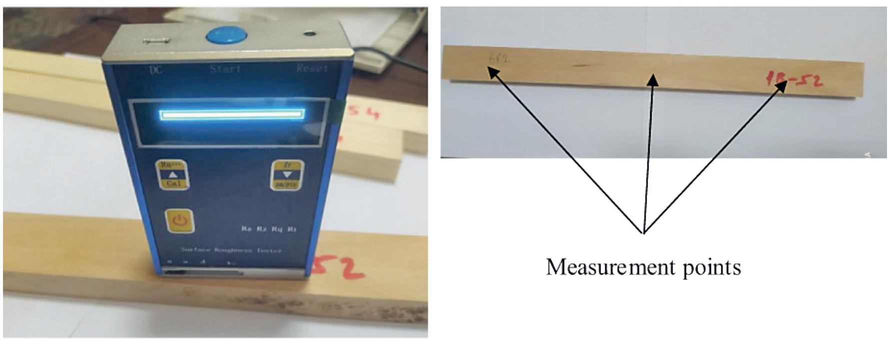

Figure 4 Measurement of roughness on samples
Slika 4. Mjerenje hrapavosti na uzorcima

Table 2 Mean values of measurement results
Tablica 2. Srednje vrijednosti rezultata mjerenja

Test mark *Oznaka uzorka*	**Number of measurements** *Broj mjerenja*	*Ra*, µm	*Rz*, µm	*Rq*, µm	*Rt*, µm
1B-52	9	2.41	10.42	3.01	15.48
2B-54	9	3.39	14.95	4.00	25.57
3B-82	9	3.82	13.30	4.45	22.77
4B-84	9	5.36	18.54	6.41	33.80
5T-52	9	1.61	7.37	1.96	10.79
6T-54	9	2.86	13.02	3.33	21.69
7T-82	9	2.08	9.20	2.80	15.77
8T-84	9	2.86	11.01	3.51	20.33
9J-52	9	2.53	12.13	3.20	19.27
10J-54	9	3.87	13.47	4.60	26.27
11J-82	9	3.20	13.17	3.93	19.47
12J-84	9	3.87	13.63	4.47	20.20

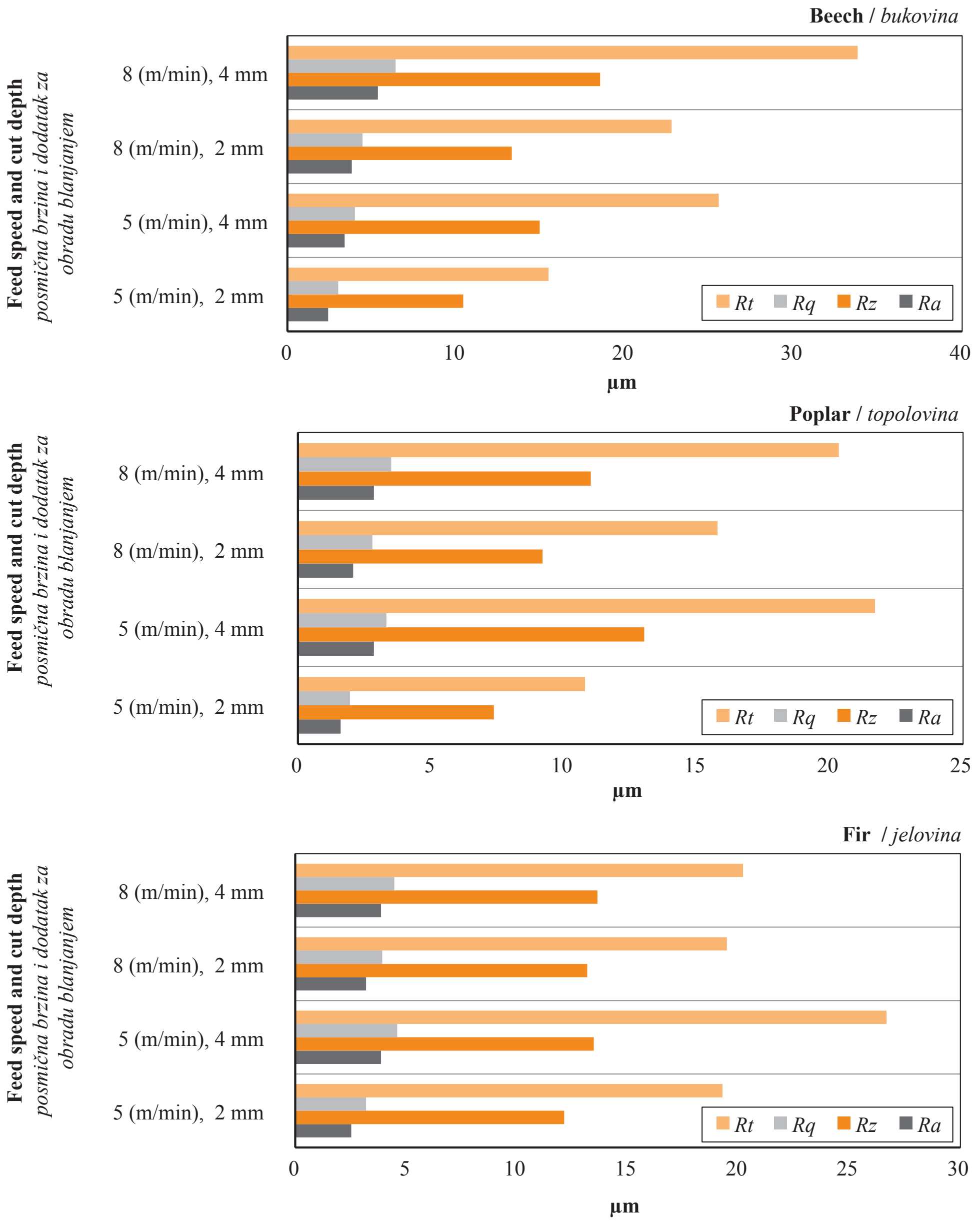

Figure 5 Graphic representation of results of measuring samples roughness according to processing parameters and wood species
Slika 5. Grafički prikaz rezultata mjerenja hrapavosti uzoraka prema parametrima obrade i vrsti drva

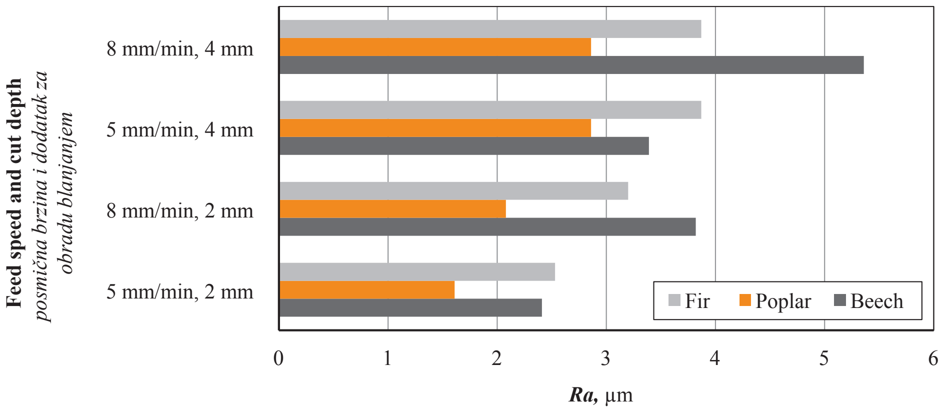

Figure 6 *Ra* – arithmetic mean value of profile deviation shown as comparison for fir, poplar and beech and given parameters
Slika 6. Usporedni prikaz aritmetičke srednje vrijednosti odstupanja profila (*Ra*) za jelovinu, topolovinu i bukovinu te za zadane parametre

The diagram shows the mean value of profile deviation (*Ra*) as an indicator of surface roughness on the measured samples. The dependence of the surface roughness on the processing parameters and the type of wood is visible, and the results showed that the lowest roughness is at feed speed of 5 m/min and cutting depth of 2 mm for all three wood species. Analogously, the highest surface roughness was obtained with increased feed speed and depth at 8 m/min and 4 mm.

The diagram and results show that the influence of feed speed on roughness also depends on the type of wood (that is, the density of the wood), so in the case of beech, the depth of cut had a greater influence on the change in roughness, while in fir and poplar, the opposite was the case as a greater change in roughness is caused by feed speed.

Changes in the mean value of the deviation of the *Ra* profile for the same processing parameters and different species of wood are obvious, with beech being the most pronounced. The difference between the highest and lowest roughness values is 2.95 µm (2.41–5.36); in poplar, this difference is 1.25 µm (1.61–2.86); and in fir, it is 1.34 µm (2.53–3.87). Likewise, the lowest roughness measured for samples made from poplar is 1.61 µm.

The quality of the machined wood surface depends on the processing parameters. The influence of the tool rotational frequency and feed speed results in varying levels of surface roughness; hence, increasing cutting speed while decreasing feed speed improves the processed surface quality in beech wood (Bojović and Janjić, 2013). The behaviour of wood during processing can also be seen in wood-based materials. According to Prakash and Palanikumar (2011), spindle rotation frequency and feed rate, tool shape, and tool diameter have the biggest effects on surface roughness. The study findings showed that the surface roughness values of wood-based panels increased with increasing cutting tool diameter while decreasing with increasing spindle rotation frequency and feed speed (Demir *et al.*, 2022). The porosity of the wood has an impact on the quality of the processed surface (Kudela *et al.*, 2018). This work also shows that the influence is present for the same parameters but different types of wood. For the process of managing wood milling machines or CNC woodworking machines using the milling method, design data is needed with the aim of achieving the highest productivity and lowest energy consumption, while ensuring the desired surface quality. As a result, optimal processing parameters for this type of wood are provided (Loc and Hung, 2021). Further to the above, it is important to know the influence of all processing factors on the quality of the processed surface in order to create the conditions for optimal processing with the desired quality.

4 CONCLUSIONS
4. ZAKLJUČAK

In this study, the surface roughness of beech, poplar, and fir was examined. The samples were processed at various feed speed rates - 5 and 8 m/min and cutting depths - 2 and 4 mm. After examining the surface roughness results on the above samples, the following can be concluded:

- The influence of the processing parameters on the roughness is visible, and the results show that the smallest roughness is at feed speed of 5 m/min and at cutting depth of 2 mm, for all three species. Analogously, the highest surface roughness was obtained with increased feed speed and depth at 8 m/min and 4 mm. The reason for this is that the increase in feed speed and depth directly affects the roughness of the processed surface; the higher these parameters are, the higher is the roughness in all three species.
- The density and anatomical structure of the different wood species provide variable results when the same processing conditions are used. The mean value of

profile deviation Ra for the parameters feed speed of 5 m/min and cutting depth of 2 mm shows that poplar has the lowest value while beech has the highest. This difference is also obvious when considering other factors - *Rz*, *Rt*, and *Rq*.

- The difference between the highest and lowest measured value of *Ra* is 2.95 µm for beech (Ra_{min} = 2.41, Ra_{max}= 5.36). It is 1.25 m for poplar (Ra_{min} =1.61, Ra_{max} =2.86) and 1.34 m for fir (2.53 - 3.87). Furthermore, the lowest value of *Ra* was observed for poplar samples and is 1.61 m. Such changes are also visible in other measured values of *Rz*, *Rt* and *Rq*.

This research shows the influence of the processing parameters on the surface roughness, that is, the influence of the feed speed and depth of cut, along with the effect of wood species on the roughness at the same processing parameters. Knowing this, but also considering other research, opens the possibility of improving the processing quality, such as the surface roughness, by changing the processing parameters of feed and cutting depth, but also by choosing the type of wood that can provide improved results.

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Integration of Sustainable Development Goals in Higher Education and Research Processes Related to Forestry and Wood Science

Uključivanje ciljeva održivog razvoja u visoko obrazovanje i znanstvena istraživanja vezana za šumarstvo i drvo

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ABSTRACT • *Global challenges, including climate change, land and ecosystem degradation, and a growing population have direct or indirect impact on natural resources and are forcing people to seek new ways of production and consumption that respect the ecological limits of our planet. To meet the challenges, the 2030 Agenda for Sustainable Development was adopted by 193 countries at the United Nations Summit in September 2015. The forest-wood chain has also been identified as closely linked to sustainable development and the Sustainable Development Goals (SDGs). Especially in countries with high forest cover, such as Slovenia, forest-wood chain is recognized in policy documents as key element for achieving the SDGs. The importance of the SDGs is not only recognized in national legislation, but also in relation to higher education and research institutions. With the aim to investigate how well higher education teachers and students are familiar with SDGs and whether they consider them important for the forestry and wood industry and if high education teachers and researchers integrate the SDGs into educational programs and research, survey was conducted with the employees (n=61) and students (n=185) of the University of Ljubljana, Biotechnical Faculty, Department of Forestry and Renewable Forest Resources and Department of Wood Science and Technology. The results show that both employees and students of the Department of Forestry and Renewable Forest Resources as well as employees and students of the Department of Wood Science are familiar with SDGs. All the respondents find SDGs important for the forestry and wood industry. All the participants especially emphasized SDG 15 – Life on land. The results also showed that, in the future, employees of both departments plan to integrate more SDGs into their educational and research process. Finally, it was concluded that educational and research institutions and integration of SDGs into their educational and research process could be an important step towards sustainability and achieving goals of 2030 Agenda for Sustainable Development. Students who will work in the forestry and wood industry sectors in the future could integrate and promote more sustainable practises in the sectors if they have sufficient knowledge of the SDGs and high perception of sustainability.*

KEYWORDS: *sustainable development goals; forestry; wood science; education; research*

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SAŽETAK • *Globalni izazovi kao što su klimatske promjene, uništavanje zemljišta i ekosustava te sve veći broj stanovnika izravno ili neizravno utječu na prirodne resurse i potiču ljude da traže nove načine proizvodnje i potrošnje koji poštuju ekološka ograničenja našeg planeta. Kako bi odgovorile na te izazove, 193 zemlje usvojile su na skupu Ujedinjenih naroda u rujnu 2015. Agendu održivog razvoja do 2030. Lanac šuma – drvo također je prepoznat kao usko povezan s održivim razvojem i ciljevima održivog razvoja (SDG). Osobito je u zemljama koje imaju veliku pokrivenost šumama, poput Slovenije, lanac šuma – drvo u političkim dokumentima prepoznat kao ključni element za postizanje ciljeva održivog razvoja. Važnost ciljeva održivog razvoja nije naglašena samo u nacionalnom zakonodavstvu već i u obrazovnim i istraživačkim institucijama. Cilj istraživanja bio je utvrditi koliko su nastavnici i studenti u visokom obrazovanju upoznati s ciljevima održivog razvoja, smatraju li ih važnima za šumarstvo i drvnu industriju te integriraju li nastavnici i znanstvenici ciljeve održivog razvoja u obrazovne programe i istraživanja. Na Biotehničkom fakultetu Sveučilišta u Ljubljani, na Odsjeku za šumarstvo i obnovljive šumske resurse te na Odsjeku za znanost o drvu i drvnu tehnologiju, provedena je anketa sa zaposlenicima (n = 61) i studentima (n = 185). Rezultati pokazuju da su zaposlenici i studenti obaju odsjeka upoznati s ciljevima održivog razvoja. Svi ispitanici smatraju da su ciljevi održivog razvoja važni za šumarstvo i drvnu industriju. Svi su posebno istaknuli cilj održivog razvoja 15 – Život na kopnu. Rezultati su također pokazali da zaposlenici obaju odsjeka planiraju u budućnosti integrirati više ciljeva održivog razvoja u svoj obrazovni i istraživački proces. Zaključili smo da bi integracija ciljeva održivog razvoja u obrazovni i istraživački proces mogla biti važan korak prema održivosti i postizanju ciljeva Agende održivog razvoja do 2030. Studenti koji će u budućnosti raditi u sektoru šumarstva i drvne industrije mogli bi integrirati i promicati održive prakse u tim sektorima ako imaju dovoljno znanja o ciljevima održivog razvoja i visoku svijest o održivom razvoju.*

KLJUČNE RIJEČI: *ciljevi održivog razvoja; šumarstvo; znanost o drvu; obrazovanje; istraživanje*

1 INTRODUCTION
1. UVOD

Natural resources are the fundamental basis for life and human well-being. Global challenges, including hunger and poverty, gender inequality and inequality in general, climate change, land and ecosystem degradation, deforestation, biodiversity loss, resource depletion and a growing population have direct or indirect impact on natural resources and are forcing people to seek new ways of production and consumption that respect the ecological limits of our planet (European Commission, 2018).

Until 1972, these challenges were not adequately addressed neither by general public nor policy makers (UNEP, 2023). In 1972, the Declaration of the United Nations Conference on the Human Environment was adopted addressing global challenges. Since then, various policy documents were adopted such as the Rio Declaration on Environment and Development (1992), the Agenda 21 (1992), the Millennium Declaration (2000), the Johannesburg Declaration on Sustainable Development (2002) and the Rio+20 Outcome Document (2012), which, together with various other environmental and development conventions as well as regional and global commitments, led to the development of the 2030 Agenda for Sustainable Development (United Nations, 2015). The 2030 Agenda for Sustainable Development was adopted by 193 countries at the United Nations Summit in September 2015 (United Nations, 2015). The 2030 Agenda for Sustainable Development integrates the three dimensions of sustainable development - economic, social and environmental (United Nations, 2015). These three dimensions are integrated into the 17 overarching Sustainable Development Goals (hereafter: SDGs) and 169 associated targets, with the aim of addressing economic, social and environmental issues addressing crucial global challenges (Gue *et al.*, 2020; Niestroy, 2016; Ramirez *et al.*, 2019; Belmonte-Ureña *et al.*, 2021; Katila *et al.*, 2019). The SDGs and their targets form a complex, integrated system with clear sectoral emphasis but also strong interlinkages among goals and targets. The 2030 Agenda for Sustainable Development forms an overarching framework that is expected to guide government and non-state actors' efforts at different scales, from global to local, until 2030 (Katila *et al.*, 2019). Regarding the legal status of the SDGs, it is important to note that they are not legally binding at the national level. However, the 2030 Agenda for Sustainable Development influences and informs international agreements, policies and programmes (United Nations, 2015). Individual countries can integrate SDGs into their legal and policy frameworks and make them legally binding.

The 2030 Agenda for Sustainable Development offers an extensive framework for shaping and coordinating governmental policies. Cultural differences of the general public and understanding of sustainability can influence the willingness of people to accept and understand SDGs (Guan and Zhang, 2023). Some previous studies (Novieastari, 2022; Zamora-Polo *et al.*, 2019) suggest that the general public knowledge, especially among university students, about sustainability and 2030 Agenda for Sustainable Development is not sufficient.

Since forests cover about 35 % of European land, they are crucial for provisioning timber for wood industry (Forest Europe, 2020). Therefore, both sectors are crucial for sustainable development at all scales, from global to local. Forests are explicitly mentioned in the context of SDG 15 – *Life on land*, where sustainable use of forests and reversing biodiversity loss is mentioned. In relation to SDG 6 – *Clean water and sanitation,* forests are seen as water-related ecosystems (United Nations, 2015). Furthermore, there are strong synergies between forests and SDG 13 – *Climate action,* as forests are important for carbon sequestration and storage (United Nations, 2015). Although the wood industry is not explicitly mentioned in the context of the SDGs, there are strong links to SDG 9 – *Industry, innovation and infrastructure* (Mancini *et al.*, 2019). This specific goal is focused on promoting inclusive and sustainable industrialization, where wood, wood products and wood residues could represent a sustainable raw material for extended value chains organized along the key principles of circular bioeconomy (i.e., adding value through the cascading use biomass, closing the energy and material loops). In line with this, the wood industry can be considered as part of SDG 9 – *Industry, innovation and infrastructure* (United Nations, 2015; INTRuST, 2022). The forest-wood chain, considered by most countries as a sector that includes both forestry and wood processing, is also recognized in the connection with other SDGs like SDG 1 – *No poverty*, SDG 2 – *Zero hunger*, SDG 3 – *Good health and well-being*, SDG 5 – *Gender equality,* SDG 7 – *Affordable and clean energy,* SDG 8 – *Decent work and economic growth,* SDG 11 – *Sustainable cities and Communities* and SDG 12 – *Responsible Production and Consumption.* SDG 17 – *Partnerships for the goal* is also related to the forestry and wood industry because partnerships and support facilitate the achievement of the goals, not only in social initiatives, but also in the industry and above all in environmental protection strategies (Ma *et al*., 2022; Baumgartner, 2019; Hazarika and Jandl, 2019; United Nations, 2015).

Slovenia has a forest area of 58.0 % (ZGS, 2022). Consequently, wood is the most important strategic raw and industrial material (MGTŠ, 2021; MGRT, 2020). The importance of the forest-wood chain is recognized in multiple strategies and policy documents. For example, the Slovenian Development Strategy 2030 (MKRR, 2017), which is the fundamental strategic document of Slovenia, also recognizes the importance of forests and wood industry for achieving SDGs. Furthermore, the Slovenian Industrial Strategy 2021-2030 (MGTŠ, 2021) promotes wood-based industry, decarbonization of energy-intensive industries (e.g., paper industry) and transition to a low-carbon circular economy. This is also recognized in the National Energy and Climate Plan 2030 (MOPE, 2020). Forest-related policy documents (e.g., the Operational Programme of the National Forest Programme 2022-2026 (2022)), recognize sustainable forest management as a key instrument to achieve the 2030 Agenda for Sustainable Development Goals.

The importance of the SDGs is recognized not only in the national strategic documents, but also in the strategic documents of higher education and scientific research institutions (University of Freiburg, 2021; SLU, 2019; CZU, 2023; Wageningen University and Research, 2022; BOKU, 2020; Univerza v Ljubljani, 2022). The importance of SDGs is also recognized by the Biotechnical Faculty, which is part of the University of Ljubljana (Biotehniška fakulteta, 2023), where it is possible to study forestry and wood science. However, some previous research shows that there are also challenges and gaps in the implementation of SDGs in the higher education and research process globally (Grano and Correia, 2020). Anyhow, higher education and scientific research institutions can contribute to SDGs from a variety of perspectives: research, education, operations and governance and external leadership (SDSN Australia/Pacific, 2017). In addition, Belmonte-Ureña *et al.* (2021) report that scientific research plays a crucial role in the success of the SDGs, Walentowski *et al.* (2020) report that well-educated young academics are key to putting knowledge-based thinking into practice for responsible resource management for a sustainable world, and Zuluaga-Ortiz *et al.* (2022) report that universities influence the basic competencies of students in order to produce good professionals. In addition, some previous studies (e.g. Novieastari *et al.*, 2021; Omisore *et al*., 2017; Zamora-Polo *et al*., 2019) suggest that knowledge and awareness about the SDGs is not sufficient among young people, especially students in higher education. Based on the insights gained from reviewing previous research articles and the limited availability of research articles on the topic of higher education teachers and researchers' knowledge of the SDGs and their influence on students' knowledge of the SDGs, the aim of this study was to: 1) find out how well the higher education teachers, researchers and students of the Department of Forestry and Renewable Forest Resources (hereafter: Department of Forestry) and the Department of Wood Science and Technology (hereafter: Department of Wood Science) of the Biotechnical Faculty of the University of Ljubljana know the SDGs (i.e. acquaintance, understanding), and 2) whether they consider the SDGs to be important for the forestry and wood industry (i.e. perceived significance), and 3) investigate whether high education teachers and researchers integrate the SDGs into educational programmes and research, and 4) how they will integrate the SDGs into their educational and research process in the future.

2 METHODS
2. METODE

The data for this study was collected by developing the structured questionnaire for the employees (higher education teachers and researchers) and students of the Department of Forestry and the Department of Wood Science. The questionnaire for employees consisted of 17 questions and a questionnaire for students consisted of 10 questions. The questionnaire for employees was pre-tested in March 2023 by five employees, and the questionnaire for students was pre-tested by the same five employees in September 2023. Based on the pre-tests, both questionnaires were revised to make the questions as understandable and precise as possible. Most of the questions were Likert-type scale responses (Likert, 1932), some of them required additional explanations (open questions), and some were binary. The surveys were conducted on-line, using 1KA programme (FDV, 2017). The questionnaire was distributed to all employees who are engaged in educational or research process (n= 84) and all students (n= 440) of the Department of Forestry and the Department of Wood Science. The data (available in Uhan *et al.*, 2023) for employees were collected between 4 and 19 April 2023, and the data for students were collected between 15 September and 27 October 2023.

Within the employees' survey period activity, 61 persons completed the survey, which means that the response rate was 73 % (Figure 1). Within the students' survey period activity, 185 persons completed the survey, which corresponds to a response rate of 42 % (Figure 2).

The collected data were imported and coded in MS Excel and processed in JASP, version 17.1 (JASP team, 2023). To check the quality of the data and to detect errors, outliers and missing values, all data were first checked with frequencies. Variables in the questionnaires were analysed using frequency distributions and mean values. In order to determine the differences between departments, the non-parametric Mann-Whitney U test was used (Navarro *et al.*, 2019).

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

Altogether 61 higher education teachers and researchers completed the survey, 51 % from the Department of Forestry and 49 % from the Department of Wood Science. The largest share of respondents among employees of both departments were teaching assistants and researchers (31 %), followed by assistant professors (23 %), full professors (20 %), specialist advisers (13 %) and associate professors (8 %). One senior lecturer and a teacher of skills also participated in the survey. Among the respondents, the majority (over 90 % all together) are engaged in research work, and 66 % of them also identified themselves as being engaged in education (Figure 1).

Altogether 185 students responded to the survey, 44 % from the Department of Forestry and 56 % from the Department of Wood Science. Most of the students who participated in the survey in both departments were undergraduates, from Academic Study Programmes (51 %) and Professional Study Programmes (31 %), while the students enrolled in Masters' Study (18 %) made up a significantly smaller proportion of respondents (Figure 2). The distribution of the answers represents a direct consequence of number of enrolled students per specific studies and specific year as in both departments there are more students enrolled in Academic Study Programmes or Professional Study Programmes (Department of Forestry – 86 %, Department of Wood Science – 81 %).

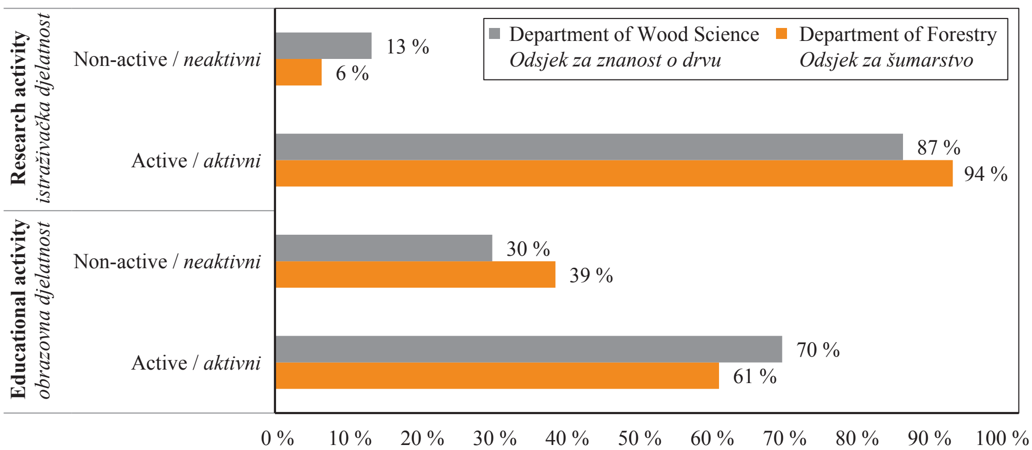

Figure 1 Respondents educational and research activities of employees
Slika 1. Obrazovne i istraživačke aktivnosti ispitanih zaposlenika

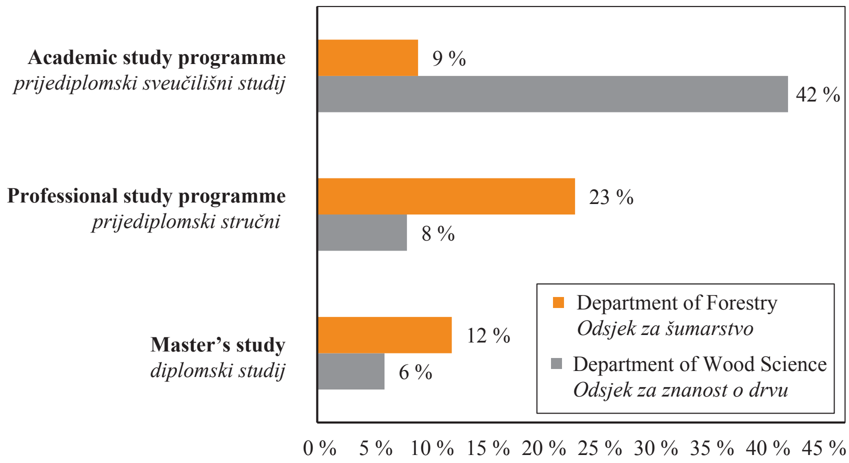

Figure 2 Distribution of students who have responded
Slika 2. Raspodjela studenata koji su odgovorili

3.1 General knowledge about sustainable development goals
3.1. Općenito poznavanje ciljeva održivog razvoja

The first part of the survey focused on the employees and students' general knowledge about the SDGs. Both rated their knowledge about the SDGs on a Likert-type scale from 1 (I know very little about the SDGs) to 5 (I know a lot about the SDGs). The employees rated their knowledge about the SDGs with an average value of 3.4, where 27 % rated their general knowledge about the SDGs as good. Furthermore, no statistical differences were found between employees of the Department of Forestry and the Department of Wood Science (*Mann-Whitney U-test, U=305*, *p=0.087*) in relation to their knowledge about the SDGs. Students rated their general knowledge about the SDGs with an average value of 2.9. 56 % of students rated their general knowledge about the SDGs as good. Among students from both departments no statistical differences were found (*Mann-Whitney U-test, U=4076.000, p=0.069*). Differences in general knowledge about the SDGs between employees and students were significant (*Mann-Whitney U-test, U=3608.000, p=0.003*), employees rated their general knowledge about the SDGs as better. The importance of familiarity with the SDGs was also studied by Jabeen (2022), who found that the knowledge about the SDGs had increased. Alm *et al.,* (2020), who studied student awareness and knowledge about the SDGs, emphasized that higher education teachers in higher education institutions and universities can play a crucial role in achieving the SDGs. However, previous studies report that low percentage of students and employees have good knowledge about the SDGs (Smaniotto *et al.,* 2019; Omisore *et al.,* 2017), which is not consistent with our study, especially concerning the results of students' survey. On the contrary, Fajar Jati *et al.* (2019) report that 89.5 % of students are aware of the SDGs and 62.5 % have good knowledge about the SDGs, which is in line with our results. However, Fajar Jati *et al.* (2019) focused only on students and did not include employees.

3.2 Importance of sustainable development goals for forestry and wood industry sectors
3.2. Važnost ciljeva održivog razvoja za sektor šumarstva i drvne industrije

The employees and students of both departments were asked to judge the general importance of the SDGs for the forestry and wood industry sectors (Figure 3 and Figure 4) on five-point Likert-type scale from 1 (Not important) to 5 (Very important). Both the employees and students of the Department of Forestry have rated SDG 6 – *Clean water and sanitation* and SDG 15 – *Life on land* as the most important SDGs for forestry. Employees rated both SDGs with an average value of 4.7, while students gave SDG 6 – *Clean water and sanitation* a lower value (average value 4.5) and SDG 15 – *Life on land* even lower rating (average value 4.4). The results are in line with expectations as forests are seen as one of the water-related ecosystems in SDG 6 – *Clean water and sanitation*. The emphasis on SDG 15 – *Life on land* is also expected as it focuses on the protection, restoration and sustainable use of terrestrial ecosystems, and halting the loss of biodiversity recognizes forests and their rich biodiversity as essential for sustainable development (Katila *et al.*, 2019). In addition, employees also rated SDG 7 – *Affordable and clean energy* (average value 4.5), SDG 12 – *Responsible consumption and production* (average value 4.5), and SDG 13 – *Climate action* (average value 4.5) as very important. Students additionally recognized the importance of SDG 12 – *Responsible consumption and production* (average value 4.2), and SDG 13 – *Climate action* (average value 4.2). Results are in line with previous research since forests play an important role in the supply chain for energy production, and because the role of forest is also recognized for ensuring access to affordable, reliable and sustainable energy services

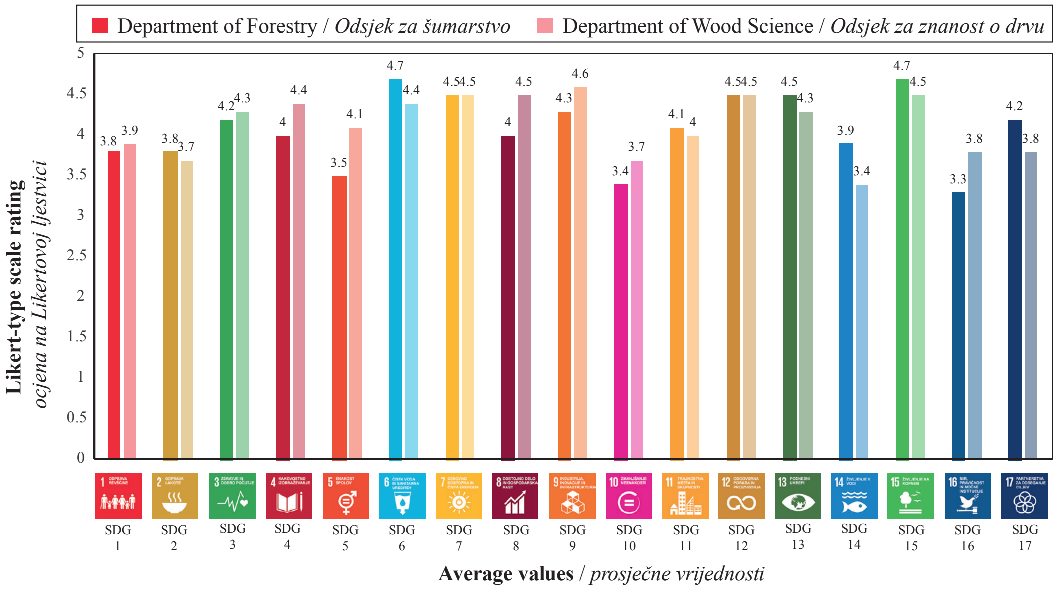

Figure 3 Importance of each SDG for forestry or wood sector (employees)
Slika 3. Značajnost svakoga od SDG-ova za šumarstvo ili drvnu industriju (zaposlenici)

though SDG 7 – *Affordable and clean energy* (Katila *et al.*, 2019). In addition, responsible consumption and production can make a positive contribution to forest conservation and contribute to a more sustainable supply of wood and forest raw materials (Katila *et al.*, 2019). Forests are regularly mentioned in the context of the climate change, carbon sequestration and storage, and reduction of greenhouse gas emissions, which is the main goal of SDG 13 – *Climate action* (Katila *et al.*, 2019).

The employees of the Department of Wood Science have rated SDG 9 – *Industry, innovation and infrastructure* as the most important, with an average value of 4.6. Results are expected as this goal is centred on three main pillars: industry, infrastructure and innovation, which is the focus of wood industry sector (United Nations, 2015). In addition, the employees also recognized SDG 7 – *Affordable and clean energy* (average value 4.5), SDG 12 – *Responsible consumption and production* (average value 4.5), and SDG 15 – *Life on land* (average value 4.5) as important. Students from the Department of Wood Science recognize the majority of SDGs as important for wood industry: SDG 2 – *Zero hunger*, SDG 3 – *Good health and well-being,* SDG 4 – *Quality education,* SDG 6 – *Clean water and sanitation,* SDG 7 – *Affordable and clean energy,* SDG 8 – *Decent work and economic growth,* SDG 9 – *Industry, innovation and infrastructure* and SDG 15 – *Life on land* (with average value 4.5 each). The recognition of SDG 7 - *Affordable and clean energy* is also expected as wood industry contributes to the affordable and clean energy through sustainable

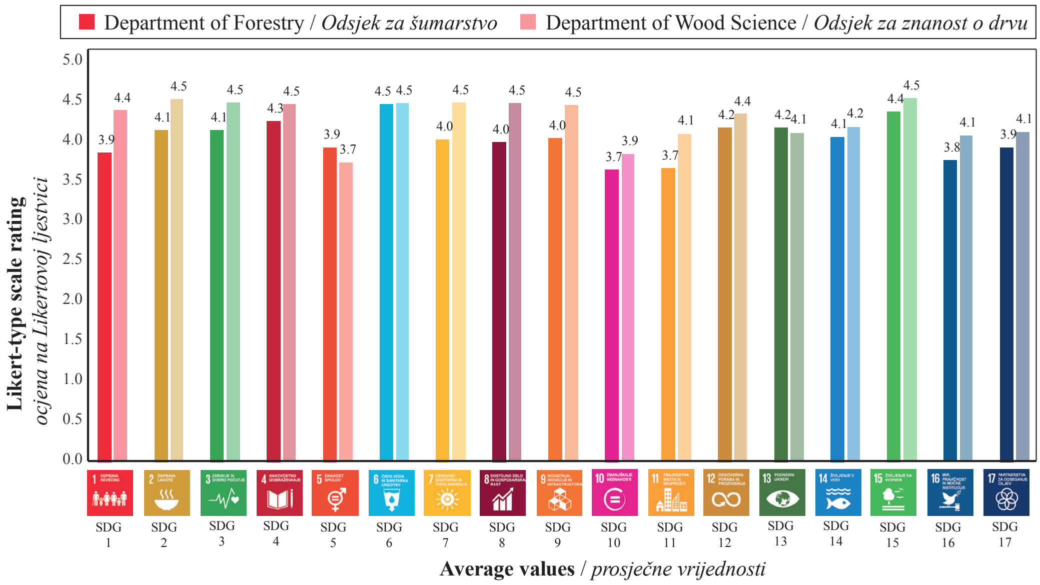

Figure 4 Importance of each SDG for forestry or wood sector (students)
Slika 4. Značajnost svakoga od SDG-ova za šumarstvo ili drvnu industriju (studenti)

use of traditional wood fuels (firewood and charcoal), processed wood fuels (pellets), liquid biofuels and bio-power, which can play an important role in the provision of energy (Katila *et al.*, 2019). The relation to SDG 12 – *Responsible consumption and production* is also expected as the wood industry promotes responsible consumption and production, as wood is neither a toxic raw material nor hazardous waste (Verkerk *et al.*, 2021; Maier, 2021). Furthermore, wood industry offers complementary opportunities to forestry to achieve the SDGs within the forest-wood chain, so the importance of the wood industry in the context of SDG 15 – *Life on land* is predictable. SDG 15 – *Life on land* focuses mainly on the sustainable management of all types of forests and halting deforestation, to which a responsible and sustainable wood industry can contribute (Katila *et al.*, 2019); this can explain why the employees and students emphasized this SDG. Finally, SDG 8 – *Decent work and economic growth,* that was identified by both employees and students of the Department of Wood Science as an important one (average value 4.5), which mainly refers to the promotion and support of sustainable economic growth, is probably recognized because wood industry is related to sustainable economic growth (Maier, 2021).

3.3 Integration of sustainable development goals into higher educational and research process
3.3. Uključivanje ciljeva održivog razvoja u proces visokoškolskog obrazovanja i istraživanja

The survey conducted among employees of both departments also included the part about the integration of the SDGs in their research and educational process. These results were compared with the students' answers, to determine whether the students recognize the integration of the SDGs into the educational process in both departments. The results of the employees of both departments (Figure 5 and Figure 6) show that higher education teachers at the Department of Forestry most frequently integrate SDG 13 – *Climate Action* (average value 4.0) and SDG 15 – *Life on land* (average value 4.0) into their educational process, which is expected as both SDGs are related to the sustainable use of natural resources (United Nations, 2015). For example, SDG 13 – *Climate action* and SDG 15 – *Life on land* are integrated in the courses: Wildlife Ecology and Management Planning, Management of Wildlife Populations, Close-to-nature Silviculture, Silviculture, Dendrology with Breeding of Forest Trees, Introduction to Nature Conservation, The Basics of Forest Protection, Use of Forest Biomass and Forest Policy. It was also found that the research process of the employees of the Department of Forestry is mainly related to SDG 15 – *Life on land* (average value 3.1) and SDG 13 – *Climate action* (average value 2.7); however, the integration of the SDGs into their research work is much lower. For example, the employees of the Department of Forestry are currently engaged in the projects related to SDG 13 – *Climate action* (average value 2.7) (*Learning to realize multiple forest policy objectives under climate related stress and disturbance* and *Natural regeneration and tending of forests following large-scale disturbances: harmonization of ecological, economic and forest policy aspects)* and to SDG 15 – *Life on land* (average value 3.1) (*Efficient management of private forests to support wood mobilization*, *Learning to realize multiple forest policy objectives under climate related stress and disturbance*, and *Support for improved management of the interactions between large herbivores and forests*).

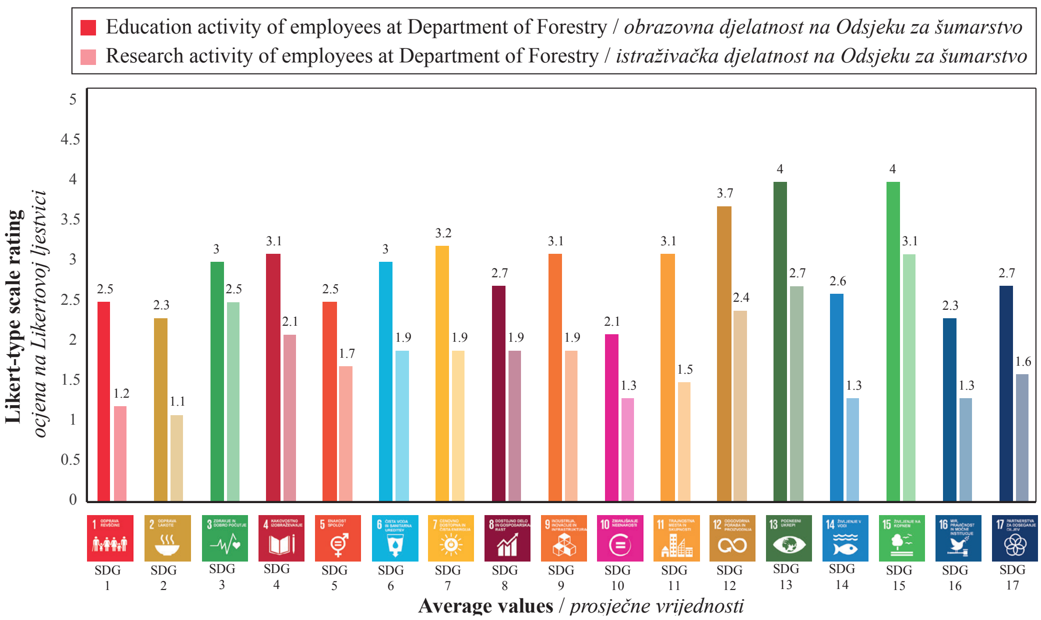

Figure 5 Involvement of SDGs in teaching and research at Department of Forestry and Renewable Forest Resources
Slika 5. Uključenost SDG-ova u obrazovanje i istraživanje na Odsjeku za šumarstvo i obnovljive šumske resurse

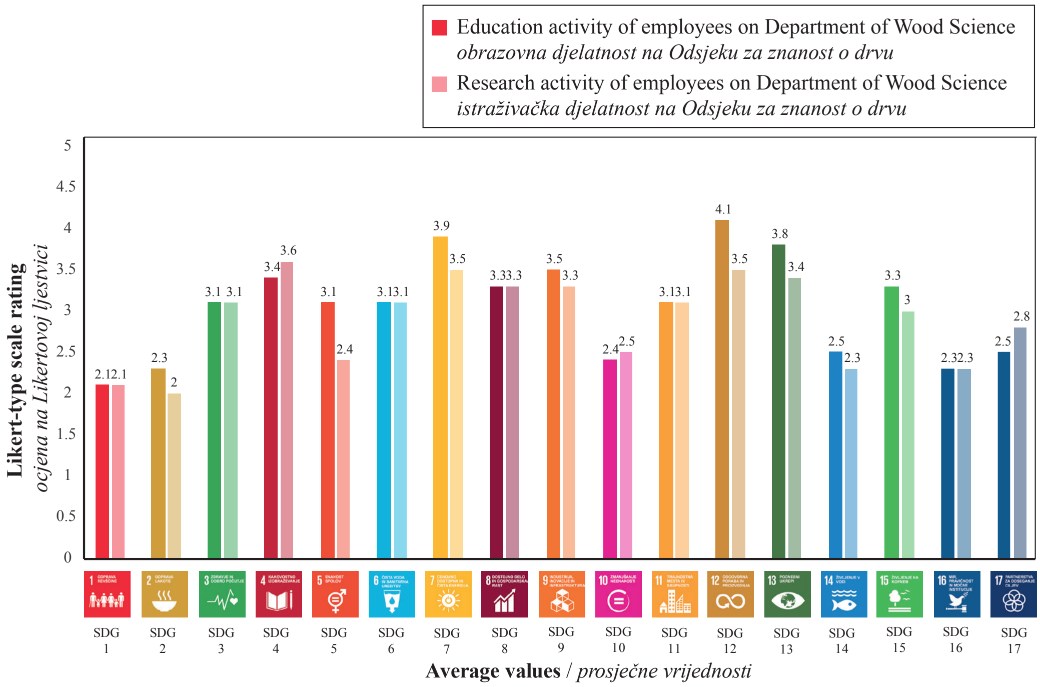

Figure 6 Involvement of SDGs in teaching and research at Department of Wood Science and Technology
Slika 6. Uključenost SDG-ova u obrazovanje i istraživanje na Odsjeku za znanost o drvu i drvnu tehnologiju

On the contrary, higher education teachers from the Department of Wood Science most frequently integrate SDG 12 – *Responsible Consumption and Production* (average value 4.1) and SDG 7 – *Affordable Clean Energy* (average value 3.9) into their education and research process. For example, SDG 12 – *Responsible consumption and production* and SDG 7 – *Affordable and clean energy* are integrated, in the courses: Environmental Protection in Wood Industry, Biotechnology in Wood Industry, Biotechnology of Higher Fungi, Environmental and Economic Aspects of Wood Protection and Wood Modification, Economics of Wood-Industry Company and Basics of Entrepreneurship, Management of Wood Industry Company with Microeconomics, Management of Production Processes in Wood Industry, Organization of Production in Wood Industry Company. Similar to the Department of Forestry, the SDGs are considered to a lower extent in the research process of employees of the Department of Wood Science than in the educational processes. The research process currently relates to SDG 4 – *Quality education* (average value 3.6) (*Alliance of Centres of Vocational Excellence in the Furniture and Wood Sector, Study in the shortage of personnel in the wood industry as a basis for the renewal of training* and SDG 7 – *Affordable clean energy* (average value 3.5) (*Affordable Clean Energy*: *Wood and Lignocellulosic Composites)*.

Further, answers of the employees from both departments were compared with the students results (Figure 7). The results show that the students from the Department of Forestry find SDG 12 – *Responsible consumption and production* (average value is 4.1) as the most integrated SDG in the forestry studies, followed by SDG 12 - *Responsible consumption and production*, SDG 7 – *Affordable and clean energy* (average value is 3.9), SDG 13 – *Climate action* (average value is 3.8) and SDG 9 – *Industry, innovation and infrastructure* (average value is 3.5). These SDGs are integrated, for example, into the courses: Close-to-nature Silviculture, Silviculture, Forest Management and Planning, Introduction to Nature Conservation, Basics of Forest Protection, Forest Policy, Meteorology, Old-growth Forests and Forest Reserves and Planning of Harvesting Technologies. Students from the Department of Wood Science emphasized only two SDGs– SDG 4 – *Quality education* (average value is 3.6) and SDG 7 – *Affordable and clean energy* (average value is 3.5). These SDGs are integrated, for example, into the courses: Environmental Protection in Wood Industry, Environmental and Economic Aspects of Wood Protection and Wood Modification, Economics of Wood-Industry Company and Basics of Entrepreneurship, Management of Wood Industry Company with Microeconomics, Management of Production Processes in Wood Industry, Organization of Production in Wood Industry Company and Technical Mechanics.

SDGs are integrated into education and research process at the Department of Forestry and the Department of Wood Science, probably because most of the current research programmes (e.g., Horizon Europe, Interegg Europe, Forestvalue, Forestvalue2) include topics related to sustainable development and achieving SDGs (Borchardt *et al*., 2023; European Commision, 2020; Interreg Europe, 2023; Forestvalue, 2023). Moreover, research process in higher education institutions is

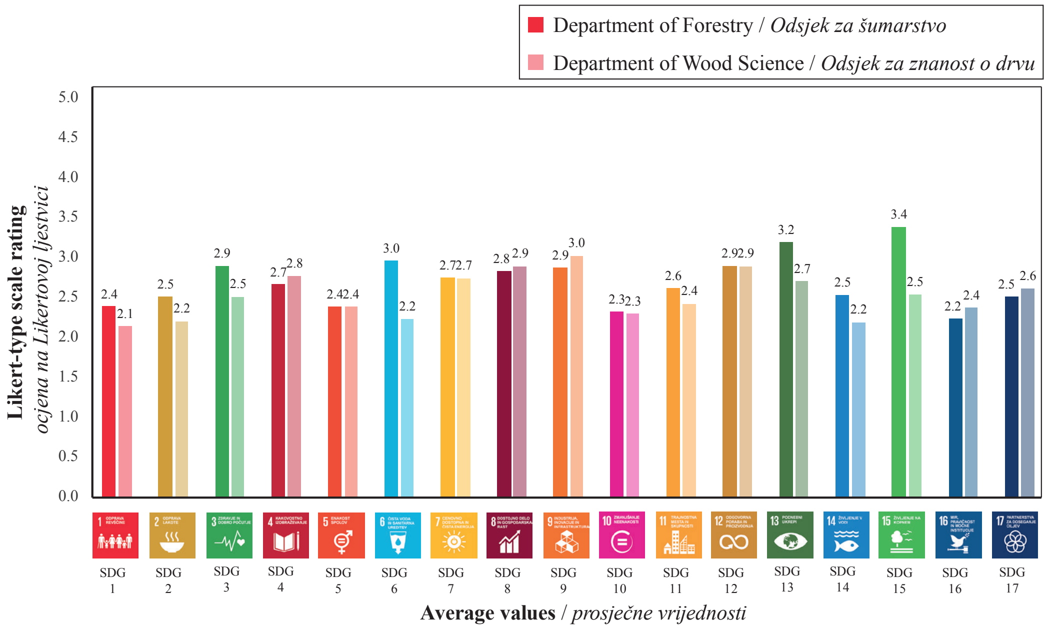

Figure 7 Involvement of SDGs in teaching at Department of Forestry and Department of Wood Science based on students' responses
Slika 7. Uključenost SDG-ova u obrazovanje i istraživanje na Odsjeku za šumarstvo i Odsjeku za znanost o drvu na temelju studentskih odgovora

directly related to the education process and therefore SDG 4 – *Quality education* (Elsen *et al.*, 2009). The student's responses confirm that courses in both departments integrate SDGs, but students do not always recognize that specific SDGs are integrated into the course, as the results show that students recognized different courses in relation to SDGs and emphasized different SDGs than employees, which could be a consequence of the limitations regarding emphasis on the SDGs, which was also recognized by Leal Filho (2023).

3.4 Future integration of sustainable development goals into educational and research process
3.4. Uključivanje ciljeva održivog razvoja u proces visokog obrazovanja i istraživanja u budućnosti

This part of the research focused only on employees of both departments. They were asked whether they intend to incorporate more SDG-related content in their educational and/or research process in the future. Employees of both departments rated this question with an average value of 3.7 on a Likert-type scale from 1 (I will not include the SDGs at all) to 5 (I will definitely include the SDGs). From the results, it can be concluded that SDGs are likely to be more included in the educational and research process of employees of both departments. The results are promising since integrating sustainable development and SDGs into the educational programmes, curricula and research creates meaningful learning outcomes (Lozano *et al.*, 2017; Alm *et al.*, 2020; Walentowski *et al.*, 2020; Belmonte-Ureña *et al.,* 2021).

4 CONCLUSIONS
4. ZAKLJUČAK

Sustainable development is a comprehensive topic that affects the economy, society and environment. Further, forest-wood chain represents an opportunity to achieve the 2030 Agenda for Sustainable Development. Moreover, higher education and research institutions were recognized as important actors who should integrate SDGs into their research and educational process and spread awareness about sustainability. Employees at the Department of Forestry and the Department of Wood Science of the Biotechnical Faculty of the University of Ljubljana already integrate SDGs in both educational and research processes. Further, it is important to note that the employees intend to include more SDGs in their work in the future, recognizing that their curriculum influences students' knowledge about the SDGs and their perception of sustainability. Based on these facts it can be concluded that educational and research institutions and integration of SDGs into their educational and research process could be an important step towards sustainability and achieving goals of 2030 Agenda for Sustainable Development. Students who will work in the forestry and wood industry sectors in the future could integrate and promote more sustainable practises in the sectors if they have sufficient knowledge of the SDGs and high perception of sustainability. New approaches in the industry could help in achieving goals of the 2030 Agenda for Sustainable Development. The study and its findings open up an interesting discussion on the relevance of the SDGs for higher education in the forest-wood chain.

The study must consider some limitations regarding the interpretation of the results, as there were some limitations in the survey implementation. The main drawback of the study is that the population studied is very limited as it focuses on only two departments of one university. However, the results open up promising possibilities for a future extension of the study to similar higher education programmes at other universities (Europe and globally). Another major drawback is that the results are based on self-assessment. Therefore, the results, especially the answers to the questions on familiarity with the SDGs, cannot be viewed with absolute certainty.

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Water-Related Properties and Biological Durability of Wood-Based Composites

Svojstva kompozita na bazi drva u doticaju s vodom i njihova biološka trajnost

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ABSTRACT • *There is insufficient data regarding the biodegradation of wood-based composites (WBC) by wood decay fungi. This study aimed to evaluate the biological durability and water-related properties of different WBC types. Although WBC are primarily designed for dry environments, in building applications, they may face increased moisture risks due to water leakage, condensation, or humid air. The panels, including oak-pine shield parquet (OPP), oriented strand board (OSB), birch plywood (BP), particle board (PB), laminated particle board (LPB), moisture-resistant particle board (MRPB), medium density fibreboard (MDF), laminated medium density fibreboard (LMDF) and moisture resistant medium density fibreboard (MRMDF), were subjected to attack by brown rot fungus Coniphora puteana. After 16 weeks of exposure, the most resistant WBC against biodegradation were BP, moisture-resistant MDF, and laminated MDF, as they exhibited a mass loss lower than 5 %. Conversely, all other WBC types showed high susceptibility to biodegradation, with a mass loss exceeding 35 %. LMDF (8 – 51 %) and MRMDF had the lowest water absorption (WA) within 168 h (2 – 46 %), while non-treated MDF exhibited the highest WA among all composite types with 190 % water uptake. With regards to thickness swelling, all WBC types, except for LPB and MDF, demonstrated values below 20 %. The influence of adhesives (phenol-formaldehyde or melamine urea-formaldehyde) used in WBC did not show a clear impact on water-related properties or biological durability.*

KEYWORDS: *biodegradation; fungi; thickness swelling; water absorption; wood-based composites*

SAŽETAK • *Ne postoji dovoljno podataka o biorazgradnji kompozita na bazi drva (WBC) gljivama truležnicama. Cilj ovog istraživanja bio je procijeniti biološku trajnost i svojstva različitih vrsta kompozita na bazi drva u doticaju s vodom. Iako su kompoziti na bazi drva ponajprije dizajnirani za suhe okolišne uvjete, u zgradama se mogu naći izloženi povećanom riziku od vlage zbog curenja vode, kondenzacije ili povećane vlage u zraku. Napadu gljiva smeđe truleži Coniphora puteana izloženi su površinski obrađeni višeslojni parket od hrastovine i borovine (OPP), ploča iverica s usmjerenim makroiverjem (OSB), brezova furnirska ploča (BP), ploča iverica (PB), ploča iverica obložena laminatom (LPB), vodootporna ploča iverica (MRPB), ploča vlaknatica srednje gustoće (MDF), ploča vlaknatica srednje gustoće obložena laminatom (LMDF) i vodootporna ploča vlaknatica srednje gustoće (MRMDF). Nakon 16 tjedana izlaganja u ovom istraživanju najotpornijim kompozitima na bazi drva na biorazgradnju pokazali su se brezova funirska ploča, vodootporni MDF i MDF obložen laminatom jer su imali gubitak mase manji od 5 %. Suprotno tome, svi ostali tipovi kompozita na bazi drva pokazali su visoku sklonost biorazgradnji, uz gubitak mase veći od 35 %. Najnižu apsorpciju vode (WA) unutar 168 sati imali su LMDF (8 – 51 %) i*

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MRMDF (2 – 46 %), dok je neobrađeni MDF pokazao najveću apsorpciju među svim vrstama kompozita, uz 190 %-tno upijanje vode. Kad je riječ o debljinskom bubrenju, sve su vrste kompozita na bazi drva, osim ploče iverice obložene laminatom i MDF-a, pokazale vrijednosti niže od 20 %. Za ljepila (fenol-formaldehidno ili melamin urea-formaldehidno) upotrijebljena u kompozitima na bazi drva nije potvrđen jasan utjecaj na svojstva u doticaju s vodom ili njihov utjecaj na biološku trajnost kompozita.

KLJUČNE RIJEČI: *biorazgradnja; gljive; debljinsko bubrenje; apsorpcija vode; kompoziti na bazi drva*

1 INTRODUCTION
1. UVOD

Wood-based composites (WBC) are widely used in furniture production and building construction for both interior and exterior applications. Scientific studies still address the issue of moving WBCs towards improved performance and higher sustainability (Zanuttini and Negro, 2021). The wood composite products are made of thin veneers, strands, flakes, particles, and fibres glued with an adhesive resin. Accordingly, the wood within the composites often has the same physical and biological properties as the original log. These wood properties include hygroscopicity, tendency to swell as moisture content (MC) increases, and susceptibility to biological attack at the same moisture level.

The type of adhesive and manufacturing process are among the most important factors contributing to differences in wood moisture relationships between wood composites and solid wood. The more water-resistant the bonded resin system becomes and the more deeply that resin system penetrates the wood cell wall, the more durable the wood composite becomes (Winandy and Morrell, 2017). Phenol-formaldehyde (PF) resins are typically used in the manufacture of construction plywood and oriented strand board, where exposure to weather during construction is a concern. Other moisture exposure situations, such as temporary weather exposure, occasional plumbing leaks, or wet foot traffic, may also necessitate the use of PF resins. Urea-formaldehyde (UF) resins are typically used in the manufacture of products used in interior applications, primarily particleboard and medium-density fibreboard (MDF). Melamine-formaldehyde (MF) resins are used primarily for decorative laminates and paper coating. Melamine urea-formaldehyde (MUF) resins are often used when greater water resistance is required than that obtained with UF resin (Stark *et al*., 2010).

Many WBC are generally intended for interior uses in a dry environment. However, in buildings, they are potentially subjected to elevated moisture risks due to water leaks, condensation, or damp air. When water intrusion occurs, a critical factor that affects water penetration is the water absorption rate of the wood-based materials (TenWolde and Rose, 1993). As a result, high moisture levels in building materials reach conditions where biological attack is possible, and this can result in substantial repair costs. Biodegradation has a big influence on the life cycle of various materials. Rot fungi are among the biggest groups of organisms that degrade wood materials (Schmidt, 2006). The biodegradation surveys in wooden constructions have mainly been focused on the type of decayed wood (soft- or hardwood), fungal species, and location of fungal attack (roof, floor, ceiling, etc.) (Alfredsen *et al*., 2005; Irbe *et al.,* 2012), and environmental conditions (Viitanen *et al.,* 2010). There is a lack of data on the biodegradation of WBC by wood decay fungi. The literature survey showed that laboratory studies on fungal biodegradation of WBC are mainly limited to the testing of a few panel types (Curling and Murphy, 1999; Kartal and Green III, 2003) or focused specifically on wood-plastic composites (Feng *et al.,* 2020; Yeh *et al.,* 2021; Buschalsky *et al.,* 2022) or deterioration of WBC by mould fungi (Yang, 2008). In the present study, the different types of WBC, including laminated and moisture-resistant panels, were investigated in relation to moisture and decay resistance properties. The brown rot fungus *Coniphora puteana* was selected for experiments as a widespread and economically important wood decay fungus in wooden constructions.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Wood-based composites
2.1. Kompoziti na bazi drva

Commercial oak-pine shield parquet (OPP) (thickness 14.1 mm), oriented strand board (OSB) (thickness 20.7 mm), birch plywood (BP) (thickness 14.7 mm), particle board (PB) (thickness 15.8 mm), laminated particle board (LPB) (thickness 16.1 mm), moisture resistant particle board (MRPB) (thickness 16.1 mm), medium density fibreboard (MDF) (thickness 16.2 mm), laminated medium density fibreboard (LMDF) (thickness 18.2 mm), and moisture resistant medium density fibreboard (MRMDF) (thickness 16.1 mm) were purchased from the retail market. The panels were conditioned at a temperature of 20 °C and 65 % relative humidity (RH) to a constant weight. The density of materials was determined for specimens 5cm × 5cm in size using the conditioned volume and conditioned mass.

2.2 Water absorption and thickness swelling
2.2. Apsorpcija vode i debljinsko bubrenje

The water absorption (WA) and thickness swelling (TS) of different WBC panels were measured according to ASTM D1037-12 (2020), with modifications using 5 cm × 5 cm specimens (6 replicates in each group). The specimens were immersed in distilled water and the weight was measured after 2 h, 4 h, 6 h, 8 h, 24 h, 48 h, 72 h, 96 h, and 168 h. Water absorption was calculated using the following equation:

$$WA\ (\%) = ((m_1 - m_0)/\ m_0))\ \times 100 \quad (1)$$

Where m_0 is the mass of the specimen before immersion and m_1 is the mass of the specimen after immersion.

Thickness swelling was measured after 24 h, 48 h, 72 h, 96 h, and 168 h and calculated as follows:

$$TS\ (\%) = ((t_1 - t_0)/\ t_0))\ \times 100, \quad (2)$$

Where t_0 is the thickness of the specimen before immersion and t_1 is the thickness after immersion.

2.3 Fungal resistance
2.3. Otpornost na gljive

The fungal resistance of WBC specimens was determined according to the modified European Prestandard ENV 12038:2002. Six parallel specimens with dimensions of 50 cm × 25 cm × 15 cm were cut from the pre-conditioned panels and exposed to the brown rot fungus *C. puteana* (Schumacher ex Fries) Karsten (BAM Ebw. 15) on a medium containing 5 % malt extract concentrate and 3 % Fluka agar (Sigma-Aldrich) in Kolle flasks. Scots pine wood was used as a control. Sterilized specimens were aseptically placed on a 3-mm glass supports and incubated at 22±2 °C and 70 % RH for 16 weeks. Aftero cultivation, the specimens were removed from the culture vessels, brushed free of mycelium, and oven dried at (103 ± 2) °C. The loss in dry mass (%) of the specimens was used as the criterion for determining the extent of the fungal attack. If the mean mass loss is greater than 3 %, the decay susceptibility index (*DSI*) is calculated as follows:

$$DSI = T/\ S \times 100 \quad (3)$$

Where *T* is the mass loss (%) of an individual test specimen, *S* is the mean mass loss (%) of the appropriate set of control specimens.

DSI values of 100 indicate the same decay resistance as that of the timber used for the control. Materials with lower *DSI* values are more resistant to fungal attack.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

The density of composites ranged from 526 kg/m^3 for OPP composite to 771 kg/m^3 for MDF (Figure 1). The highest density of over 700 kg/ m^3 was determined for BP, MDF, LMDF, and MRMDF materials.

Figure 2 shows the water absorption results of all tested specimens during the 2 to 168 h immersion. The lowest absorption was observed for BP, LMDF, and MRMDF reaching ~50 % at the end of the experiment. The lowest absorption correlated with the higher density of these composites (except for MDF) (Figure 1). OPP, OSB, PB, and MRPB specimens demonstrated similar absorption behaviour during the test reaching 28-47 % after 24 h, and 70-80 % after 168 h.

MDF had the highest absorption after 168 h reaching ~190 %, although after 24 h, OPP, BP, PB,

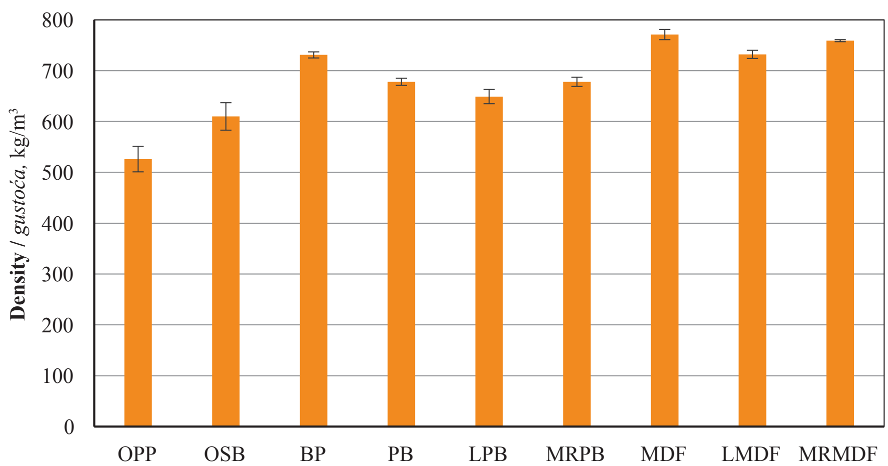

Figure 1 Density of tested specimens - oak-pine shield parquet (OPP), oriented strand board (OSB), birch plywood (BP), particle board (PB), laminated particle board (LPB), moisture-resistant particle board (MRPB), medium density fibreboard (MDF), laminated medium density fibreboard (LMDF), moisture resistant medium density fibreboard (MRMDF)
Slika 1. Gustoća ispitivanih uzoraka: površinski obrađenoga višeslojnog parketa od hrastovine i borovine (OPP), ploče iverice s usmjerenim makroiverjem (OSB), brezove furnirske ploče (BP), ploče iverice (PB), ploče iverice obložene laminatom (LPB), vodootporne ploče iverice (MRPB), ploče vlaknatice srednje gustoće (MDF), ploče vlaknatice srednje gustoće obložene laminatom (LMDF) i vodootporne ploče vlaknatice srednje gustoće (MRMDF)

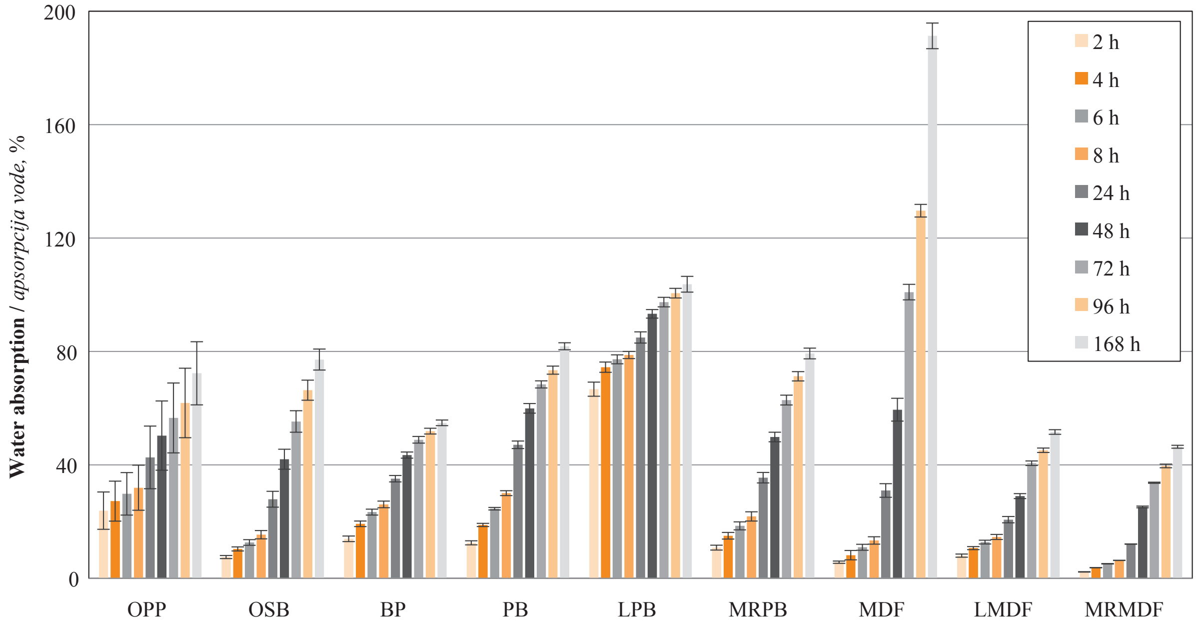

Figure 2 Water absorption of tested specimens after 2 h to 168 h - oak-pine shield parquet (OPP), oriented strand board (OSB), birch plywood (BP), particle board (PB), laminated particle board (LPB), moisture resistant particle board (MRPB), medium density fibreboard (MDF), laminated medium density fibreboard (LMDF), moisture resistant medium density fibreboard (MRMDF)

Slika 2. Apsorpcija vode ispitanih uzoraka nakon 2 do 168 h: površinski obrađenoga višeslojnog parketa od hrastovine i borovine (OPP), ploče iverice s usmjerenim makroiverjem (OSB), brezove furnirske ploče (BP), ploče iverice (PB), ploče iverice obložene laminatom (LPB), vodootporne ploče iverice (MRPB), ploče vlaknatice srednje gustoće (MDF), ploče vlaknatice srednje gustoće obložene laminatom (LMDF) i vodootporne ploče vlaknatica srednje gustoće (MRMDF)

LPB, and MRPB specimens had higher absorption compared to MDF. In general, MDF lamination and moisture resistance treatment decreased the water uptake, resulting in significantly lower absorption of LMDF (8-51 %) and the lowest absorption for MRMDF (2-46 %) specimens.

The laminated surface of PB did not protect the composite from elevated water uptake. LPB had significantly higher absorption during all immersion periods (67-103 %) than PB (13-82 %), while moisture-resistant PB had slightly lower absorption (11-79 %) compared to PB. LPB had the highest absorption after 24 h (~80 %) compared to other composites. In similar research, the physical-mechanical properties of PB manufactured with Eucalyptus wood, bamboo, and rice husk particles were assessed. Water absorption after 24 h of wood particle board was ~43 %, while for other combinations it was 67-72 % (de Melo *et al.,* 2014).

The findings provide proof that the commercial treatment aimed at reducing water absorption was effective for moisture-resistant PB and MDF compared to untreated or laminated composites. An alternative method to enhance the water-repellent properties of WBC is through modification techniques such as acetylation or thermal modification. Research conducted by Pipíška *et al.* (2020) demonstrated that OSB strand boards made of acetylated strands and thermally modified strands exhibited a substantial improvement in water resistance.

Figure 3 illustrates the thickness swelling of all WBC specimens over the period of 24 to 168 hours. Wood products incorporating larger-size wood particles like BP and OPP demonstrated consistently low and minimal swelling ranging from 6.5 % to 8.6 % throughout the entire 168h testing duration. In contrast to most particle-based panels, plywood undergoes negligible irreversible thickness swelling when subjected to moisture (Stark *et al.,* 2010). Additionally, OSB panels displayed relatively low swelling levels ranging from 7 % to 15 % during the entire testing period. Previous studies have reported that untreated BP experienced swelling between 6 % and 12 % during immersion periods of 2 to 72 hours (Bekhta *et al.,* 2020), while for untreated OSB the swelling was 20 % and 25 % after 24 h and 168 h, respectively (Pipíška *et al.,* 2020). There was no clear relation between the material density and swelling. For example, OPP and BP with similarly low swelling had different densities, while MDF with the highest density demonstrated the highest thickness swelling.

Notably, PB exhibited lower thickness swelling (13-18 %) after all testing periods, in contrast to laminated PB (22-26 %). Moisture-resistant PB demonstrated similar swelling levels to untreated PB, suggesting that lamination and moisture-resistant treatment did not effectively protect PB from long-term water exposure. In a study by de Melo *et al.* (2014), PB manufactured with Eucalyptus wood, bamboo, and rice

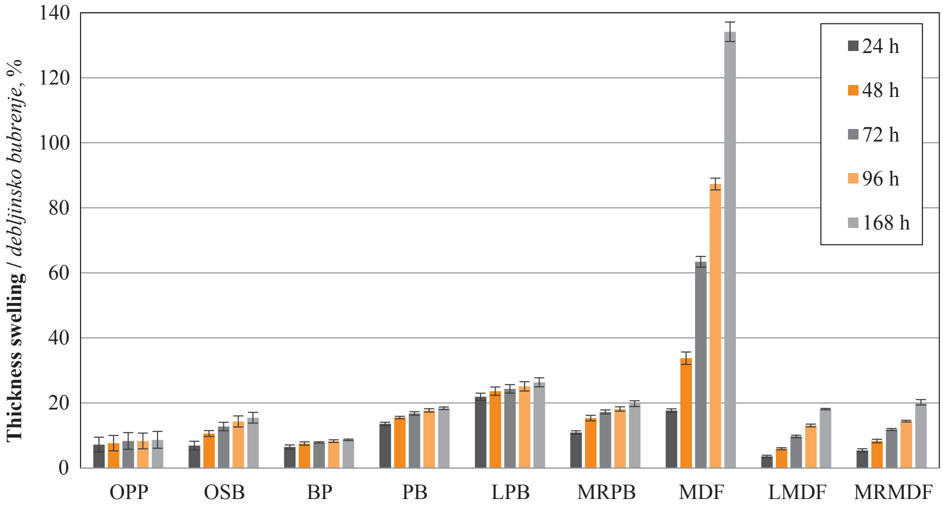

Figure 3 Thickness swelling after 24 h to 168 h immersion in water of tested specimens - oak-pine shield parquet (OPP), oriented strand board (OSB), birch plywood (BP), particle board (PB), laminated particle board (LPB), moisture resistant particle board (MRPB), medium density fibreboard (MDF), laminated medium density fibreboard (LMDF), moisture resistant medium density fibreboard (MRMDF)

Slika 3. Debljinsko bubrenje ispitivanih uzoraka nakon 24 do 168 h potapanja u vodi: površinski obrađenoga višeslojnog parketa od hrastovine i borovine (OPP), ploče iverice s usmjerenim makroiverjem (OSB), brezove furnirske ploče (BP), ploče iverice (PB), ploče iverice obložene laminatom (LPB), vodootporne ploče iverice (MRPB), ploče vlaknatice srednje gustoće (MDF), ploče vlaknatice srednje gustoće obložene laminatom (LMDF) i vodootporne ploče vlaknatice srednje gustoće (MRMDF)

husk exhibited thickness swelling of 31 %, 30 %, and 49 % after 24 h, respectively.

MDF showed the highest swelling after 48 hours, reaching 34 %, and remarkably increased to 134 % after 168h. Laminated MDF and moisture-resistant MDF displayed similar swelling patterns during the experiment, reaching 4-5 % after 24 hours and 18-20 % after 168 h. For MDF panels manufactured using bamboo, swelling reached 11-21 % after 24 hours (Marinho *et al.,* 2013). PB bonded with MUF resin and MDF panels bonded with UF resin demonstrated higher thickness swelling compared to panels containing PF resin (Figure 3), as moisture exposure leads to the breakdown of bond-forming reactions (Stark *et al.,* 2010).

Various board types displayed different susceptibility to the fungal decay after 16 weeks of exposure

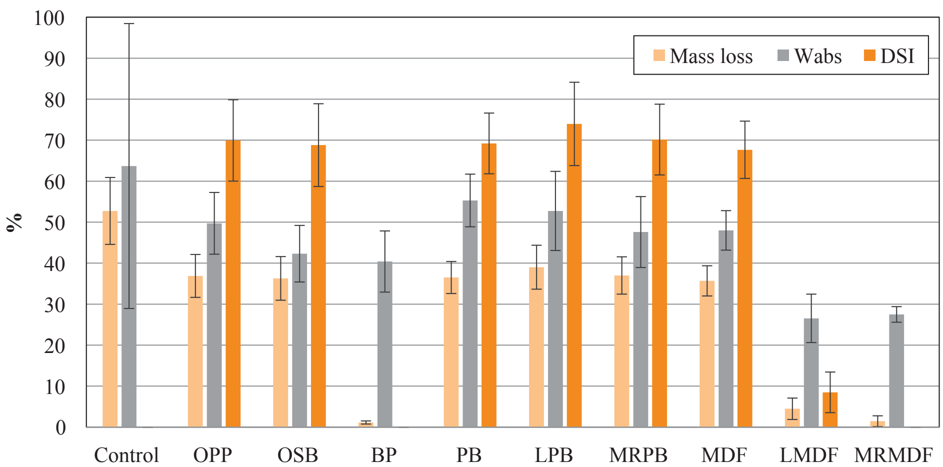

Figure 4 Mass loss (%), moisture content (W_{abs} %), and decay susceptibility index (*DSI*) of tested specimens (n = 6) after 16 weeks of degradation by brown rot fungus *C. puteana.* Abbreviations: oak-pine shield parquet (OPP), oriented strand board (OSB), birch plywood (BP), particle board (PB), laminated particle board (LPB), moisture resistant particle board (MRPB), medium density fibreboard (MDF), laminated medium density fibreboard (LMDF), moisture resistant medium density fibreboard (MRMDF)

Slika 4. Gubitak mase (%), sadržaj vode (W_{abs} %) i indeks osjetljivosti na truljenje (*DSI*) ispitivanih uzoraka (*n* = 6) nakon 16 tjedana razgradnje gljivom smeđe truleži *C. puteana*. Kratice: OPP – površinski obrađeni višeslojni parket od hrastovine i borovine, OSB – ploča iverica s usmjerenim makroiverjem, BP – brezova furnirska ploča, PB – ploča iverica, LPB – ploča iverica obložena laminatom, MRPB – vodootporna ploča iverica, MDF – ploča vlaknatica srednje gustoće, LMDF – ploča vlaknatica srednje gustoće obložena laminatom i MRMDF – vodootporna ploča vlaknatica srednje gustoće

(Figure 4). The average mass loss of the WBC specimens varied between 1.1 % for BP and 39.0 % for LPB, with pinewood control exhibiting significantly higher mass loss at 52.7 %. Among the WBC types, BP, moisture-resistant MDF, and laminated MDF reached the highest resistance to biodegradation, with mass losses below 5 %. Conversely, all other WBC types demonstrated a high susceptibility to biodegradation, with mass losses exceeding 35 %.

A mass loss above 20 % in the virulence control specimens provided confirmation of the viability of the fungal strain (ENV 12038:2002). The moisture content of both the test specimens and controls after the test exceeded 25 %, ensuring favourable conditions for fungal growth (Figure 4). The majority of tested WBC cannot be designated as fully resistant (except for BP and MRMDF) to attack by *C. puteana* since the mass loss of specimens was greater than 3 %. In this case, the DSI was calculated, with the mass loss of each test specimen being expressed as a percentage of the mean loss in mass of the control specimens. The DSI results demonstrated that laminated MDF performed the lowest value. Consequently, this material was more resistant (*DSI* 8.5) to fungal attack than other composites (*DSI*~70).

Several factors may contribute to a higher resistance to fungal attack, including physical properties, adhesive type, and wood particles. In terms of physical properties, certain composite types with a higher density above 700 kg/m³ (Figure 1) aligned with lower mass losses observed in BP, MRMDF, and LMDF panels, with the exception of MDF. However, the high mass loss observed in MDF can be attributed more to the absence of additional treatments such as moisture resistance in MRMDF or lamination in LMDF, rather than density alone.

The influence of adhesives on decay resistance yielded contradictory results. Various factors, including the fluid properties of the resin, wood anatomical characteristics and permeability, and processing conditions, contribute to resin penetration (Kamke and Lee, 2007), making it complex to isolate the specific impact of the adhesive. BP and OSB panels bonded with PF resins displayed distinct biodegradability, with BP experiencing a minor mass loss (1.1 %) and OSB panels showing high mass loss (over 30 %). This characteristic can be attributed more to the larger size of wood particles (veneers) in BP rather than the adhesive specific influence. According to Stark *et al.* (2010), plywood properties depend on the quality of veneer plies, their arrangement, the adhesive used, and the level of control of bonding conditions during production. Previous studies have also observed low mass loss in BP after decay (Irbe *et al.,* 2017). This property has been associated with the potential fungicidal effect of the PF adhesive due to the presence of phenols and formaldehyde. When water-soluble phenolic compounds were leached out, the plywood became susceptible to fungal attack (Irbe *et al*., 2017). OSB, manufactured from thin wood strands with controlled size, placement, and orientation, aims to enhance performance like structural plywood (Stark *et al.,* 2010). However, the wood strands in OSB remained vulnerable to fungal attack, indicating the need for further improvement in its resistance to decay (Figure 4).

PB panels, including laminated and moisture-resistant PB, bonded with MUF resin, were susceptible to fungal decay, resulting in mass losses exceeding 30 %. The production process of PB involves mechanically reducing the wood raw material into small particles, applying the adhesive to the particles, and consolidating them under heat and pressure to form a panel product (Stark *et al*., 2010). The presence of small wood particles and an adequate MC exceeding 47 % contributed to the efficient biodegradation of PB. Additional treatments such as lamination and moisture-resistant additives did not provide protective effects against fungal attack. In the case of lamination (LPB), the lack of protection against the fungus can be attributed to unsealed specimen edges. The moisture resistance property of MRPB is more likely associated with relative humidity, rather than material wetting. However, in practical applications, occasional wetting in constructions can lead to increased water absorption and thickness swelling, making the material more susceptible to penetration by fungal hyphae and subsequent degradation (Figure 4).

Except for moisture-resistant MDF and laminated MDF, MDF panels bonded with MUF resin demonstrated susceptibility to fungal degradation. The reduced biodegradation observed in these panels can be attributed to their hydrophobic properties with a lower MC (27 %). There are several distinguishing factors between fibreboard and particleboard, with the physical configuration of the wood element being the most notable difference. Fibreboard leverages the inherent strength of wood to a greater extent compared to particleboard, as wood is fibrous by nature (Stark *et al*., 2010).

There is ongoing exploration of various approaches to improve the biological durability of WBC. Ustaömer *et al.* (2010) noted that the decay resistance of MDF was enhanced by increasing the melamine content in the MUF resin and by using higher chemical concentrations. Treatment with vapor-boron was found to improve WBC resistance to fungal decay (Tsunoda, 2001), while the effectiveness of vacuum-impregnation of WBC with alkaline copper quat (ACQ) depended on the specific type of WBC and wood rot fungi (Tascioglu and Tsunoda, 2010). Pressure to reduce the use of wood preservatives is growing, prompting exploration of nonchemical methods that may be suitable for certain applications (Winandy and Morrell, 2017). For instance, acetylation of wood prior to composite manufacturing can be used to enhance moisture resistance. Another proposed method is thermal modification, which is believed to decrease the availability of carbohydrate compounds, thereby reducing the risk of fungal attack. Additionally, an alternative approach to enhancing the biological durability of composite materials could involve using naturally durable wood species or environmentally friendly biocides.

4 CONCLUSIONS
4. ZAKLJUČAK

Laminated and moisture-resistant MDF exhibited limited water absorption compared to other tested composite materials, reaching an absorption range of 46 – 52 % which was attributed to lamination, moisture resistance treatment, and higher density. The lowest thickness swelling observed in OPP and BP materials can be related to their larger wood particle size, and not to density. The impact of adhesive on water-related properties was not clearly evident from the observations. In terms of decay resistance, all tested WBC samples showed higher resistance compared to the control specimens. BP, as well as laminated, and moisture-resistant MDF panels, demonstrated the highest resistance, with mass losses below 5 %. The reduced fungal resistance observed in other WBC types may be related to factors such as smaller wood particle size, higher moisture susceptibility, or the absence of moisture treatment for the wood particles.

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Development of Laminated Flooring Using Wood and Waste Tire Rubber Composites: A Study on Physical-Mechanical Properties

Izrada laminata upotrebom kompozita od drva i otpadne gume: studija o fizičko-mehaničkim svojstvima

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ABSTRACT • *This study aimed to develop laminate flooring composite using a combination of wood and waste tire rubber (WTR). Plywood panels were produced by using beech (Fagus orientalis), alder (Alnus glutinosa), and poplar (Populus) veneers in a 7-ply configuration. To enhance the physical-mechanical properties of the panels, three loadings of nano-SiO_2 (0, 2, and 4 wt%) along with 2 wt% of hexamethyldisilazane (HMDS) were added. Commercial urea-formaldehyde (UF) resin and methylene diphenyl diisocyanate (MDI) were used to bind the wood layers and rubber layers together. The mechanical properties, including modulus of rupture (MOR), modulus of elasticity (MOE), impact strength (IS), hardness strength (HS), and physical properties, such as density (D), water uptake (WU), and thickness swelling (TS), were evaluated. The results showed that increasing the WTR content led to improvements in the physical properties (D, WU, and TS), while negatively affecting the mechanical properties (MOR, MOE, IS, and HS) of the resulting panels. However, the addition of nano-SiO_2 improved both the physical and mechanical properties (MOR, MOE, and HS) of the panels. Furthermore, it was observed that the mechanical properties were enhanced with increasing the number of beech layers, although the WU of panels decreased compared to panels made with alder and poplar. Overall, the improvement in the physical properties of the panels followed the order of the arrangement of rubber layers > nano-SiO_2 content > veneer layers.*

KEYWORDS: *silica nanoparticles; rubber waste; plywood; physical-mechanical properties; flooring*

SAŽETAK • *Postavljeni je cilj bio izraditi kompozitni laminat kombinacijom drva i otpadne gume (WTR). Furnirske su ploče proizvedene upotrebom sedam slojeva furnira drva bukve (Fagus orientalis), johe (Alnus glutinosa)*

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i topole (Populus). Kako bi se poboljšala fizička i mehanička svojstva ploča, dodana su tri punjenja nano-SiO$_2$ (0, 2 i 4 wt%), zajedno s 2 wt% heksametildisilazana (HMDS). Za lijepljenje slojeva drva i gume rabljena su komercijalna urea-formaldehidna (UF) smola i metilen difenil diizocijanat (MDI). Zatim su proučavana mehanička svojstva tako dobivenih laminata uključujući modul loma (MOR), modul elastičnosti (MOE), udarnu čvrstoću (IS), otpornost na zasijecanje (HS) i fizička svojstva kao što su gustoća (D), upijanje vode (WU) i debljinsko bubrenje (TS). Rezultati su pokazali da je povećanje sadržaja WTR-a pridonijelo poboljšanju fizičkih svojstava dobivenih ploča (D, WU i TS), dok je negativno utjecalo na njihova mehanička svojstva (MOR, MOE, IS i HS). Međutim, dodatkom nano-SiO$_2$ poboljšana su fizička i mehanička svojstva ploča (MOR, MOE i HS). Nadalje, uočeno je da su mehanička svojstva poboljšana povećanjem broja slojeva bukovine, iako su ploče od bukovine upijale manje vode u usporedbi s pločama od drva johe i topole. Općenito, poboljšanje fizičkih svojstava ploča slijedilo je načelo: raspored slojeva gume > sadržaj nano-SiO$_2$ > slojevi furnira.

KLJUČNE RIJEČI: *nanočestice silicija; gumeni otpad; furnirska ploča; fizikalno-mehanička svojstva; podovi*

1 INTRODUCTION
1. UVOD

The world has agreed to move from a linear to a circular economy. This has led to various inventions, new business models, and strategies. They support the push for further institutionalization of circular economy practices. Following globalization, more waste is produced, surpassing the earth's capacity for renewal and natural resilience (Greer *et al*., 2021). For example, it is estimated that the annual production of waste in the world will reach 50-70 % by 2050 (Kaza *et al*., 2018). Thus, it is of great interest to considerably reduce the amounts of produced waste and to develop alternative strategies to manage wide spectra of wastes considering the attitudes of the circular economy. One of the materials that offer a lot of opportunities for recycling and reuse is rubber. Currently, rubbers are mainly being used to fabricate a wide range of materials for numerous applications like toys, automotive, technical, construction and structures, etc. However, rubber does not degrade at the end of its life and can remain in the environment for decades, predominately due to the presence of reinforcing fillers, antioxidants, antiozonants, and curating agents (Fazli and Rodrigue, 2020). The landfilling and combustion are not in accordance with the circular economy principles or with the waste management hierarchy (Pires and Martinho, 2019). Therefore, it is potentially valuable to develop alternative strategies for the reuse and recycling of rubber. One of the possible alternatives is wood-rubber composites (Shao *et al*., 2016). The idea of these composites is to combine wood with superior mechanical properties and a good density to strength ratio (Madsen and Gamstedt, 2013) with rubber. On the other hand, the benefits of the rubber are high compressive performance, good water sorption properties, absorption of damping vibration reduction, unique energy absorption, good toughness, strong abrasion resistance, low biodegradability, etc (Mancel *et al*., 2022). Therefore, producing composites based on wood and rubber could have superior properties (Ayrilmis *et al*., 2009). There are various approaches how to produce wood-rubber composites. Wood can be used as a filler in the rubber (Nuzaimah *et al*., 2018; Flour *et al*., 2004; Vladkova *et al*., 2004; Vladkova *et al*., 2006), and rubber can be used in particleboards (Mancel *et al*., 2022) or oriented strand boards (Ayrilmis *et al*., 2009a). The last but not least alternative is to use rubber as a layer between the veneers in plywood. In such applications, rubber can even serve as an adhesive (Shao *et al*., 2016). Ashori *et al.* (2015) investigated the possibility of using waste tire rubber in fabricating hybrid plywood composite panels. The results indicate that utilizing rubber in the produced panels enhanced physical properties such as water uptake (*WU*), thickness swelling (*TS*), and sound absorption. However, mechanical properties like modulus of rupture (*MOR*), modulus of elasticity (*MOE*), and impact strength (*IS*) were declined. Recently, nanotechnology has improved the efficiency of materials in different areas of science. Among nanoparticles, nano-silica (SiO_2) with very small sizes and high surface area are widely used in the polymer and surface coating industries (Fang *et al*., 2014; Xiong *et al*., 2021). Plenty of studies have focused on the application of nanosilica in numerous polymers. However, only a few studies refer to wood/rubber and wood/plastic composites. It has been reported that nano-SiO_2 can improve polymer matrix mechanical properties such as strength, hardness, modulus of crystallinity as well as viscosity and adhesion (Parvinzadeh *et al*., 2010). Deka and Maji (2013) investigated the influence of nanoclay and nano-SiO_2 on the properties of wood/plastic composites. The results showed that mechanical properties, thermal properties, and flame retarding were improved by increasing nanoparticles by up to 3 wt% of each nanoparticle. The impact strength of wood /plastic composite decreased with the increase of clay and nano-SiO_2 (3 and 4 wt%) (Nourbakhsh *et al*., 2011). Xu and Zhang (2021) investigated the preparation and properties of hydrophobically modified nano-SiO_2 nanoparticles with hexadecyltrimethoxysilane (HDTMS). Nano-SiO_2 is a common inorganic silicon material. However, its surface is very hydrophilic due to the presence of many hydroxyl groups, which limits its use in some fields. Therefore, in their research, they used HDTMS to modify nano-SiO_2. The results of water contact angle analysis (WCA), Fourier transform infrared (FTIR), two-dimensional correlation spectroscopy (2D-COS), thermal analysis (TG), and scanning electron microscopy (SEM) showed that HDTMS was successfully grafted on the nano-SiO_2 surface due to its long hydrophobic alkyl chain. In general, their results showed that, when the ratio of nano-SiO_2 and HDTMS was 0.25:1, the WCA reached 170.9°, which was about 5.62 times higher than before the modification. In fact, it showed the property of superhydrophobicity (Xu and Zhang, 2021).

This research attempted to develop a more sustainable and innovative rubber-based plywood by optimizing its composition, employing structural engineering principles, and incorporating additional nano-SiO_2. By utilizing advanced technology and innovative materials, this research aims to contribute to the growing field of sustainable building materials and promote the adoption of environmentally friendly alternatives in the construction industry.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

Rotary-cut veneers of beech (*Fagus orientalis*), alder (*Alnus glutinosa*) and poplar (*Populus alba* L.) with densities of 0.68 g/cm³, 0.49 g/cm³ and 0.40 g/cm³, respectively, were used for the work. The mentioned wood species are the most dominant species used for plywood manufacturing in Iran. A total of 288 defect-free veneer samples with a dimension of 400 mm × 400 mm and a thickness of 1.8 mm were conditioned to a moisture content of 10 % prior to experiments. The chemical materials used, their characteristics and suppliers are listed in Table 1 for easy reference.

2.2 Methods
2.2. Metode

2.2.1 Preparation of nanoparticle soluble polymer
2.2.1. Priprema polimera s otopljenim nanočesticama

A combined solution of nano-SiO_2 and HMDS was used to reinforce the rubber powder filler (Figure 1). In fact, distilled water and ethanol were used as solvents in the reaction, crosslinking and providing proper stabilization of the silica particles. To prepare the nanoparticle solution with suitable concentrations, nano-SiO_2 powder with weight ratios of 0, 2, and 4 wt% and HMDS with 2 wt% dry weight ratio of rubber powder were added to 150 L of a mixture of distilled water and ethanol and placed in a magnetic electric mixer and combined for 2 h. The combined solution of nano-SiO_2 and HMDS was then placed in a drying chamber (laboratory dryer) at 8 °C for 8 h to obtain a polymer nanoparticle solution. Finally, the generated residues were mixed as a dry powder tablet and mixed with rubber powder (Song *et al*., 2014).

2.2.2 Hybrid plywood composite fabrication
2.2.2. Izrada kompozita od hibridne furnirske ploče

Table 2 summarizes the features of some seven-layer plywood samples, comprising a combination of beech (B), alder (A), and poplar (P) veneers with rubber particles. Plywood panels were made using a wide range of permutations across four independent variables: layering pattern, WTR content, nano-SiO_2 content, and hot-

Table 1 Chemical materials used in the study
Tablica 1. Kemikalije upotrijebljene u istraživanju

Chemical material *Kemikalija*	**Characteristics** *Svojstva*	**Supplier (Country)** *Dobavljač (država)*
Waste tire powder (WTP) *prah od otpadnih guma*	Bulk density / *nasipna gustoća*: 0.32 g/cm³ Particle size / *veličina čestica*: 0.2 mm	Isatis Yazd (Iran)
nano-SiO_2	Bulk density / *nasipna gustoća*: <0.10 g/cm³ True density / *stvarna gustoća*: 2.4 g/cm³ Particle size / *veličina čestica*: 20-30 nm Purity / *čistoća*: > 99 %	Evonik Industry (Germany)
Hexamethyldisilazane (HMDS) *heksametildisilazan* (HMDS)	Bulk density / *nasipna gustoća*: 0.77 g/cm³ Purity / *čistoća*: > 97 % Boiling point / *vrelište*: 126 °C	Tokyo Chemical Industry (Japan)
Ethanol / *etanol*	Bulk density: 0.79 g/cm³ Purity / *čistoća*: > 99 % Boiling point / *vrelište*: 78.5 °C	Scharlau Chemical Industry (Spain)
Methylene diphenyl diisocyanate (MDI) resin *metilen difenil diizocijanat (MDI).*	True density / *stvarna gustoća*: 1.27 g/cm³ Solid content / *sadržaj suhe tvari*: 100 %	Aras Chemicals (Iran)
Urea formaldehyde (UF) resin *urea-formaldehidna (UF) smola*	True density / *stvarna gustoća*: 1.26 g/cm³ Solid content *sadržaj suhe tvari*: 60 %	Iran-Choob Ghazvin (Iran)

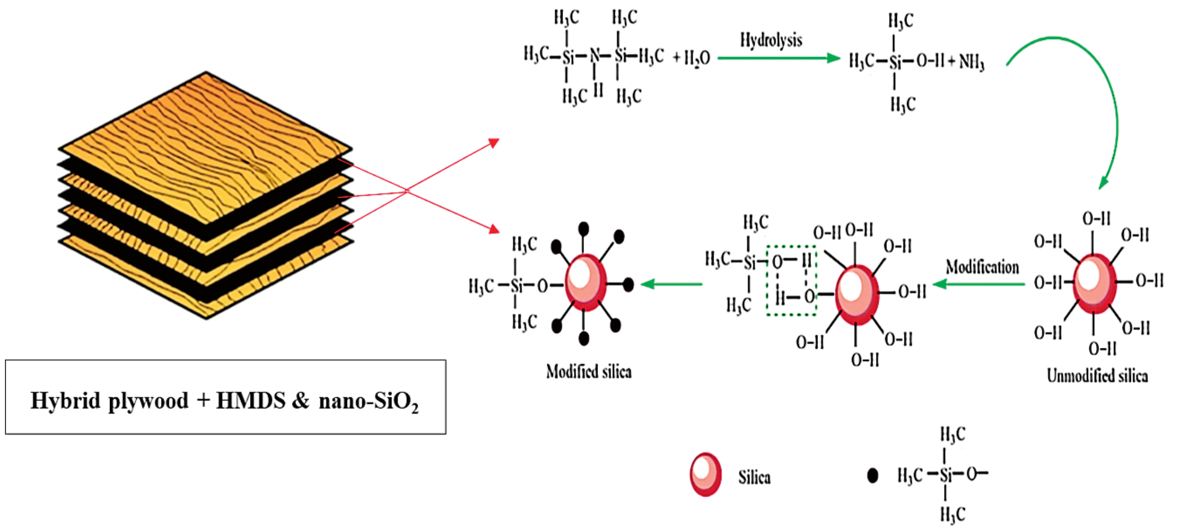

Figure 1 Schematic diagram showing influence of nano-SiO2 modified by HMDS on bonding of rubber between wood layers (Song *et al*., 2014)
Slika 1. Shematski dijagram utjecaja nano-SiO_2 čestica modificiranih HMDS-om na lijepljenje gume između slojeva drva (Song *et al*., 2014.)

pressing method (Table 2). The veneers were cold-pressed for 10 minutes after being bonded with UF resin at a glue spread rate of 160 g/m^2. It should be noted that MDI glue spread rate was 150 g/m^2. The plywood was produced using a two-stage hot-pressing technique under controlled conditions. To begin, a molding frame (400 mm × 400 mm) was filled uniformly with resin rubber powder. The particles served as the panel core or intermediate layer after being subjected to high temperatures during the pressing process. The nominal thickness of these rubber layers was 3 and 5 mm and the density 0.9 g/cm^3. As a result, the rubber layer and wood veneers were pressed for 10 min at 145 °C and 5 MPa pressure. In order to prepare the hybrid plywood panels for testing, they were conditioned for 7-10 days in a conditioning room with a relative humidity of 65 % and a temperature of 25 °C. Conditioned panels have a more consistent moisture levels and fewer drying strains. Three identical plywood panels were made for each group. In addition, each veneer layer was 1.8 mm thick, the thickness of the core layer of the rubber was 5 mm and its two sides were 3 mm.

2.3 Physical properties
2.3. Fizička svojstva

Density (*D*), water uptake (*WU*), and thickness swelling (*TS*) of each specimen were measured as physical parameters according to EN 323 standard and EN 317 standard, respectively. To condition the composites, the samples were submerged in distilled water at room temperature for 2 and 24 h. After drying, their weight and thickness were re-measured. The samples were immediately measured to the nearest 0.001 mm and weighed to the nearest 0.01 g. All derived values were obtained as the median of six independent calculations.

Table 2 Design of plywood sets
Tablica 2. Dizajn uzoraka furnirske ploče

Type *Vrsta*	**Sheet set** *Sklop listova*	**Layers model** *Raspored slojeva*	**Description** / *Opis*	**Total thickness, mm** *Ukupna debljina, mm*	**Construction** *Konstrukcija*
Hybrid plywood (PWH) *hibridna furnirska ploča*	4-Ply/3-R	VA/R/V/R/V/R/VA VB/R/V/R/V/R/VB VP/R/V/R/V/R/VP	Veneer (V)/Rubber Layer (R)/ Veneer (V)/Rubber Layer (R)/ Veneer (V)/Rubber Layer (R)/ Veneer (V)	18.2	V-R-V-R-V-R-V
Core plywood (PWC) *furnirska ploča s jezgrom*	6-Ply/1-R	3VA/R/3VA 3VB/R/3VB 3VP/R/3VP	3Veneer (V)/1Rubber Layer (R)/3Veneer (V)	15.8	V_3-R-V_3
Plywood veneer (PWV) *furnirska ploča*	7-Ply	REA REB REP	Reference Plywood (Beech-Alder-Poplar)	12.6	V_7

Table 3 Properties, dimensions, and number of composite hybrid plywood samples
Tablica 3. Svojstva, dimenzije i broj uzoraka kompozitne hibridne furnirske ploče

Property test *Ispitivano svojstvo*	**Dimensions**, mm *Dimenzije*, mm	**Number** *Broj uzoraka*	**Reference standard** *Referentni standard*
Density (D) / *gustoća (D)*	50×50	6	EN 323:1993
Water uptake (WU) and thickness swelling (TS) *upijanje vode (WU) i debljinsko bubrenje (TS)*	50×50	6	EN 317:1993
Flexural strength (FS) and flexural modulus (FM) *čvrstoća na savijanje (FS) i modul elastičnosti pri savijanju (FS)*	282×50 346×50	6	ASTM-D 790:2002
Impact bending strength (IBS) / *čvrstoća na udar (IBS)*	280×20	6	DIN 52189:1981
Hardness strength (HS) / *otpornost na zasijecanje (HS)*	50×50	6	EN 1534:2010

2.4 Mechanical properties
2.4. Mehanička svojstva

The samples were conditioned at 20 °C and 65 % relative humidity, and then their modulus of rupture (MOR) and modulus of elasticity (MOE) were measured using ASTM-D 790 standard. At a span-to-depth ratio of 20:1, flexural tests were performed using the third point loading method. Universal testing equipment (Zwick/Roell Z150, Germany) was used to apply a load to the specimen at a strain rate of 4 mm/min. The hardness strength (*HS*) and the impact bending strength (*IBS*) of the uncut composite were measured using a pendulum impact tester. Six replications were done for each type of plywood panel. Table 3 lists the dimensions and number of composite hybrid plywood samples used for physical and mechanical tests.

2.5 Statistical analysis
2.5. Statistička analiza

In a completely random setup, n=3 samples were analyzed using the statistical software SPSS to see what happened (version 20.0). The analysis of variance (ANOVA) and Duncan's Multi-Range test (at a 99 % confidence level) were used to compare the means and standard deviations of the groups.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Physical properties
3.1. Fizička svojstva

3.1.1 Density (*D*)
3.1.1. Gustoća (*D*)

According to the ANOVA results, the density of the plywood/rubber samples increased significantly with the addition of 4 wt% nano-SiO_2 (99 % confidence level; Table 1). Duncan's test showed that this treatment had the highest density among all groups, while the plywood samples had the lowest density (Supplement Table 2 and 3). The pressure in the hot press and wood species considerably impacts the density of plywood. The highest density was observed for beech plywood (0.61 g/cm³),

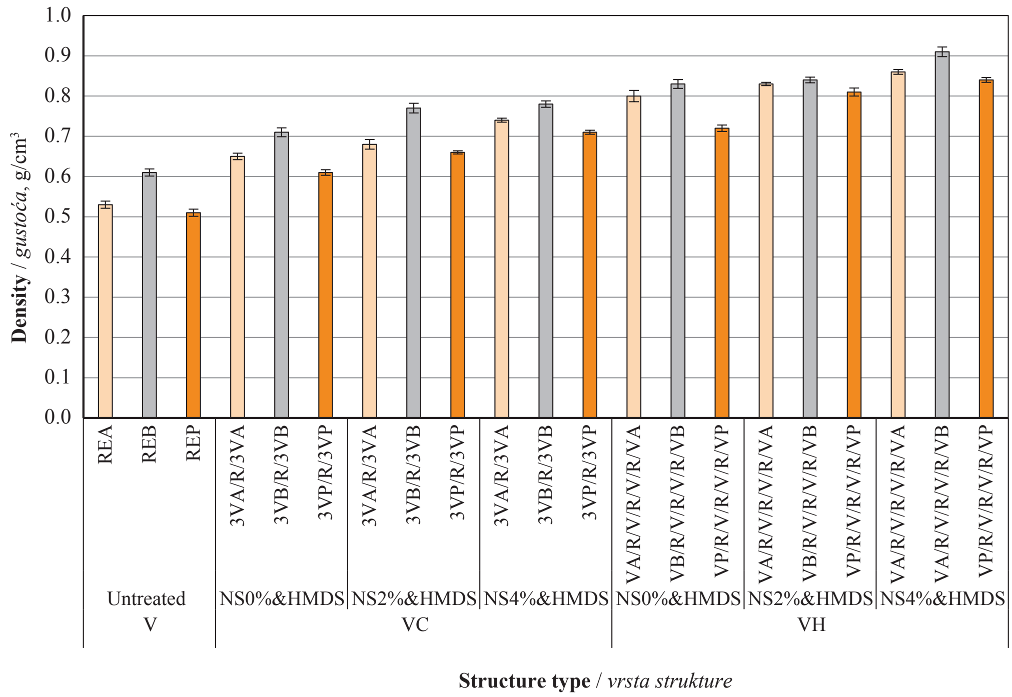

Figure 2 Density of composite plywood with different structures
Slika 2. Gustoća kompozitne furnirske ploče s različitim strukturama

Table 4 Physical-mechanical properties (Mean (± SD)) of plywood used
Tablica 4. Fizičko-mehanička svojstva (srednja vrijednost, ± SD) korištene furnirske ploče

Type of plywood panels (RE) *Vrsta furnirske ploče (RE)*	**Properties** / *Svojstva*									
	D, g/cm³	*WU*, %		*TS*, %		*FS*, MPa	*FM*, MPa	*IBS*, kJ/m²	*HS*, MPa	
		2 h	24 h	2 h	24 h				5.5 mm	7 mm
Alder *johovina*	0.53 (±0.008)	37.61 (±0.21)	56.26 (±1.25)	11.75 (±0.14)	13.65 (±0.30)	76.80 (±1.20)	7933.33 (±26.58)	53.20 (±0.29)	46.58 (±0.32)	56.31 (±0.20)
Beech *bukovina*	0.61 (±0.008)	26.57 (±0.32)	47.65 (±0.30)	12.34 (±0.17)	16.69 (±0.26)	87.71 (±1.62)	9666.66 (±22.50)	64.73 (±0.22)	65.65 (±0.32)	76.53 (±0.29)
Poplar *topolovina*	0.51 (±0.008)	33.35 (±0.32)	51.75 (±0.15)	9.83 (±0.15)	12.59 (±0.31)	67.69 (±1.76)	7858.33 (±23.16)	46.39 (±0.34)	42.64 (±0.26)	51.52 (±0.22)

followed by alder (0.53 g/cm³) and poplar (0.51 g/cm³) (Table 4). The effect of the amount of nano-SiO_2, and the arrangement or alternation of the rubber layer and the wood layer on the density is shown in Figure 2.

As seen in Figure 2, the lowest density of plywood/rubber samples in three layers of beech, alder, and poplar is related to wooden layers with 0 wt% nano-SiO_2 content and rubber core layer, while the highest density of plywood/rubber samples were observed in three layers of beech, alder, and fir, corresponding to wooden layers with 4 wt% nano-SiO_2 content and side layers of rubber. The density of rubber ranges from 0.9 g/cm³ to 1 g/cm³ (Cheremisinoff, 2023). High density (2.6 g/cm³) and proper permeability of nano-SiO_2 increase the specific surface area (specific weight) of the produced composites (Mahzan *et al.*, 2010). The high density of mineral nanoparticles could be attributed to their high atomic level since a large number of atoms and the regular arrangement of mineral nanoparticles generally improve the physical characteristics (density) of the composite (Savov *et al.*, 2023). As can be seen, the density of the beechwood layer is higher than that of alder and poplar. The higher density of the beech layer compared to the alder and poplar layer can be related to its high density and low porosity (Ashori *et al.*, 2015; Toksoy *et al.*, 2006; Ilkay and Mengeloglu, 2022). So, the greater the porosity of the wood layer, the weaker the wood, leading to the decrease of the surface layer (Rodríguez *et al.*, 2014). Rubber as a polymer filler affects the density of composites. It seems that the low porosity, small molecular structure (macromolecular structure), and compactness of rubber particles can improve the specific mass (specific weight) of composites (Jun *et al.*, 2008; Xu and Li, 2012). The density of wood layers is around 0.40-0.68 g/cm³. Therefore, as the weight percentage of rubber powder increases, the density and porosity of the composites increase and decrease, respectively (Jun *et al.*, 2008).

3.1.2 Water uptake (*WU*)
3.1.2. Upijanje vode (*WU*)

The results obtained from the ANOVA indicated that, with the increase in the amount of nano-SiO_2 (4 wt%), the amount of water uptake increased significantly in 2 and 24 h (statistical confidence level 99 %; attached Table 1). Also, Duncan's test placed this state of combined treatment in group C with the highest amount of water uptake at 2 and 24 h compared to plywood/rubber samples. But in general comparison, the highest amount of water uptake in 2 and 24 h was observed in the control plywood samples, and Duncan's grouping placed this condition in group A (Supplement Table 2 and 3). The interaction of nano-SiO_2 content, arrangement, or rotation of rubber layer and wood layer on the water uptake (2 and 24 h) of wood and rubber layered composites is shown in Figure 3.

Water uptake of the pure plywood reflects the density. As the water uptake is expressed in percentages, it appears that wood with a lower density absorbs more water. However, although the plywood uptakes the same amount of water expressed in mass, the relative uptake expressed in percentages will be higher for the plywood with lower density. This phenomenon could explain the differences in water uptake between various wood-rubber composites. As expected, the composites that contain more rubber (V/R/V/R/V/R/V) uptake less water during immersion than the pure plywood. This phenomenon can be explained by the presence of hydrophobic rubber, the higher density of these composites, and the thickness of the exposed wood layer. For example, the outer layer of the composite 3V/R/3V is much thicker than the composite V/R/V/R/V/R/V; thus, the thicker outer layer can absorb more water during immersion than the thinner outer layer. In addition, the water uptake was influenced by the presence of the nano-SiO_2. Regardless of the wood type and the composition type, the best performance was determined by composites that contain 2 wt% of nano-SiO_2. The best combination was usually associated with beech wood. However, as mentioned, this cannot only be ascribed to better performance, but could also be the result of the differences in density. The results showed that nano-SiO_2 increases water uptake of the composite plywood. Since the surface of the nano-SiO_2 molecule has three functional groups (hydroxy, hydrogen attached to hydroxy groups (OH), and siloxane

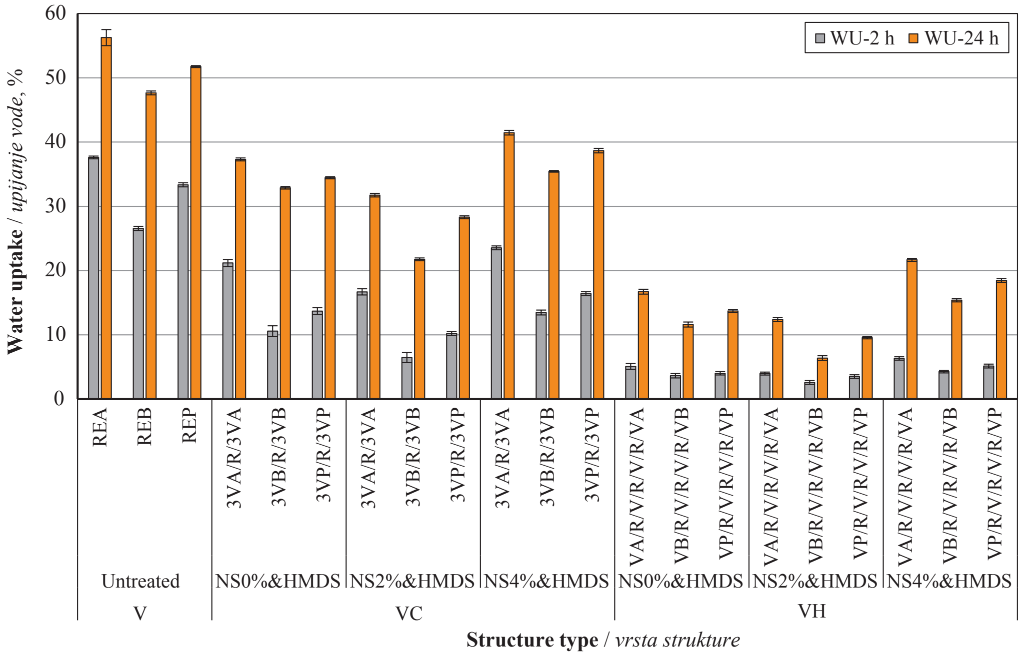

Figure 3 Water uptake (%) of composite plywood with different structures
Slika 3. Upijanje vode (%) kompozitne furnirske ploče s različitim strukturama

groups), silica particles are hydrophilic. In a sense, when the samples are placed in water, the nano-SiO_2 with very large lateral surfaces absorb water molecules and thus increase the amount of water uptake. Therefore, with the increase of nano-SiO_2 from 0 to 4 wt%, the amount of water uptake also increases, which can be due to the very high specific surface area of nano-SiO_2 particles and the hydrophilicity of their surface (Homkhiew *et al*., 2015). Also, the results of the amount of water uptake in 24 h showed that the amount of water uptake of the alder layer is higher than that of poplar and beech. However, generally, the amount of water uptake of poplar, alder, and beech layer with rubber side layer and 0 and 2 wt% nano-SiO_2 was lower than that of the control plywood.

3.1.3 Thickness swelling (*TS*)
3.1.3. Debljinsko bubrenje (*TS*)

The results obtained from the ANOVA showed that, with the increase in the amount of nano-SiO_2 (4 wt%), the amount of thickness swelling in 2 and 24 h increased significantly (statistical confidence level 99 %; attached Table 1). Also, Duncan's test placed this state of combined treatment in group C with the highest amount of thickness swelling at 2 and 24 h compared to plywood/rubber samples. But in general comparison, the highest amount of thickness swelling in 2 and 24 h was observed in the control plywood samples, and Duncan's grouping placed this condition in group A (Tables 4 and 5). Figure 4 depicts the expansion in the thickness of a wood and rubber layered composite after 2 and 24 h of immersion in water.

The visible growth in thickness agrees with the hydration information. Higher water uptakes result in higher thickness swelling. The highest thickness swelling was related to beech plywood (REB). This result is expected considering that beech is a wood species characterized by low dimensional stability (Wagenführ, 2014). The presence of rubber (R) has a positive effect on thickness swelling. First, because rubber limits water uptake, and second, because rubber absorbs part of the dimensional changes. However, there was an insignificant difference between the two respective types of composites. Both composites, 3V/R/3V and V/R/V/R/V/R/V, perform comparably. Similarly, as reported for water uptake, nano-SiO_2 has the most positive effect on a concentration of 2 wt%. Higher concentrations of nano-SiO_2 adversely affected the thickness swelling, which can be associated with higher water uptake. By increasing the amount of nano-SiO_2 up to 2 wt% level, some empty spaces and capillary tubes are occupied, which could result in a sharp reduction of these openings and make it difficult for water molecules to reach the wood material. Therefore, increasing the amount of nano-SiO_2 causes fewer water molecules to be absorbed by the wood material, resulting in reduced thickness swelling after 2 and 24 h (Kariminejad *et al*., 2022). Rubber is very effective in reducing water uptake and thickness swelling. The decrease in water uptake and thickness in the side layer of the rubber is a consequence of the increase in the amount of rubber per unit volume of these panels and the water repellency of this material. Applying rubber for constructing multi-structures also showed a decrease in water up-

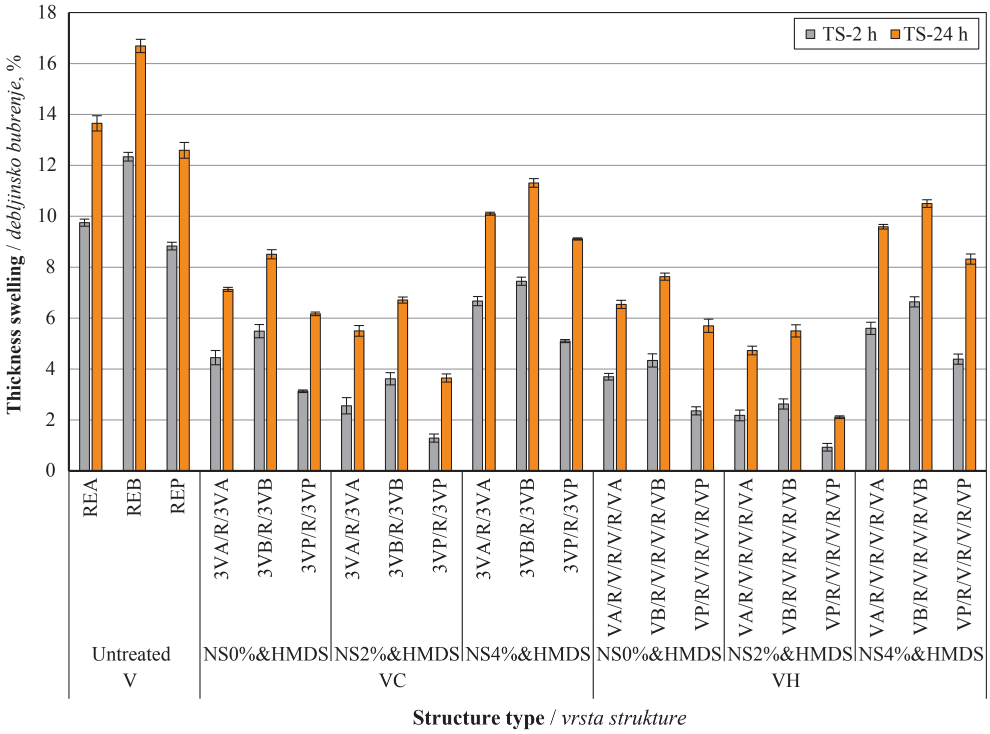

Figure 4 Thickness swelling (%) of composite plywood with different structures
Slika 4. Debljinsko bubrenje (%) kompozitne furnirske ploče s različitim strukturama

take and the thickness of these multi-structures with an increase in the ratio of rubber to wood (Mancel *et al.,* 2022; Ashori *et al.*, 2015; Ashori *et al.*, 2018). The low moisture absorption of polymeric materials (rubber) could lead to low moisture absorption of cellulose materials of composites. Therefore, by increasing the amount of rubber in the unit volume of multi-structures, the amount of water uptake and thickness swelling decreases (Ashori *et al.*, 2015; Ashori *et al.*, 2018).

3.2 Mechanical properties
3.2. Mehanička svojstva

3.2.1 Flexural strength (*FS*) and flexural modulus (*FM*)
3.2.1. Čvrstoća na savijanje (*FS*) i modul elastičnosti pri savijanju (*FM*)

The combined treatment of plywood/rubber samples did not significantly affect their modulus of elasticity (*MOE*) and modulus of rupture (*MOR*) at the 99 % confidence level, according to the ANOVA results (Table 1). Duncan's test indicated that the control plywood samples had the highest *MOE* and *MOR* among all groups, while the plywood/rubber samples with beech, alder and poplar layers, rubber side layer, and 0 wt% nano-SiO_2 content had the lowest *MOE* and *MOR* (Supplement Table 2 and 3). Adding rubber to the laminate flooring composite negatively impacted the flexural strength. Rubber is known for its elastic properties, which contributes to the decrease in *MOE* and *MOR*. As the wood is positioned in the outer layer of the composite plywood, which has the most considerable effect on the bending strength, the presence of rubber insignificantly influenced the *MOE* and *MOR* mechanical properties. Regardless of the composition, the highest *MOE* and *MOR* were reported for the composites based on beech wood. This result is reasonable, as beech wood has much better mechanical properties than alder and poplar, predominately due to higher density. In addition, the positive effect of the nano-SiO_2 is evident as well. *MOE* and *MOR* of the composites with higher concentrations of nano-SiO_2 are considerably better than those of the parallel composites without respective additives (Figure 5). The mechanical resistance of wood/plastic composites (*MOE* and *MOR*) is improved by increasing the content of nanoparticles (Deka and Maji, 2012). This occurs because an increase in nanoparticles leads to a greater dispersion of nanoparticles in the polymer matrix. As silica is spherical, it may be evenly distributed within the poly framework, where it will fill the voids between the wood fibers and the polymer matrix to produce a dense feel. As the composite density rises and its brittleness increases, the *MOE* and *MOR* rise as well (Moezzipour *et al.*, 2013; Mohseni Tabar *et al.*, 2015). Some mechanical qualities of composites can be modified by adjusting the rubber-to-wood weight ratio. Both *MOE* and *MOR* go down as the rubber-to-wood weight ratio rises (Ashori *et al.*, 2015; Ashori *et al.*, 2018). Rubber, as a filler and cross-linking agent in composites, improves

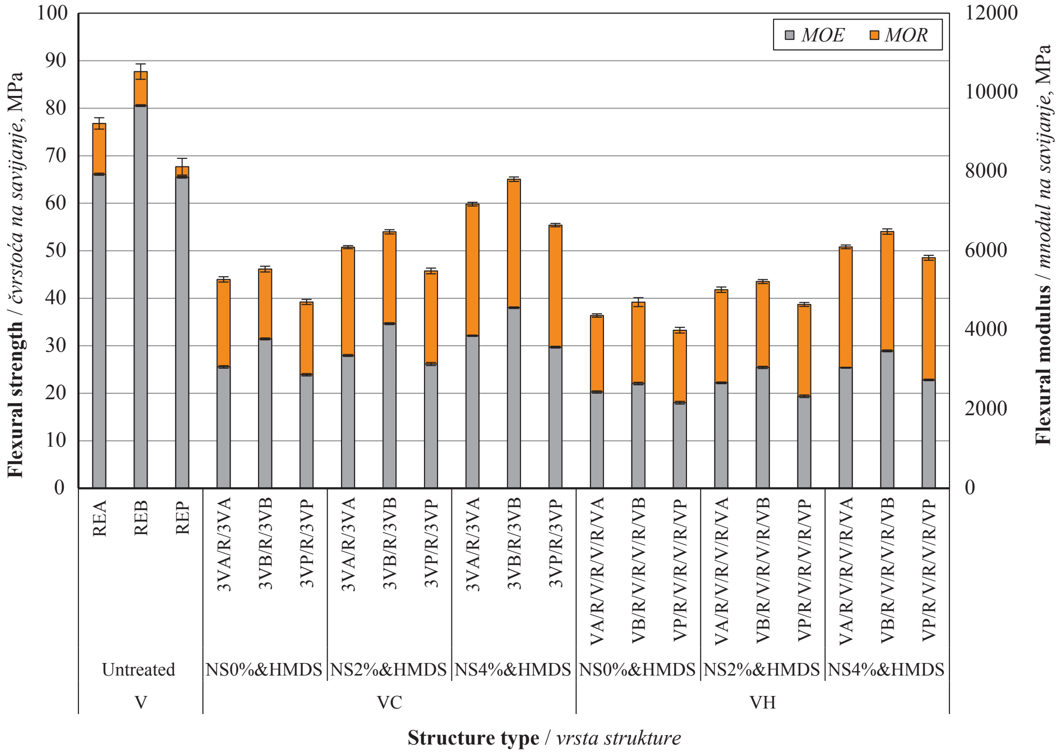

Figure 5 Flexural modulus and flexural strength of composite plywood with different structures
Slika 5. Modul elastičnosti pri savijanju i čvrstoća na savijanje kompozitne furnirske ploče s različitim strukturama

MOE and *MOR* (Ashori *et al*., 2018; Jayadevan *et al*., 2018; Cosnita *et al*., 2014) despite its relatively modest amount compared to wood. The small molecular structure of rubber, or the small size of rubber particles, contributes to an increase in the specific surface area of rubber and a decrease in mechanical resistance (*MOE* and *MOR*) of composites (Ashori *et al*., 2015).

3.2.2 Impact bending strength (*IBS*)
3.2.2. Udarna čvrstoća (*IBS*)

The ANOVA results showed that the combined treatment of plywood/rubber samples did not have a significant effect on their impact bending strength (*IBS*) at 99 % confidence level (Supplement Table 1). Duncan's test revealed that the control plywood samples had the highest IBS among all groups, while the plywood/rubber samples with beech, alder, and poplar layers, 4 wt% nano-SiO_2, and rubber side layer had the lowest *IBS* (Supplement Table 2 and 3). The results of the *IBS* are in line with the bending tests. Among plywood samples, the highest IBS was determined for beech plywood. This is predominately the result of the beech higher density compared to the other two hardwood species. Consequently, all composites that were based on beech wood had higher *IBS* in comparison to other composites based on poplar and alder. In addition, the composites with structure 3V/R/3V perform better than the ones with structure V/R/V/R/V/R/V, which is likely due to the fact that the outer layer of the composites 3V/R/3V was more rigid than the other one. In addition, it can be seen that the addition of the additive nano-SiO_2 reduces the IBS (Figure 6). With the increase of nanoparticles, the impact resistance decreases (Nourbakhsh and Ashori, 2009; Yao *et al*., 2015). In fact, nano-SiO_2 particles tend to attract each other because of their hydrophilic hydroxyl groups. In other words, nano-SiO_2 particles have high surface energy because hydroxyl groups can cause accumulation on the surface of the polymer by generating hydrogen bonds, leading to a decreased impact resistance (Elbarbary, 2022).

3.2.3 Hardness strength
3.2.3. Otpornost na zasijecanje

The ANOVA results showed that the combined treatment of plywood/rubber samples did not have a significant effect on their hardness strength at the 99 % confidence level (Supplement Table 1). Duncan's test revealed that the control plywood samples had the highest hardness strength among all groups, while the plywood/rubber samples with beech, alder and poplar layers, 4 wt% nano-SiO_2, and rubber side layer had the lowest hardness strength (Supplement Tables 2 and 3). Surface hardness is predominately affected by the hardness of the outer layer. Beech was the hardest among all tested wood species. Hence, the beech-rubber composites performed better than the ones based on poplar- and alder-rubber composites. As the outer layer of the rubber-wood composites, 3V/R/3V was

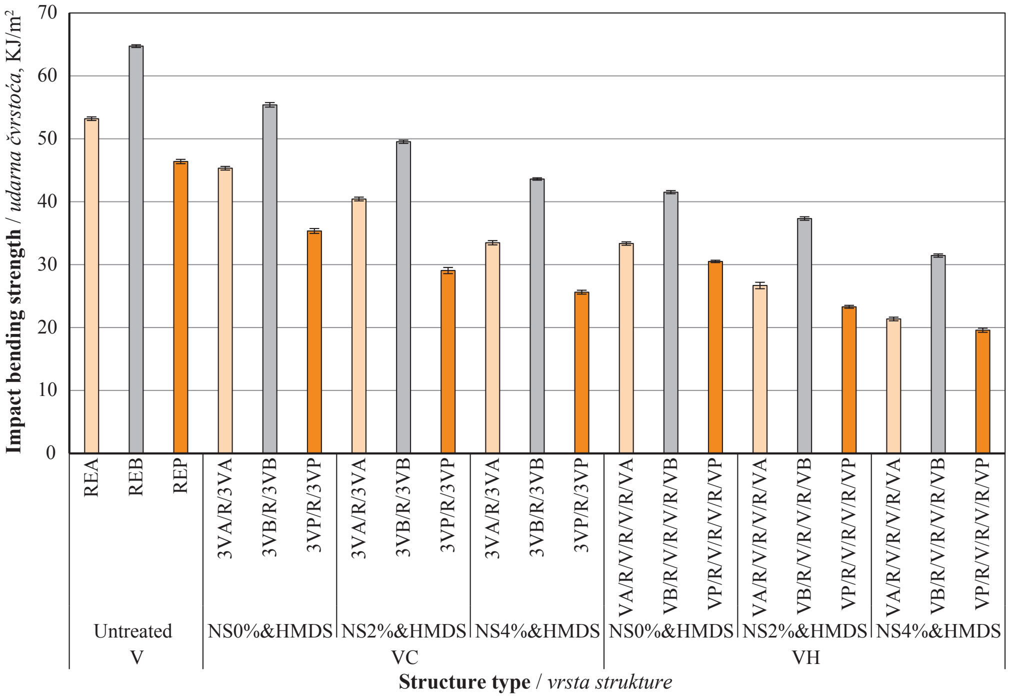

Figure 6 Impact bending strength of composite plywood with different structures
Slika 6. Udarna čvrstoća kompozitne furnirske ploče s različitim strukturama

thicker than V/R/V/R/V/R/V, resulting in higher hardness. The outer layer of the composite V/R/V/R/V/R/V was too thin to resist the penetration of the ball. The presence of the additive nano-SiO_2 has a similar effect as that of water. Middle concentration (2 wt%) has a positive effect, while the highest concentration of nano-SiO_2 did not contribute to the hardness (Figure 7). The higher specific surface ratio of rubber to wood reduces the adhesion between wood and glue; therefore, in addition to reducing the surface adhesion between wood layers, the mechanical characteristics (impact and hardness strength) of the composite are also re-

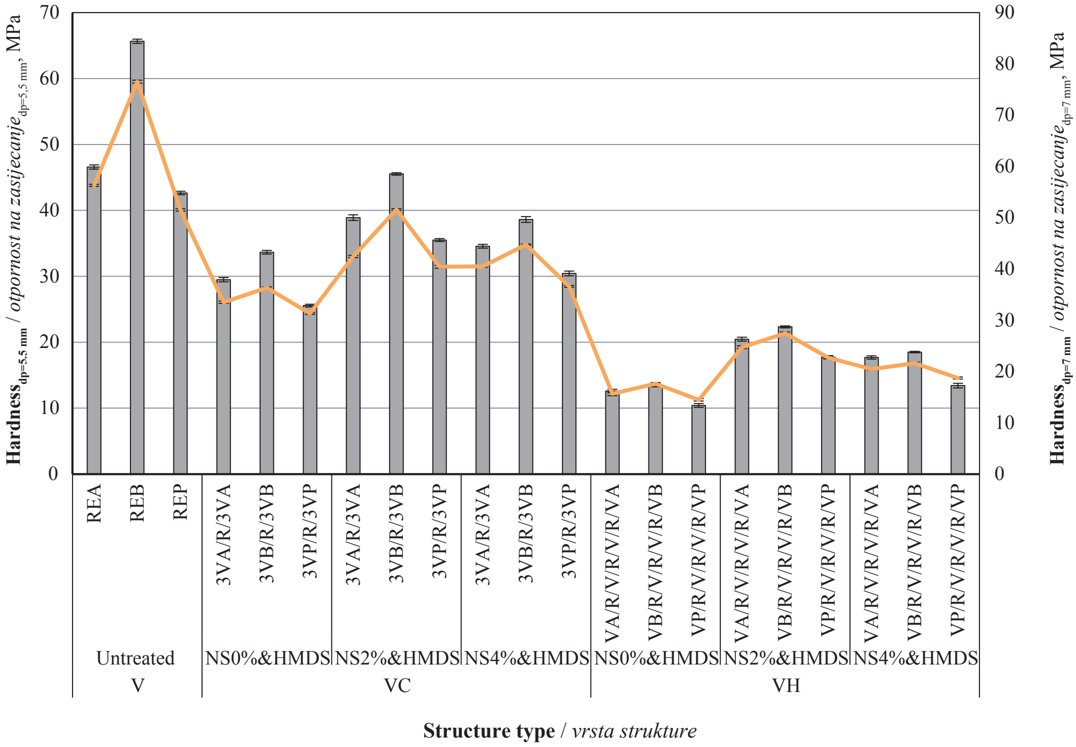

Figure 7 Surface hardness strength of composite plywood with different structures
Slika 7. Otpornost na zasijecanje kompozitne furnirske ploče s različitim strukturama

duced (Ashori *et al.*, 2015). The hardness strength of the composite is different in different penetration depths, so the hardness level increases with the increase of the penetration depth of the bullet (Taghiyari and Norton, 2014). The increase in hardness with a higher penetration depth compared to a lower penetration depth can be related to the greater penetration of the diameter of the bullet to the surface and finally the higher strength of the surface of the composite (Taghiyari and Norton, 2014).

4 CONCLUSIONS
4. ZAKLJUČAK

The objective of this study was to use recycled wood and rubber in the production of soft and strong composite flooring and also to investigate the physical-mechanical properties of the resulting multi-layered flooring. The results of this investigation showed that the three factors examined in this research, including the amount of modified nano-SiO_2, the wood layer, and the frequency or arrangement of the rubber layer, have a substantial effect on the physical and mechanical resistances (*D*, *WU* and *TS* in 2 and 24 h, *MOR* and *MOE*, *IBS*, and *HS*). In investigating the effect of the modified nano-SiO_2 loading, it was found that the density of the composites has increased. The amount of water uptake and thickness swelling after 2 and 24 hours of immersion in water showed that nano-SiO_2 did not have a meaningful effect on reducing water uptake and thickness swelling. Nano-SiO_2 had an effect on the improvement of *MOE*, *MOR* and *HS*, but it did not have a decisive effect on the improvement of *IBS*. The density of the wood layer had a great effect on the physical and mechanical resistance. So the wood layer with higher density enhanced the physical and mechanical resistance compared to the wood layer with lower density. However, in thickness swelling, wood layers of high density had more swelling; in other words, the thickness swelling of heavier wood layers was more pronounced than that of wood layers of low density. The arrangement of the rubber layer had a significant effect on increasing the physical properties, but it did not have a significant effect on the mechanical properties. Finally, it can be concluded that the use of modified nano-SiO_2 has the ability to improve some physical (density) and mechanical properties (*MOR*, *MOE*, and *HS*), and it can be used to improve the properties of filling materials such as elastic, plastic and other polymer materials.

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SUPPLEMENT – DODATAK

Table 1 ANOVA results for *D, WU, TS, FS, FM, IBS* and *HS*
Tablica 1. Rezultati ANOVA analize za *D, WU, TS, FS, FM, IBS* i *HS*

Factor		**Property** / *Svojstvo*									
		D	*WU*		*TS*		*FS*, MPa	*FM*, MPa	*IBS*, kJ/m^2	*HS*	
			2 h	24 h	2 h	24 h				5.5 mm	7 mm
Type of veneer *vrsta furnira*	*F* value *P* Value	738.54 0.000	1751.81 0.000	4562.09 0.000	983.17 0.000	2250.49 0.000	1521.95 0.000	8887.01 0.000	22130.00 0.000	4465.45 0.000	6237.64 0.000
SiO_2 content *sadržaj* SiO_2	*F* value *P* Value	794.86 0.000	878.43 0.000	12020.93 0.000	2954.74 0.000	9312.61 0.000	8361.89 0.000	6720.87 0.000	10549.14 0.000	8897.46 0.000	18126.34 0.000
Arrangement of layer rubber *raspored slojeva gume*	*F* value *P* Value	5073.97 0.000	15458.97 0.000	131792.98 0.000	374.46 0.000	749.37 0.000	6542.08 0.000	29378.71 0.000	27435.56 0.000	106642.41 0.000	199758.87 0.000
Interaction *interakcija*	*F* value *P* Value	25.68 0.000	1.526 0.201	72.78 0.000	3.798 0.007	6.792 0.000	8.54 0.000	15.38 0.000	30.78 0.000	18.61 0.000	145.25 0.000

Table 2 Duncan's test results for the effect of wood veneer on studied properties
Tablica 2. Rezultati Duncanova testa utjecaja furnira drva na ispitivana svojstva

Type of veneer *Vrsta furnira*	**Property** / *Svojstvo*									
	D, g/cm^3	*WU*, %		*TS*, %		*FS*, MPa	*FM*, MPa	*IBS*, kJ/m^2	*HS*, MPa	
		2 h	24 h	2 h	24 h				5.5 mm	7 mm
Alder / *johovina*	0.76 B	12.80 A	26.88 A	4.19 B	7.27 B	47.25 B	3069.32 B	33.44 B	25.62 B	29.56 B
Beech / *bukovina*	0.81 A	6.84 C	20.58 C	5.03 A	8.36 A	50.32 A	3610.62 A	43.14 A	28.69 A	33.23 A
Poplar / *topolovina*	0.72 C	8.84 B	23.86 B	2.87 C	5.84 C	43.48 C	2799.48 C	27.24 C	22.18 C	27.38 C

Table 3 Duncan's test results for the effect of nano-SiO_2 on studied properties
Tablica 3. Rezultati Duncanova testa utjecaja nano-SiO_2 na ispitivana svojstva

Nano-SiO_2 content, wt% *Sadržaj nano-SiO_2, tež%*	**Property** / *Svojstvo*									
	D, g/cm^3	*WU*, %		*TS*, %		*FS*, MPa	*FM*, MPa	*IBS*, kJ/m^2	*HS*, MPa	
		2 h	24 h	2 h	24 h				5.5 mm	7 mm
NS0	0.72 C	9.70 B	24.45 B	3.91 B	6.95 B	39.69 C	2824.70C	40.24 A	20.87 C	24.83 C
NS2	0.76 B	7.25 C	18.36 C	2.20 C	4.70 C	45.75 B	3115.67 B	34.39 B	30.08 A	34.89 A
NS4	0.80 A	11.53 A	28.52 A	5.97 A	9.82 A	55.61 A	3539.06 A	29.18 C	25.54 B	30.45 B

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Efficiency of European Wood Science and Technology Educational Programmes in Including Green and Digital Topics

Učinkovitost europskih obrazovnih programa o znanosti o drvu i drvnoj tehnologiji u uključivanju zelenih i digitalnih tema

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ABSTRACT • *The integration of sustainable and digital competences in educational programmes is vital for shaping a promising future. Through interviews and analysis, we assessed the inclusion of green and digital (industry 4.0 and ambient assisted living) topics in wood science and technology educational programmes across different European countries. Our research revealed disparities in vertical alignment within countries and deviations among similar programs across countries. With the help of fuzzy logic and by using Data Envelopment Analysis, we evaluated the technical efficiency of programs in incorporating these topics in teaching, considering multiple factors. Results show varying performance levels, with some programs achieving optimal efficiency, while others lagging behind. To improve underperforming programs, prioritizing topic integration is crucial. National coordination and alignment across educational levels are necessary to establish a cohesive system. Equipping individuals with these competences enable them to contribute to sustainable development, leverage digital technologies, and meet societal demands.*

KEYWORDS: *wood science and technology; education; green; digital; DEA*

SAŽETAK • *U izgradnji obećavajuće budućnosti ključnu ulogu ima integracija kompetencija za održivi razvoj i digitalnih kompetencija u obrazovne programe. Putem intervjua i analiza procijenili smo učinkovitost uključivanja zelenih i digitalnih tema (ambijentalno potpomognut život i industrija 4.0) u obrazovne programe o znanosti o drvu i drvnoj tehnologiji u različitim europskim zemljama. Naše istraživanje otkrilo je razlike u vertikalnom usklađivanju unutar zemalja i odstupanja među sličnim programima u različitim zemljama. Uz pomoć neizrazite logike i primjenom analize omeđivanja podataka procijenili smo tehničku učinkovitost programa u uključivanju tih tema u nastavu s obzirom na više čimbenika. Rezultati su pokazali različite razine izvedbi, pri čemu se uvođenjem nekih programa postiže optimalna učinkovitost, a drugi zaostaju za njima. Kako bi se poboljšali programi s lošom*

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izvedbom, najvažnije je dati prioritet integraciji tema. Za uspostavu kohezivnog sustava nužni su nacionalna koordinacija i usklađivanje među obrazovnim razinama. Osposobljavanje pojedinaca za te kompetencije omogućuje im da pridonose održivom razvoju, iskoriste digitalne tehnologije i zadovolje društvene zahtjeve.

KLJUČNE RIJEČI: *znanost o drvu i drvna tehnologija; obrazovanje; zeleno; digitalno; DEA*

1 INTRODUCTION
1. UVOD

The increasing environmental and other challenges have raised significant concerns (European Commision *et al.*, 2016; Campbell-Johnston *et al.*, 2020; Pędzik *et al.*, 2021) prompting countries to develop a range of policies, strategies, and actions, such as the Sustainable Development Goals (SDGs) (Baeyens and Goffin, 2015). In line with these efforts, the European Commission (EC) has identified its primary priorities for the period of 2019-2024, among which it is also the European Green Deal (von der Leyen, 2019). The bioeconomy plays a crucial role in the attainment of the goals set forth by the European Green Deal by sustainable utilization of biomass (European Commission, 2012). The wood-based industry is one of the conventional bioeconomy industries strongly connected to forestry-based industries and can help address many environmental challenges. (MGRT, 2021). It is a very important EU economic sector and covers a range of forestry downstream activities (Scarlat *et al.*, 2015). Efficient utilization of wood to meet the growing demands for wooden products and wood-based composites is a central tenet of the circular economy (Janiszewska *et al.*, 2016; Antov *et al.*, 2021). Companies can further advance this principle by embracing digitalization to optimize processes, enhance the responsible use of raw materials, improve waste management practices, and contribute to sustainable, environmentally friendly development (Watanabe *et al.*, 2019). However, the successful implementation of the digital and green transition hinges on the knowledge and skills of employees within organizations. Without the appropriate expertise, organizations may encounter difficulties in adopting and integrating sustainable and digital practices (Kropivšek and Zupančič, 2016; Koch *et al.*, 2022). Therefore, it is imperative for the education system to adapt by enriching curricula, which can also affect teaching formats and methods. As part of the preparation and planning of educational programmes, an important step involves examining the current state (Ličen, 2015). However, when analyzing and comparing the current state of educational programmes from different countries, one must consider that education, training, and qualification systems vary across them due to their intricate dynamics between the states, the labour market, and employers (Mikulec and Ermenc, 2016), and recognize that education development is interconnected with the broader economy (Mingat and Tan, 1988).

European Qualifications Framework (EQF) is a European instrument designed to enable the comparability of qualifications in European countries and is intended for all types and categories of qualifications, from general and vocational qualifications to higher education qualifications, and also qualifications obtained in a non-formal or informal context (Mikulec and Ermenc, 2022), and thus a comparison between countries that have adopted the EQF is possible. To consider multiple factors, e.g. when studying outputs of education, data envelopment analysis (DEA) can be used for frontier-efficiency analysis.

The concept of frontier-efficiency, which explores the connection between inputs and outputs initially introduced by Farrell (1957) gained substantial recognition following the seminal work of Charnes, and Rhodes (1978). Since then numerous studies have been conducted to measure the efficiency using DEA, including those focusing on educational institutions, where DEA has emerged as the most widely used technique (Zuluaga-Ortiz *et al.*, 2022), because of the ability to offer a mathematical solution for calculating efficiency, particularly in situations where organizations contend with multiple inputs and outputs (Moore, 2021). However, assessing the efficiency of educational institutions and determining the value of numerous inputs and outputs can be challenging. Moreover, there is no definitive study providing clear guidance on the selection of inputs and outputs for evaluating the efficiency of education (Joumady and Ris, 2005). This complexity underscores the need for careful consideration and contextual understanding when applying methods like DEA to measure the efficiency of education. Different authors study the relative efficiency of education from various angles. For instance, many studies have relied on the outcomes of standardized tests as indicators, with a majority utilizing the results from the Programme for International Student Assessment (PISA) (Henriques *et al.*, 2022) conducted by the Organization for Economic Co-operation and Development (OECD). Giambona *et al*. (2011) employed the results of PISA 2006 as outputs to examine the efficiency of educational systems in EU countries, considering the educational resources accessible at home and students' family background. Similarly, Henriques *et al*. (2022) studied the efficiency of secondary schools using PISA data. Some other studies have employed the results from standardized tests typically administered at the conclusion of students' studies (Zuluaga-Ortiz *et al.*, 2022). This implies that there are various factors, or perspectives related to efficiency of education, which indicates and confirms that the efficiency of education is not consistent or uniform across all contexts or situations.

As education plays a vital role in shaping individuals and preparing them for the challenges of the future, our research endeavours to investigate the extent to which green and digital topics are integrated into wood science and technology educational programmes across various European countries. Through interviews and rigorous analysis, our aim is to assess the inclusion of these topics at different educational levels, considering the macroeconomic and institutional context as well as other factors in the countries under study. Through our examination of the relative efficiency of these programs, our aim is to gain a comprehensive understanding of the current educational landscape. By identifying areas for improvement, we hope to contribute to informed decision-making and the advancement of educational practices in this field.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

The methodology chapter of our study is divided into three parts. In the first part, the data collection process is described, detailing how the data was gathered. The second part focuses on the data processing procedures, explaining the steps taken to organize and prepare the data for analysis. Lastly, in the third part, the DEA method is introduced, employed to assess the efficiency of various educational programmes.

2.1 Data collection
2.1. Prikupljanje podataka

To evaluate the inclusion of specific green and digital topics in wood science and technology educational programmes, remote interviews were conducted with teachers and relevant individuals from educational institutions, which have a holistic view of the studied educational program. The interviews gathered data on the level of inclusion of content in teaching, focusing on green and digital topics, where digital topics were further divided into industry 4.0 (I4.0) and ambient and assisted living (AAL) subtopics (Table 1). The interviews were conducted over six months in years 2021 and 2022, as part of the ALLVIEW project, spanning various educational levels, including upper national diploma (EQF 4), higher national diploma (EQF 5), bachelor's degree (EQF 6), and master's degree (EQF 7), in seven European countries. It is important to note that the presented results reflect the situation in a single institution within each country, although the same or similar programs are offered in multiple locations within the same country.

2.2 Data processing
2.2. Obrada podataka

Based on the findings from the conducted interviews, we were able to determine the level of inclusion

Table 1 List of topics by areas
Tablica 1. Popis tema prema područjima

Green topics *Zelene teme*	**Industry 4.0 topics** *Teme o industriji 4.0*	**Ambient and assisted living topics** *Teme o ambijentu i potpomognutom životu*
Eco Design / *ekodizajn* Cascading use / *kaskadna uporaba* Natural resource management *upravljanje prirodnim resursima* Renewable energy sources *obnovljivi izvori energije* Sustainable production / *održiva proizvodnja* Environmental effects / *ekološki učinci* Circular business models / *kružni poslovni modeli* Industrial symbiosis / *industrijska simbioza* Biotechnology / *biotehnologija* Systemic thinking / *sistemsko razmišljanje* Wood residues to energy / *drvni ostatci i energija* Biorefinery / *biorafinerija* Green chemicals / *zelene kemikalije* Bioeconomy / *bioekonomija* Functional materials / *funkcionalni materijali* LCA analysis / *LCA analiza* Collection and recycling / *prikupljanje i recikliranje* Transparency in supply chains / *transparentnost u opskrbnim lancima* Hazardous chemicals / *opasne kemikalije* Green public procurement / *zelena javna nabava* Nano technology / *nanotehnologija*	Cross Reality *križna stvarnost* Cloud computing *računalni oblak* Online security *online sigurnost* Internet of Things *internet stvari* Simulations / *simulacije* Autonomous robots *autonomni roboti* Big data *velike baze podataka* Additive production *aditivna proizvodnja* Artificial intelligence *umjetna inteligencija*	Smart buildings / *pametne zgrade* Smart furniture / *pametni namještaj* Ergonomic interior design *ergonomski dizajn interijera* Sensors / *senzori* Fire and other safety *protupožarna i druga zaštita*

of individual topics within each respective educational program across different countries. These results allowed us to calculate the average representation of fully included, partially included, and not included topics within Green, I4.0 and AAL areas.

2.2.1 Fuzzy logic model
2.2.1. Model neizrazite logike

To optimize the data for DEA analysis, a 10-point fuzzy scale was devised to classify the three areas (green, I4.0 and AAL) of the individual educational programmes under study, using fuzzy set theory, introduced by Zadeh (1965). Fuzzy logic can deal with imprecise information as an element x can only partially belong to a fuzzy set A. The degree of membership of x in A is determined by the value of a membership function $u_A(x)$, which ranges from 0 to 1. For our model, linear membership functions were chosen with triangular and trapezoidal shapes because they are well suited and easy to implement (Carbajal-Hernández *et al.*, 2012).

The objective of the fuzzy logic model was to aggregate the proportions of not included, partially included, and fully included aspects of individual areas and educational programmes into a unified score. The construction of the fuzzy logic model involved four phases. First, the fuzzy logic inference system (FIS) (Carbajal-Hernández *et al.*, 2012; Jamshidi *et al.*, 2013) was constructed. The membership functions for

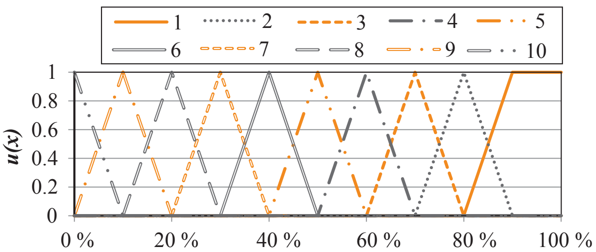

Figure 1 Membership functions for percentage of not included topics
Slika 1. Funkcije članstva za postotak neuključenih tema

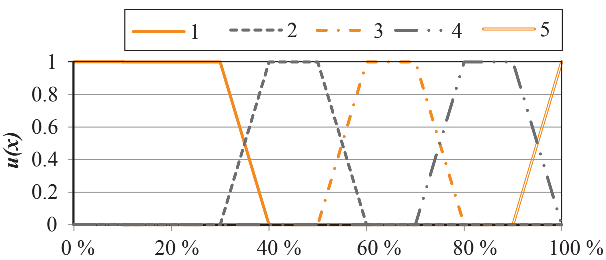

Figure 2 Membership functions for percentage of partially included topics
Slika 2. Funkcije članstva za postotak djelomično uključenih tema

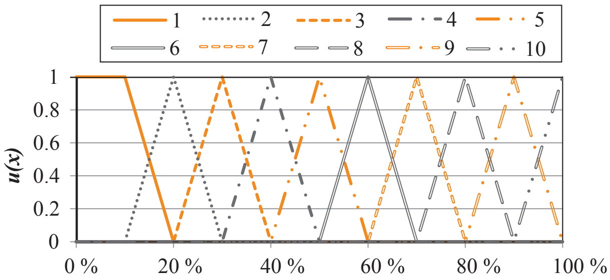

Figure 3 Membership functions for percentage of fully included topics
Slika 3. Funkcije članstva za postotak potpuno uključenih tema

not included, partially and fully included topics were determined and are shown in Figures 1-3.

To reduce the negative interference between the parameters of the partially and fully included areas, the FIS was divided into two separate parts. Part *a* aggregates not included and partially included areas, while part *b* aggregates not included and fully included areas. The Mamdani-type inference system (Mamdani and Assilian, 1975) with intuitive rules and imitation of human reasoning (Che Osmi *et al.*, 2016; Kovac *et al.*, 2012) was selected. Then, the membership functions were defined for all three areas. In the following phase, IF-THEN decision rules were established. The basic fuzzy operations employed were intersection (AND) $u_{A\cap B}(x) = \min(u_A(x), u_B(x))$ and union (OR) $u_{A\cup B}(x) = \max(u_A(x), u_B(x))$. The areas were aggregated using the max-min composition of membership functions:

$$u_c(z) = \max(\min(u_A(x), u_B(y))) \tag{1}$$

Where u_C, u_A, and u_B are membership functions of the output z and inputs x and y, respectively. Table 2 presents the possible combinations of IF-THEN rules, e.g., IF topics are absent in the given area at most 20 % and are at least 80 % fully included, THEN, the resulting fuzzy score is 8.

The third phase encompassed the defuzzification process to convert the aggregated fuzzy sets into crisp values. The centre of maximum was chosen as one of the most commonly used defuzzification methods (Pathak *et al.*, 2005; Ross, 2004), which calculates the weighted mean of the centres of areas x_k and the membership functions $k=1,..., n$ (Eq. 2):

$$x^* = \frac{\sum_{k=1}^{n} x_k u_{Ck}(x)}{\sum_{k=1}^{n} u_{Ck}(x)} = \frac{1\,u_1(x) + 2\,u_2(x) + \cdots + 10\,u_{10}(x)}{u_1(x) + u_2(x) + \cdots + u_{10}(x)} \tag{2}$$

Both parts *a* and *b* were analysed independently in all phases until a crisp value x_a^* and x_b^* were obtained. In phase 4, the individual scores were combined using equation 3, resulting in a final classification represented by x_{ab}^* on a 10-point scale. The final scores represent the outputs in our DEA model, as shown in Table 3.

$$x_{ab}^* = (x_a^* + x_b^*) - 1, \tag{3}$$

Table 2 IF-THEN rules of fuzzy inference system *a* and *b*
Tablica 2. IF-THEN pravila neizrazitih sustava zaključivanja *a* i *b*

10-point fuzzy scale *Neizrazita skala od deset točaka*	**FIS *a***		**FIS *b***	
	Not included *Nije uključeno*	**Partially included** *Djelomično uključeno*	**Not included** *Nije uključeno*	**Partially included** *Djelomično uključeno*
10	0 %	/	0 %	100 %
9	10 %	/	10 %	90 %
8	20 %	/	20 %	80 %
7	30 %	/	30 %	70 %
6	40 %	/	40 %	60 %
5	50 %	100 %	50 %	50 %
4	60 %	80-90 %	60 %	40 %
3	70 %	60-70 %	70 %	30 %
2	80 %	40-50 %	80 %	20 %
1	90-100 %	0-30 %	90-100 %	0-10 %

2.3 Data envelopment analysis (DEA)
2.3. Analiza omeđivanja podataka (DEA)

DEA involves using linear programming for measuring the efficiency of decision making units (DMUs), which convert multiple inputs into multiple outputs (Coelli *et al*., 2005). The DEA method is based on the concept of Pareto efficiency, which states that the full efficiency of a DMU is achieved when the value of none of the inputs or outputs can be improved without reducing the value of any other input or output.

The model chosen for this study is based on constant returns to scale (CRS) with output orientation that focuses on maximizing outputs while holding inputs constant, following the notation adopted by Johnes (2004). The choice of orientation is not as crucial in education as it is in econometric estimations (Coelli and Perelman, 1999). In the context of our study, where the efficiency of topics inclusion in teaching is examined based on inputs over which educational institutions have less control, an output orientation is more appropriate. Each of n DMUs requires m different inputs to produce s different outputs. Specifically, DMU k requires x_{ik} units of input i and produces y_{rk} units of output r. Here, it is assumed that each DMU has at least one positive input and one positive output. In output-oriented DEA model, the linear program has a multiplicative form and aims to maximize the weighted sum of outputs (Eq. 4), while keeping the values of the inputs constant (Eq. 6). The technical efficiency of DMU k is denoted by θ_k, and the linear program has $n+1$ constraints (Eqs. 5, 6).

$$\max \theta_k = \sum_{r=1}^{s} u_r y_{rk} \quad (4)$$

Subject to:

$$\sum_{i=1}^{m} v_i x_{ij} - \sum_{r=1}^{s} u_r y_{rk} \geq 0 \quad j = 1,\ldots,n \quad (5)$$

$$\sum_{i=1}^{m} v_r x_{ik} = 1 \quad (6)$$

$$u_r, v_i, x_{ik}, y_{rk}, \geq 0 \quad \forall_r = 1,\ldots,s; \quad i = 1,\ldots,m \quad (7)$$

2.3.1 Inputs and outputs for DEA analysis
2.3.1. Ulazi i izlazi za DEA analizu

As previously mentioned, the outputs consist of inclusion level for individual content areas (green, I4.0, AAL) within various educational programmes across different EU countries. These levels were determined through the fuzzification of interview results, mentioned above.

Since education development does not occur in isolation from the rest of the economy, our inputs were based on macroeconomic and other indicators related to our outputs, that reflect unique characteristics of each country. Therefore, the inputs used in this study include (1) country's GDP per capita (European Commission, 2022b), as countries with higher economic development often invest in improving educational standards and curriculum, (2) annual expenditure per student on educational institutions (OECD, 2021), as increased funding also contributes to offering a comprehensive and enriching learning experience, (3) digitalization level of the country (European Commission, 2022a), as the impact of digitalization on education mirrors the digital progress in the country, (4) index for digital lifelong learning readiness (Centre for European Policy Studies, 2019), as aging population in the EU is steadily increasing, (5) value added per employee in C16 (wood processing – except furniture) + C31 (manufacture of furniture) (Ronzon *et al*., 2022), as it can have a positive effect by providing opportunities for investments in developing business and educational infrastructure and technology, (6) country's greenhouse gas emissions per capita (European Environment Agency, 2022), as inclusion of green topics in the curriculum becomes more important as emissions rise, and lastly, (7) the level of topic inclusion in the lower educational programmes is considered, as inclusion level should increase along the educational vertical.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

Table 3 highlights disparities in topic inclusion among countries, even within the same educational level, pointing to inconsistencies in educational verticals within each country. However, it is important to note that our findings are based on a single institution per program in each country, and educational programmes may have unique objectives. While the importance of including these topics may vary, EU guidelines emphasize the significance of sustainable and digital competences for future students. As a result, all educational programmes will need to adapt at some point, although the extent of this adaptation remains uncertain.

Table 3 shows significant variations in input values among countries. Poland has the lowest GDP per capita, while the Netherlands has the highest. Annual expenditure per student is lower in vocational education compared to higher education across all countries, with Poland and Slovenia having the lowest. The Netherlands has the highest annual spending per student in higher education and ranks highest in digital lifelong learning readiness and digitization levels. Germany has the lowest score in digital lifelong learning readiness, and Italy has the lowest digitization level. Belgium has the highest value added per employee in the C16 and C31 sectors, while Poland has the lowest. Poland also has the highest greenhouse gas (GHG) emissions per capita, while Spain has significantly lower GHG emissions per capita.

When determining the average inclusion level from a lower EQF level, two assumptions were made. First, it was assumed that students in each country at a specific EQF level possess prior knowledge from a lower level, as studied in this research. Second, it was assumed that students have no prior knowledge before entering EQF 4.

Figure 4 illustrates the technical efficiency of individual DMUs using DEA. Among the 23 DMUs in our study, 10 are identified as fully efficient in including green, I4.0 and AAL topics in teaching, based on given inputs. These include EQF 6 and 7 in Slovenia, EQF 4 in Belgium, Germany, and Spain, EQF 6 in Ger-

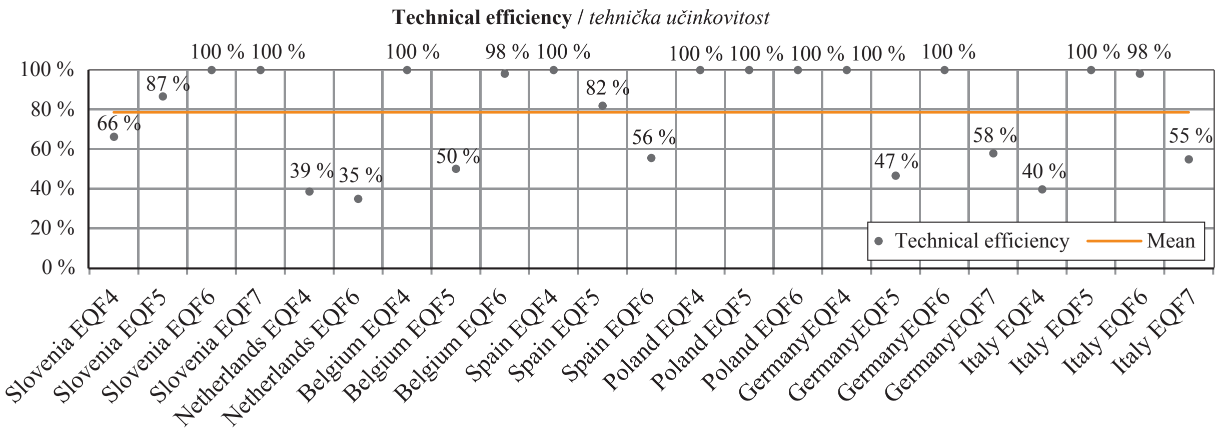

Figure 4 Efficiency of different educational programmes across various institutes in studied countries
Slika 4. Učinkovitost različitih obrazovnih programa u različitim institucijama promatranih zemalja

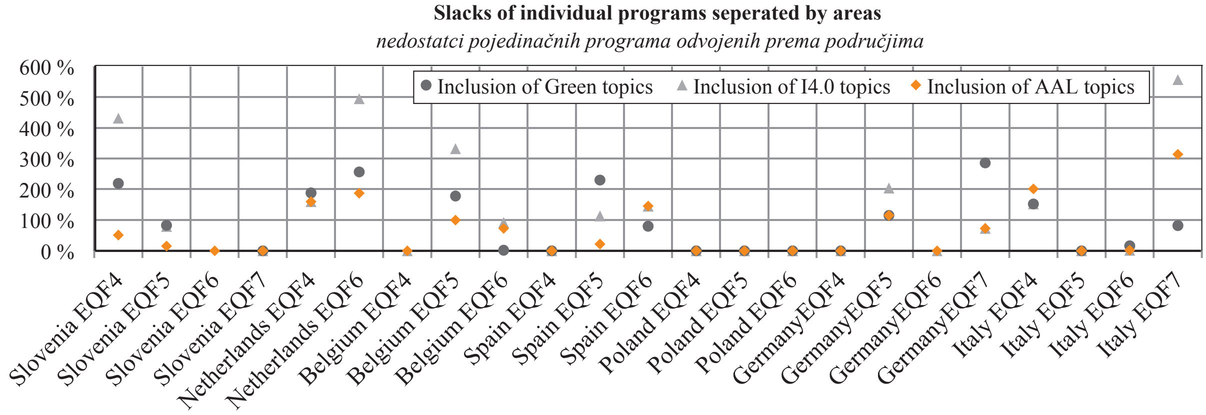

Figure 5 Slacks of areas in individual DMUs, to reach full efficiency
Slika 5. Nedostatci područja u pojedinačnim DMU-ovima kako bi se postigla potpuna učinkovitost

Table 3 Level of inclusion of different topics in wood science and technology educational programmes across different countries, used as representation of outputs and values of the inputs for DEA
Tablica 3. Razina uključenosti različitih tema u obrazovne programe za znanost o drvu i drvnu tehnologiju u različitim zemljama koja služi kao prikaz izlaznih rezultata i ulaznih vrijednosti za DEA

DMUs		**Outputs** / *Izlazi*			**Inputs** / *Ulazi*						
Country *Država*	Level of education *Razina obrazovanja*	Inclusion level of green topics *Razina uključenosti zelenih tema*	Inclusion level of Industry 4.0 topics *Razina uključenosti tema industrije 4.0*	Inclusion level of ambient and assisted living topics *Razina uključenosti tema o ambijentu i uzdržavanju*	Country's GDP per capita, € *BDP zemalja po stanovniku, €*	Annual expenditure per student on educational institutions, $ *Godišnji izdatci po učeniku u obrazovnim institucijama, $*	Score for digital lifelong learning readiness, Index *Ocjena spremnosti za digitalno cjeloživotno učenje, indeks*	Digitalization level, Index *Stupanj digitalizacije, indeks*	Value added per employee in C16 + C31, € *Dodana vrijednost po zaposleniku u C16 + C31, €*	Greenhouse gas emissions per capita *Emisije stakleničkih plinova po stanovniku*	Average inclusion of all 3 topics from lower EQF level *Prosječna uključenost svih triju tema s niže EQF razine*
Slovenia	EQF 4	2.00	1.54	4.00	21260	9772	0.60	53.5	29471	7.6	1.00
	EQF 5	2.69	2.70	6.00	21260	9772	0.60	53.5	29471	7.6	2.51
	EQF 6	7.14	7.00	10.00	21260	14060	0.60	53.5	29471	7.6	3.80
	EQF 7	9.05	7.78	10.00	21260	14060	0.60	53.5	29471	7.6	8.05
Netherlands	EQF 4	2.24	3.13	3.00	41860	14726	0.68	67.3	65798	9.8	1.00
	EQF 6	2.24	1.33	4.00	41860	20898	0.68	67.3	65798	9.8	2.79
Belgium	EQF 4	6.63	6.67	10.00	35850	14758	0.57	50.4	77448	9.5	1.00
	EQF 5	2.38	1.54	5.00	35850	14758	0.57	50.4	77448	9.5	7.77
	EQF 6	7.21	4.44	4.00	35850	20471	0.57	50.4	77448	9.5	2.97
Spain	EQF 4	6.67	7.00	7.00	23510	10290	0.63	60.7	38212	5.9	1.00
	EQF 5	2.00	2.66	6.00	23510	10290	0.63	60.7	38212	5.9	6.89
	EQF 6	3.80	2.66	3.00	23510	13800	0.63	60.7	38212	5.9	3.55
Poland	EQF 4	6.24	10.00	5.00	13580	8220	0.57	40.5	19445	10.0	1.00
	EQF 6	7.00	6.00	8.00	13580	11192	0.57	40.5	19445	10.0	7.08
	EQF 7	7.00	6.00	8.00	13580	11192	0.57	40.5	19445	10.0	7.00
Germany	EQF 4	6.24	2.70	5.00	35480	13926	0.50	52.9	55036	8.9	1.00
	EQF 5	3.00	2.00	4.00	35480	13926	0.50	52.9	55036	8.9	4.65
	EQF 6	7.14	6.67	7.00	35480	19324	0.50	52.9	55036	8.9	3.00
	EQF 7	1.76	4.22	4.00	35480	19324	0.50	52.9	55036	8.9	6.94
Italy	EQF 4	2.24	2.56	2.00	26700	11962	0.51	49.2	43598	6.5	1.00
	EQF 5	6.05	7.78	3.00	26700	12305	0.51	49.2	43598	6.5	2.26
	EQF 6	6.00	7.00	6.00	26700	12305	0.51	49.2	43598	6.5	5.61
	EQF 7	4.14	1.00	2.00	26700	12305	0.51	49.2	43598	6.5	6.33

Table 4 Ranks of DMUs by level of inclusion (before DEA) and by efficiency ranks (after DEA)
Tablica 4. Poredak DMU-ova prema razini uključenosti (prije DEA) i rangu učinkovitosti (nakon DEA)

DMUs		SUM level of inclusion *SUM razina uključenosti*	Rank by SUM *Poredak prema SUM razini*	DEA efficiency *DEA učinkovitost*	Rank by DEA efficiency *Poredak prema DEA učinkovitosti*
Country *Država*	**Level of education** *Razina obrazovanja*				
Slovenia	EQF 4	27	1	66 %	5
	EQF 5	24	2	87 %	3
	EQF 6	23	3	100 %	1
	EQF 7	21	4	100 %	1
Netherlands	EQF 4	21	4	39 %	12
	EQF 6	21	4	35 %	13
Belgium	EQF 4	21	4	100 %	1
	EQF 5	16	7	50 %	9
	EQF 6	21	4	98 %	2
Spain	EQF 4	19	5	100 %	1
	EQF 5	17	6	82 %	4
	EQF 6	14	8	56 %	7
Poland	EQF 4	11	9	100 %	1
	EQF 6	9	11	100 %	1
	EQF 7	11	9	100 %	1
Germany	EQF 4	10	10	100 %	1
	EQF 5	9	11	47 %	10
	EQF 6	8	13	100 %	1
	EQF 7	8	13	58 %	6
Italy	EQF 4	7	14	40 %	11
	EQF 5	8	13	100 %	1
	EQF 6	7	14	98 %	2
	EQF 7	9	12	55 %	8

many, EQF 5 in Italy, and all studied EQFs (4, 5, and 6) in Poland. The average efficiency across all DMUs is 79 %, and in addition to the fully efficient units, 4 other units perform above the average. However, there are nine educational programmes from various countries that demonstrate below-average performance levels. Some of these programs exhibit an efficiency of 40 % or even lower. To enhance their efficiency, the institutions where these programs were studied should significantly increase the inclusion of these topics. For example, educational institution, where the data for the EQF 4 and 5 were obtained in the Netherlands, should increase inclusion of these topics by 61 % in EQF 4 and by 65 % in EQF 5. These two EQFs were identified as the least efficient in our study.

Figure 5 provides a visual representation of the slacks for individual areas within studied DMUs. By incorporating topics from these areas more extensively in teaching, DMUs have the potential to enhance their performance and approach the efficiency frontier. It is notable that less efficient DMUs (Figure 4) tend to exhibit more significant slacks (Figure 5). It becomes apparent that the largest slacks are commonly observed in domain of industry 4.0.

Table 4 shows that the ranking changes when additional conditions are also included in the DEA as inputs. For example, EQF level 4 in Slovenia landed in first place based on content inclusion, but after the DEA analysis, when additional conditions were considered, it only showed 66 % efficiency and dropped significantly in the ranking. It was different with EQF 5 and 6 in Italy. After considering content inclusion alone, these two programs achieved last and second-to-last position in the ranking. However, after applying DEA analysis, they achieved exceptional efficiency and moved up to the 1st (EQF 5) and 2nd (EQF 6) position. This highlights the significance of considering multiple conditions, particularly in cross-country comparisons, during research of this nature.

4 CONCLUSIONS
4. ZAKLJUČAK

The increasing importance of sustainable and digital competences is playing a pivotal role in shaping a promising future. To realize this vision, it is vital to integrate these subjects into educational curricula, equipping future students with the necessary knowledge and skills. As an initial stride in exploring this domain, the inclusion of specific content in wood science and technology educational programmes has been assessed in various EU countries. As education systems in different countries vary in terms of conditions and opportunities for improving educational standards and curriculum, the efficiency of individual educational programmes in incorporating green and digital topics into teaching were further analysed, considering other relevant factors in the countries under study. Taking into account multiple factors has demonstrated its significance, particularly when conducting cross-country comparisons.

In conclusion, the findings of our research reveal significant disparities in the inclusion of green, industry 4.0, and ambient assisted living topics within wood science and technology educational programmes across different countries and educational levels. Moreover, the application of Data Envelopment Analysis (DEA) in assessing technical efficiency highlights notable disparities among programs in effectively incorporating these topics into teaching. While some programs have attained full efficiency, demonstrating optimal performance based on our selected inputs, others exhibit suboptimal levels of performance. To improve the efficiency of underperforming programs, it is crucial for the responsible institutions to prioritize the incorporation of these topics in teaching. Additionally, the calculation of slacks offers valuable insights into specific areas where certain educational programmes fall behind in the inclusion of individual topics. This analysis provides a detailed understanding of the deviations in comparison to other programs examined in our research.

In summary, our research highlights the imperative for comprehensive inclusion of green, I4.0, and AAL topics in educational programs. It is crucial to coordinate this effort at the national level, ensuring alignment across all levels of education. By doing so, a cohesive and well-rounded educational system can be fostered that effectively addresses these important topics. By equipping individuals with these essential competences, they are empowered to contribute to sustainable development and effectively leverage digital technologies to drive innovation and progress. This serves as a catalyst for building a resilient and adaptable workforce, equipped with the necessary skills to tackle the evolving demands of our society.

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Effect of Natural Weathering on Performance of Wood Flour-Recycled Polypropylene Composites

Utjecaj prirodnog izlaganja vremenskim utjecajima na svojstva kompozita od drvnog brašna i recikliranog polipropilena

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ABSTRACT • *In this study, the effect of natural weathering on the physical and mechanical properties of wood plastic composites (WPC) made from virgin and recycled polypropylene (PP) was studied. To prepare the recycled PP, virgin PP was thermo-mechanically degraded by extrusion under controlled conditions in a single-screw extruder at a router speed of 60 rpm and temperature of 190 °C. PP (virgin and recycled), wood flour, compatibilizer, and UV absorbent were physically blended, and the samples were manufactured by a twin-screw extruder. The samples were exposed to natural weathering for 270 days. The surface characteristics of the samples were investigated before and after weathering. According to the results, the composites from recycled PP exhibited a higher weathering resistance than those from virgin PP. The use of a UV absorber improved the flexural strength and modulus of the composites, but it could not significantly prevent the flexural properties loss and discoloration of the composites after weathering.*

KEYWORDS: *wood plastic composites; recycled polypropylene; natural weathering; discoloration, surface roughness*

SAŽETAK • *U ovom je istraživanju proučavan utjecaj prirodnog izlaganja vremenskim utjecajima na fizička i mehanička svojstva drvno-plastičnih kompozita (WPC) izrađenih od čistoga i recikliranog polipropilena (PP). U postupku pripreme recikliranog polipropilena čisti je polipropilen termomehanički razgrađen ekstruzijom s jednim pužem u konstantnim uvjetima i pri brzini vrtnje od 60 okr./min te pri temperaturi od 190 °C. Pomiješani su čisti i reciklirani polipropilen, drvno brašno, kompatibilizator i UV apsorber, a uzorci su proizvedeni ekstruderom s dva puža. Uzorci su bili 270 dana prirodno izloženi vremenskim utjecajima. Svojstva površine uzoraka ispitana su prije i nakon izlaganja tim utjecajima. Iz rezultata je vidljivo da su kompoziti od recikliranog polipropilena pokazali veću otpornost na vremenske utjecaje nego kompoziti od čistog polipropilena. Upotreba UV apsorbera poboljšala je čvrstoću na savijanje i modul elastičnosti kompozita, ali nije značajno spriječila slabljenje svojstava savijanja i promjenu boje kompozita nakon izlaganja vremenskim utjecajima.*

KLJUČNE RIJEČI: *drvno-plastični kompoziti; reciklirani polipropilen; prirodno izlaganje vremenskim utjecajima; hrapavost površine*

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1 INTRODUCTION
1. UVOD

The use of wood-plastic composites (WPCs) for outdoor applications has found its place in the largest and fastest consumer market for WPCs. Due to the increased use of these products in outdoor construction, attention should be focused on the durability of these products. Prolonged exposure to natural environmental changes, such as ultraviolet (UV) radiation, humidity, temperature, and atmospheric pollution, causes some complex physical and chemical reactions of WPC that lead to changes in their appearance, and physical and mechanical properties. Depending on the type of application, composite formulation, manufacturing process parameters, and environmental and climatic differences, the investigation of the natural weathering behavior of WPCs is necessary.

Since natural weathering testing is time intensive, only a few publications in this field are available (references). Taib *et al.* (2010) and Silva *et al* (2017) reported an initial increase in the flexural strength (MOR) of different WPCs in the first months of weathering, followed by a decrease to values lower than the original strength at the end of the test. The initial enhancement of strength was explained by crystallization with crosslinking after initial chain scissions. Hung *et al.* (2012) and Zhou *et al.* (2016) showed that the MOR of WPCs declined during the natural weathering test. The addition of stabilizers to the formulation significantly decreased the strength loss of WPCs. According to Taib *et al.* (2010), the addition of HALS as an antioxidant was more effective than photo-stabilizers like UV absorbers. In addition, fiber pretreatment by acetylation effectively improved the strength of WPCs against natural weathering (Hung *et al.*, 2012). Badji *et al.* (2017) reported a chain scission phenomenon caused by thermo- and photo-oxidative degradations in the polymer matrix of wood-flour reinforced polypropylene (PP) composites during natural weathering. They indicate that the presence of wood flour stabilizes the polymer against chain scissions. Ratanawilai and Taneerat (2018) studied mechanical property losses from natural weathering of WPCs over 90 days and emphasized that the WPCs based on polystyrene and polypropylene are a better choice than low-density polyethylene (LDPE) for exposure to natural weathering. Gunjal *et al.* (2020) showed that natural weathering increases the lightness and total color change of wood-flour polypropylene composites after one year of exposure. The maximum color change was observed in the initial four months, and the color change of WPCs with larger wood particle size was characterized with better color stability than that with small particle size.

The waste and recycled plastics were used for manufacturing WPCs already in 1990s and the use has significantly increased in developed and developing countries in recent years (Kazemi-Najafi *et al.*, 2006). Since recycled plastics may be obtained from various sources, having been exposed to different storage and reprocessing conditions, they may therefore exhibit different performance depending on their degradation level. Then the post-consumer plastics waste may contain many grades, colors and contaminants, leading to varying outcomes when these plastics are combined with wood flour/fillers. According to the mode of initiation, the following types of degradation can be distinguished: thermal, chemical, mechanical, and biological. Degradation processes are generally quite complex; often more than one type of degradation is operational, e.g. thermo-oxidative degradation, thermo-mechanical degradation, etc.

The degradation of plastics due to repeated processing cycles and environmental exposure complicates recycling, so specific research is required. The effect of recycled plastic on the physical and mechanical properties of WPCs has been extensively investigated by many researchers (Kazemi-Najafi *et al.*, 2006; Adhikary *et al.*, 2008; Kazemi-Najafi *et al.*, 2009) but there are few studies about the durability of WPCs from recycled plastic in outdoor applications. According to Homkhiew *et al.* (2014), Virgin polypropylene showed smaller relative changes of lightness and smaller relative loss of hardness, *MOR*, and *MOE* than recycled polypropylene, both in composites and unfilled plastic during natural weathering tests. Homkhiew *et al.* (2022) revealed that the type of post-consumer plastic influences the mechanical strength loss of WPCs after 6 months of natural weathering. Widiastuti *et al.* (2023) have observed the degradation of mechanical and physical performances, as significant loss of stiffness and increase of whitening area, after 30 days of exposure to natural weathering for ironwood-based recycled polypropylene composites.

Outdoor trials are hardly comparable because ambient climate conditions strongly depend on the location and vary significantly in temperature, humidity, global radiation dosage, and amount of precipitation. On the other hand, the use of recycled plastics in the manufacture of wood-plastic composites is increasing. This underscores the necessity for further research to elucidate the influence of various factors on the degradation of WPCs. The development of strategies to counteract these degradation factors is paramount for improving the durability of WPCs in outdoor settings. Studying the effects of natural weathering on wood-plastic composites (WPCs) made from wood flour and thermo-mechanically degraded polypropylene will provide important insights into their durability and performance in outdoor environments. This research could potentially lead to the development of more resilient WPCs and contribute to the body of knowledge in this field.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

The polypropylene (PP) powder, grade V30S, with a melt flow index of 18 g/10 min, was procured from the Marun Petrochemical Complex (Mahshahr, Iran). Beechwood flour, with a particle size of -60/+40 mesh, was obtained by screening sawdust from a local sawmill. The wood flour was oven-dried at a temperature of (100±5) °C for 24 h. Maleic anhydride-polypropylene (MAPP) (Grade G 6070 3×8), with a melt flow index of 24 g/10min, was sourced from the Kometra Company in Germany. Tinuvin327, a UV absorbent (UVA), was supplied by the Chemical Tools Company (Tehran, Iran).

2.2 Preparation of recycled polypropylene
2.2. Priprema recikliranog polipropilena

To produce the recycled PP, virgin PP was subjected to thermo-mechanical degradation under controlled conditions in a single-screw extruder. This process was conducted at a rotor speed of 60 rpm and a temperature of 190 °C. Subsequently, the degraded PP was ground into fine powder. The melt flow index (MFI) of the recycled PP, measured by the ASTM D 1238-98 standard, was determined to be 23.5 g / 10 min.

2.3 Manufacturing of wood plastic composites
2.3. Izrada drvno-plastičnog kompozita

The wood flour, recycled and virgin PP, and UV absorbent (UVA) were physically mixed in specific weight percentage ratios to produce the composites, as detailed in Table 2. The premixed materials for each formulation were extruded into strips using a counter-rotating twin-screw extruder (model WPC-65132) manufactured by GMW Company (Tehran, Iran). The strips had a cross-section of 1 cm in thickness and 7 cm in width. All strips were conditioned at a temperature of (22±2) °C and relative humidity of (65±5) % for two weeks. Following the conditioning period, they were cut into test specimens for the planned tests.

2.4 Natural weathering test
2.4. Test izlaganja vremenskim utjecajima

This test was performed in accordance with the ASTM D1435-99 Standard. The specimens were positioned on ranks at a 45° angle facing southeast. The impact of natural weathering on WPCs was assessed from January to October (270 days) at a geographic location of 52º 2′ E, 36º 34′ N, with an elevation of 15 meters below sea level. The climatic data for the research period, sourced from the Noshahr synoptic station (Mazandaran, Iran), is presented in Table 2.

Table 1 Wood plastic composite formulations (percent by weight)
Tablica 1. Formulacije drvno-plastičnih kompozita (maseni udio)

Composites code *Oznaka uzorka*	**Wood flour, %** *Drvno brašno, %*	**PP, %**	**UVA*, %**	**MAPP, %**
WVP	65	33	0	2
WRP	65	33	0	2
WRP + UVA	65	33	0.8	2
WRP + UVA	65	33	0.8	2

*by weight of PP / *u odnosu prema masenom udjelu polipropilena*
PP – polypropylene / *polipropilen*, VP – virgin PP / *čisti polipropilen*, RP – recycled PP / *reciklirani polipropilen*, W – Wood flour / *drvno brašno*, UVA – UV absorbent / *UV apsorber*

Table 2 Climatic data of research period
Tablica 2. Klimatski podatci tijekom istraživačkog razdoblja

Month *Mjesec*	**Mean monthly temperature, °C** *Srednja mjesečna temperatura, °C*			**Mean relative humidity, %** *Srednja relativna vlaga zraka, %*			**Rainfall, mm** *Padaline, mm*		**Sunshine, h** *Sunčani sati, h*
	Min	Max	Mean	Min	Max	Mean	Max	Total	Total
January	3.4	12.6	8	62	93	77	56.3	179	180
February	7.4	14	10.7	68	95	82	20.5	54.4	122.4
March	7.9	14.2	11	70	94	82	31.1	123.8	95.7
April	11.1	16.6	13.9	71	95	83	4.9	22	113.3
May	13.9	21.6	17.7	65	94	79	27.8	66.4	234.8
June	19.1	26.4	22.8	64	93	79	7.6	13.9	239.1
July	22	29.1	25.6	65	92	79	0	0	235
August	22.1	28.2	25.2	69	94	81	15.1	71.5	116.4
September	21.9	28.2	25.1	70	92	81	39.3	69.5	147.9
October	19.6	27.8	23.7	65	93	79	12.5	16.7	72.6

2.5 Microscopic imaging
2.5. Mikroskopsko snimanje

A Dino-Lite digital microscope, with a magnification of 200x, was used to observe the development of cracks, as well as the growth of molds and fungi on the surface of the specimen.

2.6 Contact angle
2.6. Kontaktni kut

The contact angle of water on the surface of the WPC was measured using a goniometer (PGX-Goniometer, Switzerland) at various intervals after weathering. The contact angle for each specimen was determined using a droplet with a volume of 3.5 micro-liters after 300 seconds. Five samples were tested for each formulation.

2.7 Surface roughness
2.7. Hrapavost površine

Two roughness parameters, namely average roughness (*Ra*) and mean peak-to-valley height (*Rz*), were determined using a HUATEC surface roughness tester, model SRT-6200, in a cut-off path line of 2.5 mm. *Ra* represents the average of all individual measurements of surface peaks and valleys, and *Rz* represents the average heights of the five highest-profile peaks and the depths of the five deepest alleys within the evaluation length. Five samples were tested for each formulation.

2.8 Color change
2.8. Promjena boje

The surface color of the five weathered samples was measured every 90 days according to the ASTM 2244-93 standard using a Sheen Spectrophotometer (400 to 700 nm). The machine data are presented in the form of the CIE $L^*a^*b^*$ systems. Color coordinates of each sample were determined before and after exposure to natural weathering. The color change (ΔE^*) was calculated using the following equation:

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

Where, ΔL^*, Δa^*, and Δb^* indicate the difference between the initial and final values of L^*, a^*, and b^*, respectively.

2.9 FTIR spectroscopy
2.9. FTIR spektroskopija

The surface chemistry of the WPCs before and after natural weathering was analyzed using FTIR spectroscopy (Nicolet spectroscope). For each WPC, %1 w/w oven dried powder was dispersed in a matrix of KBr and pressed to form pellets. The spectroscopy was conducted in the wavenumber range of 4,000 to 400 cm^{-1}.

2.10 Flexural properties
2.10. Svojstva savijanja

The three-point flexural test was conducted in accordance with the ASTM D 790-90 standard. A span length of 200 mm and a crosshead speed of 6mm/min were set for this test. The flexural modulus (*MOE*) and flexural strength (*MOR*) of WPCs were determined before and after weathering at various intervals using a DARTEC Universal Testing Machine. Five samples were tested for each formulation and each period.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Morphological analysis
3.1. Morfološka analiza

Natural weathering can alter the appearance of WPCs, leading to discoloration, surface roughening, and checking. The polypropylene-rich surface layer of extruded WPCs acts as a protective barrier against water absorption and colonization of mold fungi and other discoloring microorganisms. However, this surface layer can degrade under the influence of ultraviolet radiation, which increases water absorption and allows these destructive elements to access the wood flour. As a result, these elements can proliferate and spread throughout the WPC, causing changes in surface color and wettability.

All wooden components are prone to UV radiation damage, but lignin is the main absorber, taking in 80-95 % of UV light. Lignin contains chromophoric functional groups such as phenolics, hydroxyl groups, double bonds, and carbonyl groups, and can form free radicals. The photodegradation process starts with UV light attacking the lignin-rich middle lamella, and with prolonged exposure, the secondary walls also degrade (Stark and Gardner, 2008).

UV light transforms lignin into water-soluble compounds that are washed away by rain, leaving a cellulose-rich, fibrous surface. The impact of UV degradation is mostly seen on the surface. However, degradation has been observed deeper than this, suggesting an energy transfer process where the surface molecules absorb UV light and then dissipate excess energy to create new free radicals. These radicals then migrate deeper into the WPCs, causing discoloration (Hon and Minemura, 2001).

Microphotographs of both unexposed and exposed WPCs, with and without UV absorbent, are presented in Figure 1 and 2. As depicted in the images, the surface of all specimens became lighter in color during the initial period of weathering. Over time, the brightness of the specimens decreased, and their surface darkened.

Additionally, cracks developed on the surface of both virgin and recycled polypropylene composites af-

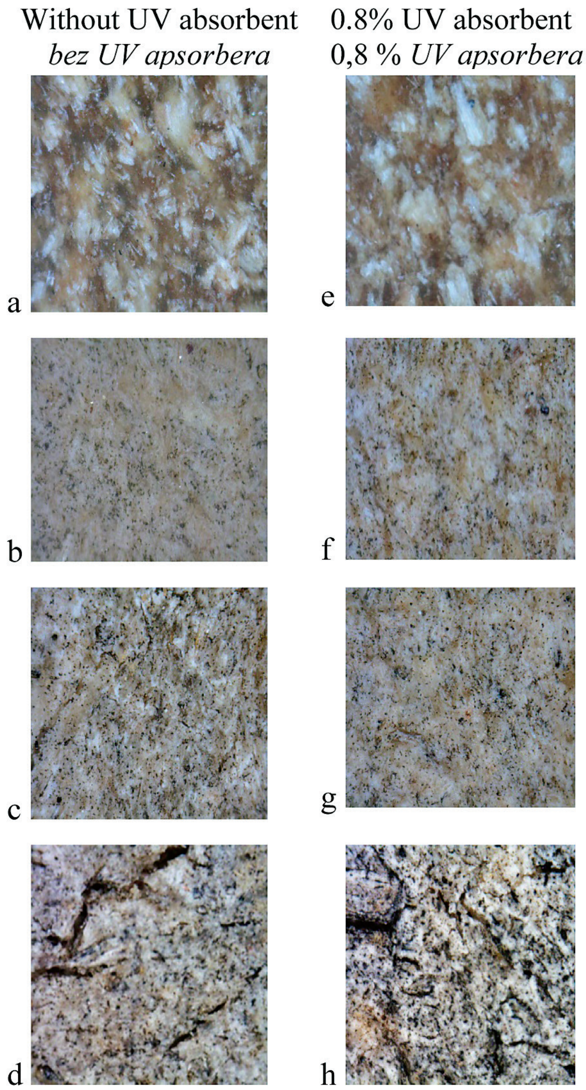

Figure 1 Microscopic images of WPC surface made from virgin polypropylene before natural weathering (a and e), after 90 days (b and f), after 180 days (c-g), and after 270 days (d and h) of natural weathering
Slika 1. Mikrografije površine WPC-a izrađenoga od čistog polipropilena prije prirodnog izlaganja vremenskim utjecajima (a i e), nakon 90 dana (b i f), nakon 180 dana (c-g) i nakon 270 dana izlaganja (d i h)

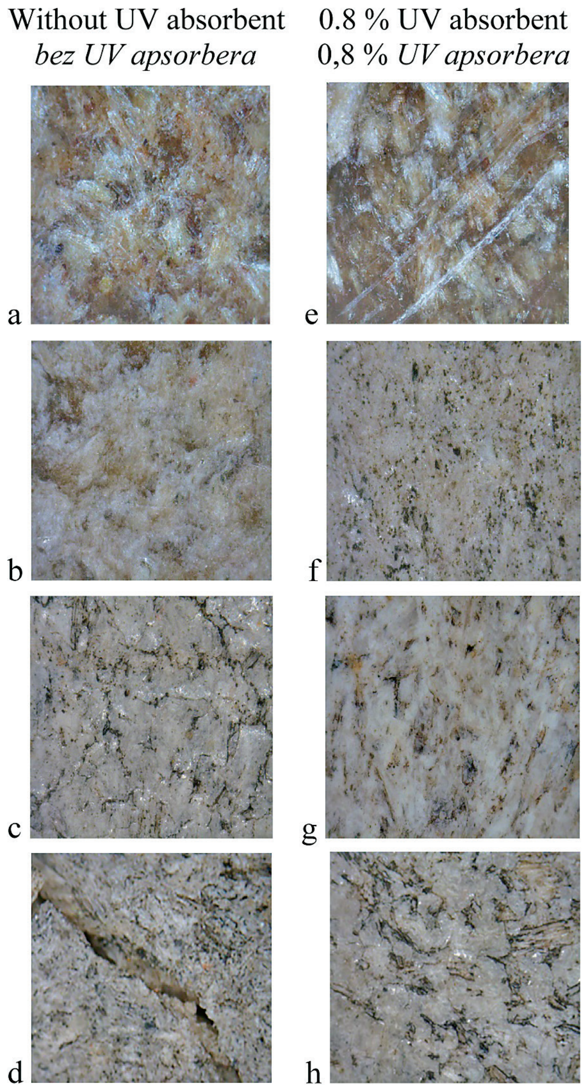

Figure 2 Microscopic images of WPC surface made from recycled polypropylene before natural weathering (a and e), after 90 days (b and f), after 180 days (c-g), and after 270 days (d and h) of natural weathering
Slika 2. Mikrografije površine WPC-a izrađenoga od recikliranog polipropilena prije prirodnog izlaganja vremenskim utjecajima (a i e), nakon 90 dana (b i f), nakon 180 dana (c-g) i nakon 270 dana izlaganja (d i h)

ter 270 days of outdoor exposure. These cracks are associated with polymer chain scission, which results from shrinking and swelling cycles (Peng *et al*., 2014).

The emergence of cracks on the polymer surface paves the way for the entry of microorganisms. In the presence of sufficient moisture, oxygen, and sunlight, these microorganisms intensify their activities, leading to a darkening of the specimen surfaces. The primary color change on the specimen surfaces can be attributed more to the growth and spread of molds, rather than to the color change of the composite itself. This is because molds, due to their extracellular metabolism, produce melatonin pigments, which increase over time and darken the surface (Muasher and Sain, 2006; Chen *et al*., 2016). Fabiyi and McDonald (2010) outlined three main stages of WPC degradation: the initial formation of tiny cuts and cracks on the composite surface; an increase in the size and number of these cracks along with visible signs of microorganism growth on the surface; and finally, the development of small cracks on the weathered surface following the second stage.

Recycling PP increases the melt flow index (MFI), leading to better mixing of wood flour by PP matrix and improved coating of wood flour by recycled PP. As a result, composites made from recycled PP absorb less moisture compared to those made from virgin PP, leading to less surface degradation and providing less opportunity for the growth of micro-organisms. On the other hand, the addition of UV absorbents to PP composites improved the surface degradation of the composites, although not significantly (Fabiyi and McDonald*,* 2010).

3.2 Contact angle
3.2. Kontaktni kut

The contact angle is a measure of the wettability of a surface. The contact angle can provide insights into the relative hydrophilicity or hydrophobicity of the WPCs. Being a hydrophilic material, polypropylene typically has a higher contact angle than wood flour, which is hydrophobic. After natural weathering, the degradation of PP on the surface of the composites is expected to lead to a significant decrease in the contact angle. This indicates an increase in surface wettability and, consequently, higher moisture absorption. This change in wettability can have implications for the performance and durability of the WPCs, particularly in outdoor applications where they are exposed to environmental changes.

Table 3 presents the contact angle of WPCs made from virgin and recycled PP. As shown, all specimens exhibit the maximum contact angle before weathering due to their smooth and hydrophobic surfaces. However, after 90 to 180 days of exposure to weathering, their contact angles decrease. By the end of the weathering period, due to the intense degradation of the WPCs surfaces, it was not possible to measure the contact angle of a water droplet, because it was quickly absorbed on the surface of the weathered WPCs. Similar results have been published by other researchers (Matuana *et al.,* 2001; Kaci *et al*., 2001; Chen *et al*., 2014; Stokke and Gardner, 2001). The addition of UV absorbers to WPCs increases the contact angles of WPCs made from virgin and recycled plastics before and after natural weathering. It means that the UVA could decrease the wettability of the composites. The difference in the contact angles of the WPCs made of recycled PP and virgin PP was not noticeable (before weathering), but more reduction in contact angles of the WPCs made from recycled PP was observed.

The decrease in contact angles of WPC during exposure to weathering can be attributed to the degradation and oxidation of the PP-rich surface layer and then lignin into water-soluble products. When rain falls and washes away the degraded products from the composite surface, the cellulose ratio on the surface increases. Due to its active hydroxyl groups, the presence of cellulose on the surface increases the composite wettability properties (Williams *et al.,* 2001; Stark and Matuana, 2006).

Cyclic water absorption and desorption by cellulose in the cell wall of wood flour causes cyclic shrinkage and swelling of WPCs. The stress resulting from consecutive shrinkage and swelling cycles causes more cracks on the surface of the polymer matrix. Over time, the number and depth of the cracks on the surface increase, and the wettability properties of the specimens significantly increase. Microscopic images confirm that, as a period of weathering passed, tiny cracks appeared on the surface of the specimens, which somewhat reduced the contact angle of the specimen surfaces. With increasing weathering duration, the number and extent of the cracks increased to the point where deep cuts appeared on the surface of the composites in the last period of weathering, the water droplets rapidly absorbed at the surface of the composites, and it was impossible to measure the contact angle.

3.3 Surface roughness
3.3. Hrapavost površine

Figures 3 and 4 illustrate the surface roughness (*Ra* and *Rz* values) of all WPCs after 270 days of natural weathering. The surface roughness values of composites increased with increasing weathering exposure time. Natural weathering causes chain scission in the polymer matrix, which results in surface cracks and embrittlement. The rain helps in removing and washing degraded polymer from the WPC surface and causes much more wood flour to be directly exposed to UV light. Exposure of wood flour to UV light and water induces numerous checks, splits, and cracks on the surface of WPCs which increase surface roughness. The longer the exposure time, the higher the degradation and the higher the surface roughness.

It can be seen from Figures 3 and 4 that the composites made from recycled PP exhibited lower surface roughness than those made from virgin PP. It can be attributed to better mixing and dispersion of wood flour in recycled PP matrix due to its higher melt flow index (MFI). Consequently, composites made from recycled

Table 3 Effect of natural weathering on contact angle variations of composites
Tablica 3. Utjecaj prirodnog izlaganja vremenskim utjecajima na varijacije kontaktnog kuta na kompozitima

Composite code *Oznaka kompozita*	**Weathering time, day** / *Vrijeme izlaganja, dan*							
	0		90		180		270	
	θ_0	θ_U	θ_0	θ_U	θ_0	θ_U	θ_0	θ_U
WVP	77.3	70.0	66.9	43.4	51.4	47.9	-	-
WRP	76.2	71.4	61.3	48.8	62.8	51.4	-	-
WVP + 0.8 % UVA	80.0	76.4	63.8	44.3	58.8	53.1	-	-
WRP + 0.8 % UVA	80.0	75.9	64.7	54.5	66.9	54.6	-	-

θ_0: Contact angle in time zero / *kontaktni kut u vremenu nula*, θ_U: ultimate contact angle / *krajnji kontaktni kut*

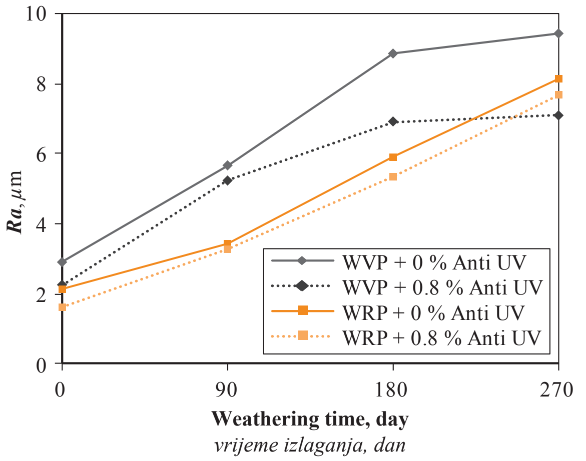

Figure 3 *Ra* variation of WPC surfaces made from virgin and recycled polypropylene
Slika 3. *Ra* varijacije površine WPC-a izrađenoga od čistoga i recikliranog polipropilena

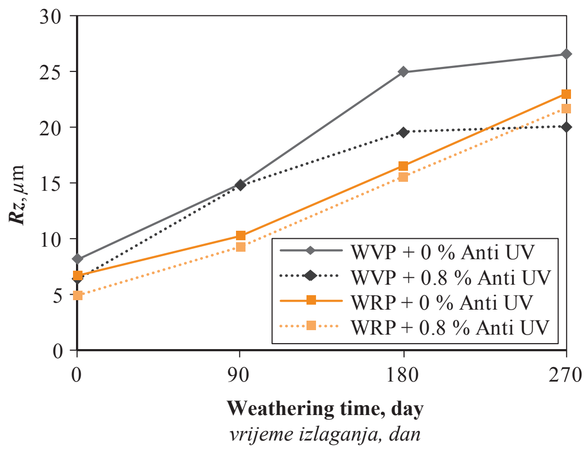

Figure 4 *Rz* variation of WPC surfaces made from virgin and recycled polypropylene
Slika 4. *Rz* varijacije površine WPC-a izrađenoga od čistoga i recikliranog polipropilena

PP absorb less moisture compared to those made of virgin PP, leading to less surface degradation, and providing less opportunity for the growth of microorganisms, and cyclic swelling and shrinkage.

On the other hand, the addition of UV absorbers to WPCS decreased the surface roughness values. A decrease in surface roughness of WPCs containing recycled PP at longer exposure time was noticeable. Improvement of the surface roughness of the WPCs using a photostabilizer has been reported by several researchers (Selden *et al*., 2004; Fabiyi *et al*., 2008; Stark and Matuana, 2003; Rabello and White, 1997).

3.4 Color change
3.4. Promjena boje

The changes in the surface color of WPCs (containing virgin and recycled PP), with and without UV stabilizer, were evaluated based on lightness (L^*) and discoloration after 180 days of exposure to natural weathering. The value of L^* varies between 0 and 100 (black and white, respectively). An increase in L^* indicates that the WPC surface is lightening. The variations of the L^* parameter of WPCs after natural weathering are shown in Figure 5. The lightness (L^*) values increased to maximum values after 60 days of natural weathering for all the WPCs, followed by a gradual decrease up to 180 days. The highest and lowest L^* were obtained for WPC containing recycled PP (without UVA) and WPC containing virgin PP (with UVA), respectively. The highest value of ΔL^* was determined after 60 days of weathering for both WPCs (Figure 6).

Discoloration occurred in two stages: initial photo-bleaching within the first 90 days of exposure, followed by darkening up to the last day of weathering. The photobleaching primarily affected the wood component, particularly lignin, which absorbs 80-90 % of the total amount of light absorbed by wood flour (Stark and Gardner, 2008). The absorption of UV radiation by lignin leads to its degradation and breakdown into car-

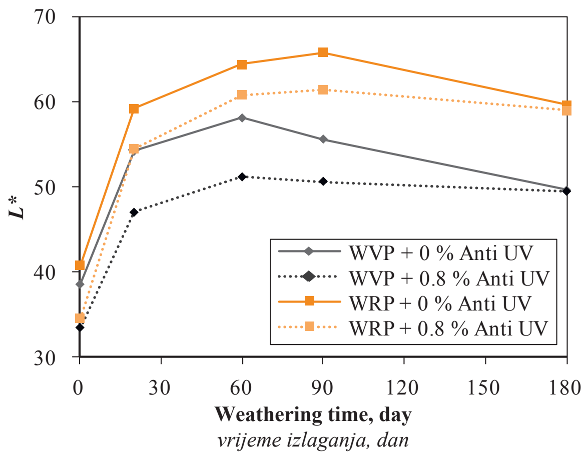

Figure 5 L^* factor variation of WPC made from virgin and recycled polypropylene
Slika 5. Varijacije svjetline WPC-a izrađenoga od čistoga i recikliranog polipropilena

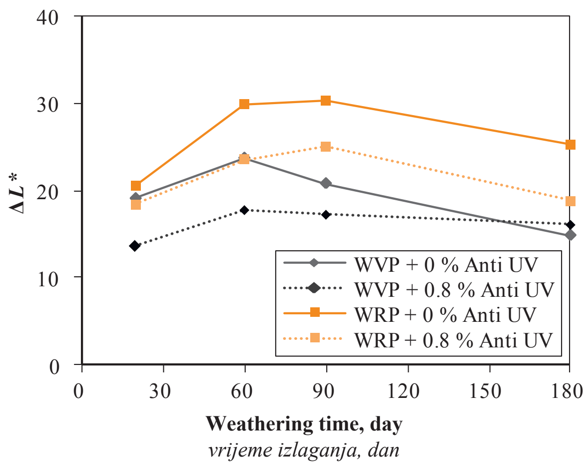

Figure 6 ΔL^* factor variation of WPC made from virgin and recycled polypropylene
Slika 6. Varijacije promjene svjetline WPC-a izrađenoga od čistoga i recikliranog polipropilena

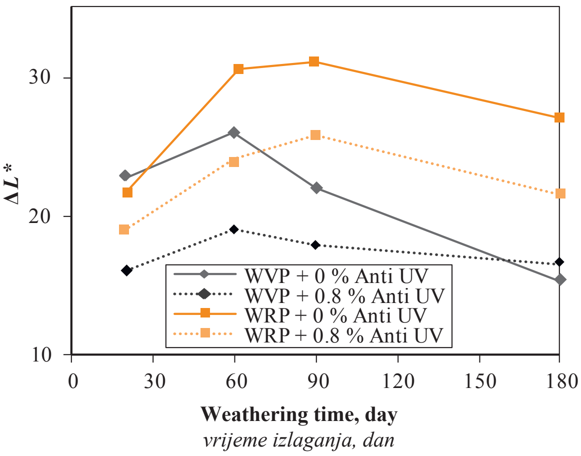

Figure 7 ΔE^* variation of composites made from virgin and recycled polypropylene
Slika 7. Varijacije promjene boje kompozita izrađenoga od čistoga i recikliranog polipropilena

bonyl, carboxyl, and aldehyde chromophores. These are the main factors of light absorption and composite photochemical reactions responsible for the color change (Ayadi *et al*., 2003; Stark, 2006).

Darkening may have been caused by surface oxidation and the presence of mildew on the composite surface. Mildew, a type of fungus, causes surface discoloration due to the production of extracellular metabolism pigments, creating green to red and dark spots (Muasher and Sain, 2005; Rowell *et al.,* 2000).

Composites made from virgin PP showed lower lightness than those made from recycled PP, likely due to the better encapsulation of wood flour in virgin PP, with good dispersion and interfacial bonding between wood flour and polymer.

The result of total color changes or discolorations is shown in Figure 6. The ΔE^* considerably increased in the initial 90 days of exposure to weathering and then slightly decreased up to the last day of weathering. The WPCs containing virgin PP exhibited lower ΔE^* than those containing recycled PP. The addition of 0.8 % UVA reduced the ΔE^* of WPCs but it was more effective for WPCs made from recycled PP.

3.5 FTIR spectroscopy
3.5. FTIR spektroskopija

Fourier Transform Infrared (FTIR) spectroscopy provides a clear insight into material degradation by determining the changes in the surface chemistry of wood plastic composites during weathering. The IR spectra of WPC made from virgin and recycled PP with 8 % UV absorbent, before and after exposure, are depicted in Figure 8 and 9, respectively.

The broad peak around 3400 cm^{-1} band is assigned to hydroxyl groups originating mainly from the cellulose of wood flour (Stark and Matuana, 2004). The peak around 1050 cm^{-1} is associated with both the C-O stretch in cellulose and the C-O deformation in the primary alcohols of lignin. The peaks at 2922 cm^{-1} and around 1450 are due to the C-H stretching and bending of methylene groups, respectively, which appear as strong peaks in PP (Krehula *et al*., 2014). The assigned peak at 1700-1750 cm^{-1} corresponds to carbonyl groups. The absorption at 1630-1650 cm^{-1} belongs to the stretching vibrations of the vinyl groups (C=C) in the polymeric phase (Fabiyi *et al.,* 2008).

After weathering, the intensity of peaks at 3400 cm^{-1} and around 1050 significantly decreased with increasing weathering exposure time indicating a de-

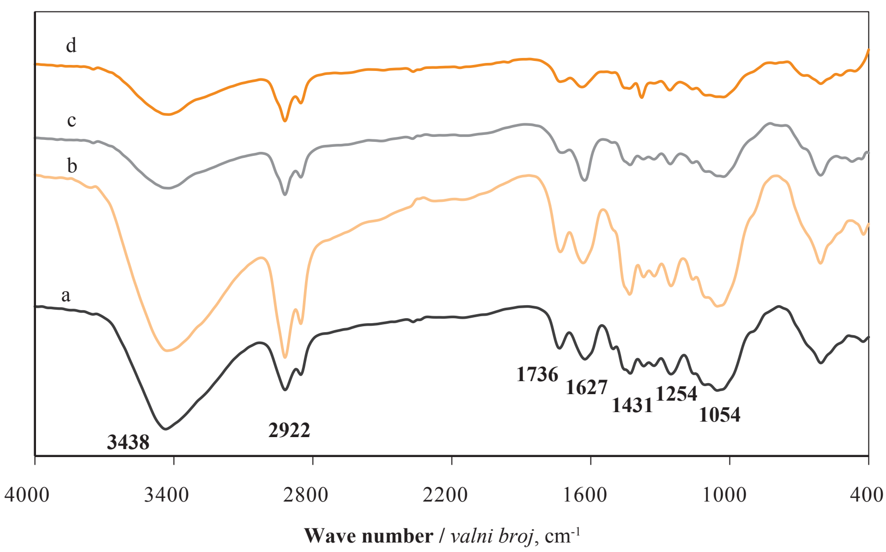

Figure 8 Effect of natural weathering on FTIR spectrum of WPCs made from virgin PP with 0.8 % UVA: before weathering (a), after 90 days of weathering (b), after 180 days of weathering (c), and after 270 days of weathering (d)
Slika 8. Utjecaj prirodnog izlaganja vremenskim utjecajima na FTIR spektar WPC-ova izrađenih od čistog polipropilena s 0,8 % UVA: prije izlaganja (a), nakon 90 dana izlaganja (b), nakon 180 dana izlaganja (c), nakon 270 dana izlaganja (d)

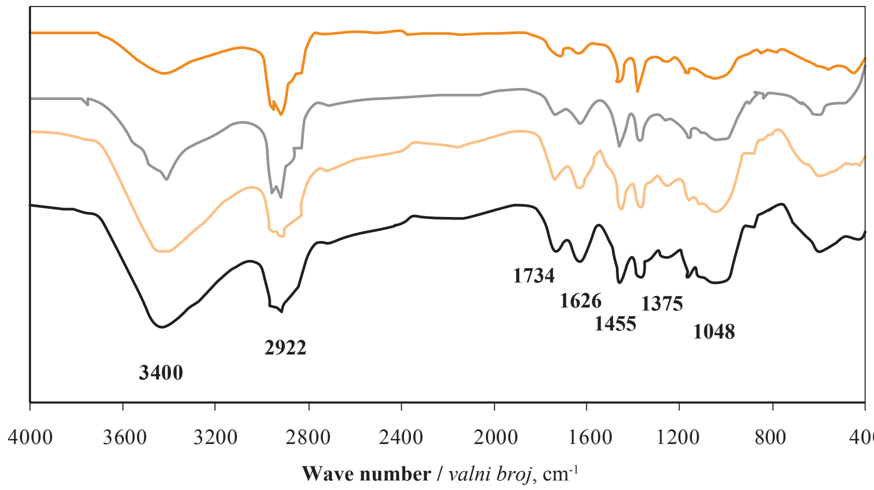

Figure 9 Effect of natural weathering on FTIR spectrum of WPCs made from recycled PP with 0.8 % UVA: before weathering (a), after 90 days of weathering (b), after 180 days of weathering (c), and after 270 days of weathering (d)
Slika 9. Utjecaj prirodnog izlaganja vremenskim utjecajima na FTIR spektar WPC-ova izrađenih od recikliranog polipropilena s 0,8 % UVA: prije izlaganja (a), nakon 90 dana izlaganja (b), nakon 180 dana izlaganja (c), nakon 270 dana izlaganja (d)

crease in hydroxyl groups at the surface of WPCS (Wang *et al*., 2010).

It is well known that the carbonyl groups are formed after PP degradation under UV light, which can be monitored by change in intensity of peak at 1700-1750 cm^{-1} (Fabiyi *et al*., 2008). However, degradation of PP in the first step, manifested as the chain scission, must be detected through the monitoring of peaks absorbance intensity at 2922 cm^{-1} assigned to CH stretching in –CH2- groups (Krehula *et al*., 2014). In this research, no noticeable changes were observed in the intensity peak of 2922, and on the other hand, the intensity peaks at 1700-1750 cm^{-1} were slightly decreased especially in prolonged exposure. This phenomenon can probably be attributed to the washing of the damaged surface of the samples by rainwater and the appearance of a new surface, free of degraded PP.

3.6 Flexural properties
3.6. Svojstva savijanja

Figures 10 and 11 display the flexural strength (*MOR*) and flexural modulus (*MOE*) of WPCs, respectively. It can be seen that the *MOR* of most WPCs slightly increased in the initial 90 days of exposure to weathering and decreased after 270 days. The initial increase in *MOR* can be attributed to crystallization with crosslinking after initial chain scissions. Similar results have been reported by Taib *et al.* (2010) and Silva *et al.* (2017). At higher exposure times, severe chain scission occurs, whereby the crystalline regions are also affected, leading to a decrease in crystallinity and MOR. Additionally, exposure to moisture degrades the mechanical properties of composites due to the cyclic shrinkage and swelling of the wood particles. The stress caused by cyclic shrinkage and swelling of wood particles creates micro-cracks in the matrix, causing a decrease in flexural strength and reducing the efficiency of stress transfer from fiber to the matrix, which results in a decrease in strength (Rangaraj and Smith, 2000; Wei *et al*., 2013; Saputra and Simonsen, 2004).

The composites containing recycled PP exhibited higher *MOR* than those containing virgin PP both before and after weathering. The statistical analysis showed that the differences were significant. The higher *MOR* for recycled plastics-based WPCs has been reported by several researchers (Kazemi-Najafi *et al.*, 2006; Adhikary *et al.*, 2008; Kazemi-Najafi *et al.*, 2009). The higher MOR of recycled PP composites can be attributed to the lower melt viscosity of the recycled PP. Lower melt viscosity yielded a better dispersion of the wood flour and enhanced the mechanical properties. The addition of UVA increased the *MOR* of the

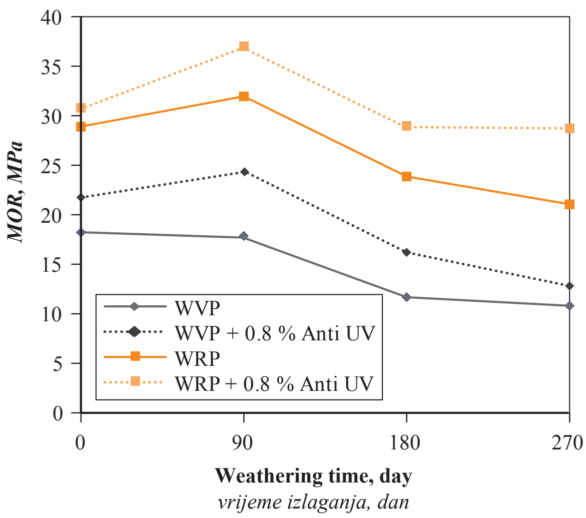

Figure 10 Flexural strength of WPCs made from virgin and recycled polypropylene
Slika 10. Čvrstoća na savijanje WPC-ova izrađenih od čistoga i recikliranog polipropilena

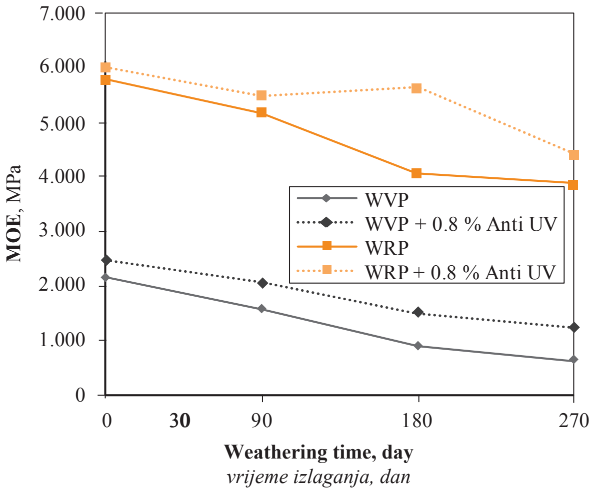

Figure 11 Flexural modulus of WPCs made from virgin and recycled polypropylene
Slika 11. Modul na savijanje WPC-ova izrađenih od čistoga i recikliranog polipropilena

WPC made from both virgin and recycled PP. A similar result has been reported by Ovali and Sancak for natural fiber LDPE composite (Ovali aad Sancak, 2022). They showed that the addition of UV additives improved the flexural strength of the composites between 46 % and 68 %. The UVA could not prevent the loss of flexural strength with increasing weathering exposure time.

Figure 11 shows that the *MOE* of WPCs decreases with increasing exposure time. Exposure to UV lights affected the modulus of the WPC negatively because of matrix polymer crystallinity loss, surface oxidation, and interfacial degradation. Additionally, water absorption during exposure can negatively affect *MOE* of the composites.

The composites made from recycled PP exhibited significantly higher flexural modulus than those made from virgin PP. This phenomenon can be attributed to the better dispersion of wood flour in recycled PP matrix due to an increase in the MFI of PP after thermos-mechanical degradation. Similar to *MOR*, the addition of UVA increased the *MOE* of the WPC made from both virgin and recycled PP. The addition of UVA could not prevent the decrease of the modulus after natural weathering.

4 CONCLUSIONS
4. ZAKLJUČAK

This research examined the impact of natural weathering on the physical and mechanical properties of wood flour polypropylene composites, both virgin and recycled. Both types of composites underwent changes in physical properties and a loss of mechanical properties due to weathering. The use of a UV absorber improved the properties of the composites made from virgin and recycled polypropylene in the short term, but it was not effective in the long term. Natural weathering severely damaged the surface of both types of composites made from virgin and recycled polypropylene. Composites made from virgin polypropylene need more UV absorbers compared to composites made from recycled polypropylene. To achieve higher resistance characteristics in the long term for both types of composites, higher amounts of UV absorbers or simultaneous use of antioxidants and pretreatment of wood flour and polymer are probably needed. This research provides valuable insights into the behavior of these composites under weathering conditions and can guide future efforts to improve their performance. In addition, the results of this research provide the possibility of choosing the appropriate polymer matrix based on the desired physical and mechanical properties of WPCs and for developing various applications of these products.

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Drvo crnog oraha *Juglans nigra* L.

OPĆENITO O VRSTI

Juglans nigra L. vrsta je drva iz porodice *Juglandaceae*. Trgovački su nazivi te vrste American walnut (Velika Britanija); American black walnut, Eastern black walnut, Black walnut (SAD); Canadian walnut, gunwood (Kanada); amerikanische Nuss (Njemačka); Noyer noir (Francuska); Noce nero americano (Italija). Crni orah je vrsta istočnog dijela Sjeverne Amerike. Stabla porodice *Juglandaceae* rastu u umjerenim i suptropskim područjima sjeverne polutke, a nekoliko se vrsta širi i na južnu polutku, na područje od Malezije do Nove Gvineje i zapadnog dijela južne Afrike. Kao vrsta istočnog dijela Sjeverne Amerike, crni orah ima osobito veliku važnost za šumarstvo SAD-a, pa stoga i za drvnu industriju. Neupitno je i njegovo značenje za urbano šumarstvo ne samo u SAD-u nego i u mnogim drugim zemljama umjerene klime diljem svijeta. Crni je orah vrlo zahtjevna vrsta s obzirom na svojstva tla. Drvo crnog oraha tradicionalno je vrlo cijenjeno te se upotrebljava u proizvodnji prvorazrednog namještaja, furnira, kundaka za puške, tokarenih i drugih ukrasnih predmeta. U posebnim primjerima crni se orah može smatrati alternativom ebanovini i drugim listačama tamne boje. Nije na popisu ugroženih vrsta međunarodne organizacija CITES, a prema IUCN-u, vodi se kao vrsta drva najmanje zabrinjavajućeg opstanka.

RELEVANTNE SPOZNAJE O DRVU CRNOG ORAHA

Drvo crnog oraha grube je, ali vrlo ukrasne teksture. Pore i godovi običnim su okom dobro vidljivi. Bjeljika je bijela do žućkastosmeđa, a može biti i siva do sivosmeđa, širine 3 – 7 cm, dok je srž smeđa do ljubičastosmeđa. Boja srži važan je parametar kvalitete drva pri klasiranju furnira crnog oraha.

Pregled ranih istraživanja drva crnog oraha donosi spoznaje (a) o stablima uzgajanim u sastojinama koja bi imala tamnije obojenu srž s uskom zonom bjeljike, a pojedinačna bi stabla na otvorenom prostoru imala svjetlije obojenu srž i širu bjeljiku (Landt i Phares, 1973.); (b) o stupnju i brzini stvaranja srži koji ne ovise o volumenu uključenih anatomskih obilježja (parenhima), već o metaboličkom okviru koji uvjetuje prijelazna zona (Nelson, 1975.; 1976.); (c) o varijabilnosti osvjetljenja (tj. tame ili svjetlosti) koja je primarni uzrok varijacija boje srži (Phelps i dr., 1983.); (d) o

GENERAL INFORMATION ON SPECIES

Juglans nigra L. is a wood species of the *Juglandaceae* family. Common names are: American walnut (Great Britain); American black walnut, Eastern black walnut, Black walnut (USA); Canadian walnut, gunwood (Canada); amerikanische Nuss (Germany); Noyer noir (France); Noce nero americano (Italy). Black walnut is a species of the eastern part of North America. Members of the *Juglandaceae* family inhabit the temperate and subtropical regions of the Northern Hemisphere, and several species migrate to the South, spreading from Malaysia to New Guinea and the western part of South Africa. As a species of the eastern part of North America, black walnut has a very high importance especially for the forestry of the USA, therefore also for the wood industry. It is also an important species for urban forestry, not only in the USA, but also in many other countries in temperate climates around the world. Black walnut is a tree species with high demands on soil properties. Black walnut wood is traditionally highly valued and is used in the production of first-class furniture, veneers, rifle stocks, turned and other decorative items. In limited situations, black walnut can sometimes be considered as an alternative to ebony and other dark colored hardwoods. This wood species is not listed in the international organization CITES Appendices and is reported by the IUCN as a species of least concern.

RELEVANT KNOWLEDGE ABOUT BLACK WALNUT WOOD

Black walnut wood is considered to be coarse-textured, but also very decorative. Vessels and annual growth rings are clearly visible to the naked eye. The sapwood is white to yellowish brown, and can be gray to gray-brown, 3-7 cm wide, while the heartwood is brown to purple-brown. Heartwood color is an important wood quality parameter in grading black walnut veneer.

A review of early research on black walnut wood provides the knowledge on: (a) the forest-grown stems that would have a dark-colored with a narrow sapwood band, while open-grown trees would have lighter heartwood and wider sapwood rings (Landt and Phares, 1973); (b) the scale and rate of heartwood formation not depending on the volume of anatomical resources involved (parenchyma) but on the metabolic frame proniskoj nasljednosti boje srži drva, što sugerira da čimbenici okoliša i/ili uzgoja imaju bitnu ulogu u određivanju toga važnog parametra kvalitete drva (Rink, 1987.); (e) o gustoći srži koja je bila u negativnoj korelaciji s bojom srži, a u pozitivnoj korelaciji s ukupnom visinom stabla, dok gustoća bjeljike nije korelirala s ukupnom visinom (Rink i Phelps, 1989.); (f) o brže rastućim plantažnim stablima koja su imala šire godove, sa širom zonom kasnog drva (Phelps i Workman, 1992.).

Novija istraživanja crnog oraha rezultirala su zaključcima: a) o brzini rasta, za koju je utvrđeno da su na nju snažno utjecale kontrola korova i uzgojne metode (Cutter i Garrettz, 1993.); b) o općenito boljem prirastu oraha kada se sadi s drugim drvenastim vrstama (Pedlar i dr., 2006.); c) o dosezanju vrhunca visine i promjera stabala prije starosti od 30-35 godina (Nicolescu i dr., 2020.); d) o populacijama iz toplih klima koje imaju višu maksimalnu apsolutnu stopu rasta i dosežu je ranije nego populacije iz hladnih klima (Onofrio i dr., 2021.).

Napomena: Podatci o tehničkim i tehnološkim svojstva drva crnog oraha dostupni su na web stranicama i u priručnicima navedenima u literaturi ovog prikaza.

VAŽNOST CRNOG ORAHA I ISTRAŽIVANJA TE VRSTE U HRVATSKOJ

Crni orah je vrsta drveća koja je u Europu unesena 1629. godine. Danas ima važnu gospodarsku ulogu u proizvodnji drva i plodova u agrošumarskim sustavima, kao ukrasno drvo u parkovima i avenijama te kao vrsta za sanaciju/obnovu devastiranih zemljišta. Stabla su impresivnih dimenzija te se od njih dobiva vrlo kvalitetno, trajno i vrijedno drvo, a u Hrvatskoj raste i u kulturama. Tradicija uzgoja kultura crnog oraha na području istočne Slavonije dulja je od stotinu godina. Crni se orah počeo uzgajati na staništima nizinske Hrvatske koja su previše suha za uzgoj najvažnije domaće vrste – hrasta lužnjaka. Kulture crnog oraha uz rijeku Dunav daju izvrsne šumskogospodarske i s ekonomskog stajališta više nego opravdane rezultate. Stoga Mayer i Rajković (2008.) u monografiji o crnom orahu u Podunavlju proširuju znanja o crnom orahu među stručnjacima šumarske i drvnotehnološke struke, detaljno prikazuju podatke o prirodnom staništu, biologiji i uporabi te vrste u njezinu podneblju te donose svoje spoznaje o crnom orahu u Hrvatskoj.

Rana istraživanja o plantažama crnog oraha u Hrvatskoj donose Sevnik (1926.) i Krajina (1973.). U novije vrijeme o osnivanju kultura crnog oraha pisao je Mayer (2011.), a o rasadničkoj proizvodnji sadnica crnog oraha Oršanić i dr. (2007.). Provedeno je i istraživanje rasta i prirasta crnog oraha u istočnoj Hrvatskoj

vided by the transition zone (Nelson, 1975; 1976); (c) the variability in luminance (i.e., darkness or lightness) being the primary cause of heartwood color variability (Phelps *et al*., 1983); (d) low heritability of heartwood color suggesting that environmental and/or management factors play a significant role in determining this important wood quality parameter (Rink, 1987); (e) heartwood density that was negatively correlated with heartwood color and positively correlated with total height, while sapwood density was not correlated with total height (Rink and Phelps, 1989); (f) faster-growing plantation trees that had wider growth rings, which in turn had wider latewood zones (Phelps and Workman, 1992).

Later research concludes the following: (a) growth rate was found to be strongly affected by weed control and cropping practices (Cutter and Garrett, 1993); (b) generally better walnut growth when interplanted with other woody species (Pedlar *et al.*, 2006); (c) tree height and diameter growth reach their peaks before the age of 30–35 (Nicolescu *et al.*, 2020); (d) populations from warm climates have higher maximum absolute growth rate and reach it earlier in age compared to populations from cold climates (Onofrio *et al*., 2021).

Note: data on technical and technological properties of black walnut wood are available on web pages and in manuals listed in the reference section.

IMPORTNACE OF BLACK WALNUT AND ITS RESEARCH IN CROATIA

Black walnut is a tree species introduced in Europe in 1629. Nowadays, it has an important economic role in the production of wood and fruits in agroforestry systems, as an ornamental tree in parks and avenues, and in the rehabilitation/restoration of degraded lands. Trees have impressive dimensions and produce high-quality, durable and valuable wood; in Croatia it is also grown in plantations. The tradition of growing black walnut trees in the area of Eastern Slavonia started more than a hundred years ago. Black walnut was introduced on the sites of lowland Croatia, which were too dry for growing the most valuable indigenous species, pedunculate oak. Along the Danube river, black walnut plantations give excellent forest management results and more than good results, from an economic point of view. For this reason, Mayer and Rajković (2008), in their monography on black walnut in the Danube region, expand knowledge about black walnut among forestry and wood technology experts, giving detailed information about the natural habitat, biology and use of this species in its climate, and make their own conclusions about black walnut in Croatia.

Early research on black walnut plantations in Croatia was reported by Sevnik (1926) and Krajina

(Čavlović i dr., 2007.) radi određivanja uvjeta u kojima je ta vrsta visokoprinosna. Kremer i dr. (2008.) potvrđuju veliki uzgojni potencijal crnog oraha u Hrvatskoj te upućuju na daljnja istraživanja rasta te vrste, osobito na području istočne Hrvatske. Do sada nema dostupnih podataka o kvaliteti drva crnog oraha u Hrvatskoj. Međutim, na Fakultetu šumarstva i drvne tehnologije u tijeku su istraživanja strukture i svojstava drva crnog oraha iz kultura s područja Podunavlja koja će pokazati kvalitetu proizvedene sirovine.

(1973). More recently, Mayer (2011) wrote about the establishment of black walnut plantations, and Oršanić *et al*. (2007) about nursery production of black walnut seedlings. Research on the growth and increment dynamics of black walnut in eastern Croatia was also carried out (Čavlović *et al*., 2007), with the aim of determining the conditions in which this species is highly productive. Kremer *et al*., (2008) confirmed the great breeding potential of black walnut in Croatia and pointed to further research on the growth of this species, especially in areas of eastern Croatia. So far, there has been no information available on the black walnut wood quality in Croatia. However, research on the structure and properties of black walnut wood from plantations in the Danube area is in progress at the Faculty of Forestry and Wood Technology, and is expected to show the quality of the produced raw material.

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Upute

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

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

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

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

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

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

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

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

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

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

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

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

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

Primjeri navođenja literature

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

Primjer

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

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

Primjeri

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

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

Ostale publikacije (brošure, studije itd.)

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

Web stranice

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

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

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

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HRVATSKA KOMORA INŽENJERA ŠUMARSTVA I DRVNE TEHNOLOGIJE

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