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LES/WOOD

## UVODNIK / EDITORIAL

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### Revija Les/Wood izhaja pod okriljem Založbe Univerze v Ljubljani

Univerza v Ljubljani je s 40.000 študentkami in študenti največja in najstarejša univerza v Sloveniji. Sestavlja jo kar 26 fakultet in akademij in pokriva vsa znanstvena področja. Posledično je bogata tudi njena založniška dejavnost, ki pa je zelo razvejana. Konec študijskega leta 2019/2020, ko je Univerza praznovala 100. obletnico, so se uresničile dolgoletne želje, da bi Univerza dobila skupno založbo. Nastala je Založba Univerze v Ljubljani (University of Ljubljana Press), ki s svojevrstno organiziranostjo povezuje založniško dejavnost na fakultetah in akademijah. Osnova Založbe UL sicer ostaja založniška dejavnost na članicah UL, pomemben korak pa smo naredili z začetkom izdajanja publikacij pod skupnim imenom Založba UL. Ko bo ta proces v celoti končan, bo šele res razvidno, da je Založba UL ena večjih evropskih univerzitetnih založb, ki letno izdaja več kot 250 monografij.

V okviru Univerze v Ljubljani izhaja tudi 50 mednarodnih znanstvenih revij. Skoraj vse revije izhajajo v odprtem dostopu, kar pomeni, da so članki dostopni takoj po izidu posamezne številke. Ker so bile do pred kratkim revije objavljene na najrazličnejših naslovih in platformah, je Založba UL vzpostavila skupni spletni portal Revije UL. Portal temelji na odprtokodnem sistemu Open Journals Systems, ki poleg objave posameznih člankov v ozadju omogoča tudi vse potrebne založniške korake za urednikovanje (elektronsko sprejemanje člankov, posredovanje člankov recenzentom, oblikovalcem, lektorjem itd.). Portal je dostopen na naslovu https://journals.uni-lj.si/. Na portalu trenutno gostuje 19 revij, objavljenih pa je več kot 6.500 člankov.

Ena izmed takih revij je tudi revija *Les/Wood*, ki od leta 2017 dvakrat letno izhaja z odprtim dostopom po diamantnem modelu. Revija sicer nadaljuje zapuščino revije *Les: revija za lesno gospodarstvo*, ki je neprekinjeno izhajala med leti 1949 in 2013. Revija obravnava širše področje lesarstva in z lesarstvom povezana področja, teme, ki so še kako pomembne za trajnostno naravnan odnos do današnjega sveta. Les je namreč edina obnovljiva surovina, ki jo imamo v Sloveniji v izobilju, zato je še toliko bolj pomembno, da tudi preko znanstvenih člankov podpiramo poglobitev znanj o lesu in lesnih kompozitih ter sodobnih tehnologijah za predelavo in obdelavo lesa, hkrati pa tudi spodbujamo raziskave o konstruiranju in oblikovanju ter gospodarjenju z lesom in lesnimi proizvodi. Sedež uredništva revije *Les/Wood* je na Biotehniški fakulteti Univerze v Ljubljani, z letom 2021 pa je revija začela izhajati pod imenom Založbe Univerze v Ljubljani. Verjamemo, da bo s tem korakom revija *Les/Wood* pridobila večjo mednarodno prepoznavnost in tako še okrepila svojo interdisciplinarno naravnanost.

Uredništvu revije *Les/Wood* na tem mestu izrekam pohvale za vse opravljeno delo, zaradi katerega revija dosega visoke akademske založniške standarde. S skupnimi močmi bomo tako še bolj spodbudili globalno izmenjavo znanja in tako pripomogli k bolj trajnostnemu razvoju sodobnega sveta.

## The journal Les/Wood is published by the University of Ljubljana Press

With some 40,000 students, the University of Ljubljana is the largest and oldest university in Slovenia. It includes 26 faculties and academies, and covers all scientific disciplines. Consequently, its publishing activity is also very rich and wide-ranging. At the end of the academic year 2019/2020, when the University of Ljubljana celebrated its 100th anniversary, a long-held wish came true: that the university would have a single, united publishing house. The University of Ljubljana Press was thus established, joining the publishing activities of the faculties and academies through a unique organisational structure. While the basis of the University of Ljubljana Press remains the publishing activities of the faculties, an important step has been taken by starting to publish our publications under the common name of the University of Ljubljana Press. When this process is fully completed, it will become clear that the University of Ljubljana Press is one of the largest university presses in Europe, publishing more than 250 monographs per year.

The University of Ljubljana also publishes 50 international scientific journals. Almost all of the journals are open access, meaning that articles are available immediately after the publication of each issue. Since the journals have been published on a variety of websites and platforms, the University of Ljubljana Press has initiated a single web portal for the UL journals. The portal is based on the Open Journals Systems, which also provides all the necessary publishing steps for editors (electronic submission of articles, peer reviewing, etc.). The portal is available at https://journals.uni-lj.si/. The portal currently hosts 19 journals and has published more than 6,500 articles.

One of these journals is *Les/Wood*, which has been published twice a year in the open access diamond model since 2017. The journal continues the legacy of *Wood: The Journal of the Wood Economy* (*Les: revija za lesno gospodarstvo*), which was published continuously between 1949 and 2013. The journal publishes original research articles that explore the broad field of wood science and technology, topics that are particularly relevant to a sustainable approach in today's world. Wood is the only renewable resource we have in abundance in Slovenia. Therefore, it is very important to promote knowledge about wood and wood composites, as well as modern technologies for wood processing and treatment, and also to support research in construction and design, as well as the management of wood and wood products. The editorial office of *Les/Wood* is located at the Biotechnical Faculty of the University of Ljubljana and since 2021 the journal is published under the brand the University of Ljubljana Press. We believe that this step will help the journal gain greater international visibility and thus strengthen its interdisciplinary focus.

I would like to congratulate the editors of *Les/Wood* for all the work they have done to ensure that the journal achieves the high standards of academic publishing. Together, we will continue to promote the global exchange of knowledge and thus contribute to a more sustainable development of the modern world.

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# THE SYNERGISTIC EFFECT OF MICROWAVE DRYING AND PLASMA SURFACE TREATMENTS ON THE WETTABILITY OF GREEN WOOD

# SINERGISTIČNI UČINEK MIKROVALOVNEGA SUŠENJA IN OBDELAVE S PLAZMO NA OMOČLJIVOST SVEŽEGA LESA

Sauradipta Ganguly[1], Jure Žigon[2], Kavyashree Srinivasa[2], Marko Petrič[2], Sebastian Dahle[2*]

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## *Abstract / Izvleček*

**Abstract:** In spite of being both a one-step solution to several problems associated with woodworking and also energy efficient, the application of microwave (MW) modification in wood research has remained very limited and this promising method has practically no use in wood industries across the globe. Research done so far in this field primarily sheds light on its potential in enhancing wood permeability, treatability and uniform wood drying. While MW treatments are mostly used on wet or green wood, another modification technique, plasma, has potential benefits to synergistically enhance the effects of MW treatment, but so far has not been applied on wet or green wood specimens. This study takes a first step to investigate the effects of plasma treatments (PT) on green wood specimens, as well as combinations of MW and plasma treatments. As a preliminary study, the methodology focuses on water contact angle measurements, since these are most commonly used as indicators for surface modifications in industrial applications. An exponential time dependence was found for the contact angle on the investigated samples of Norway spruce (*Picea abies* Karst.). Initial contact angles after droplet deposition increased due to drying and migration of organic molecules during treatments. In comparison with the literature, the effect of plasma was significantly less pronounced on wet wood specimens. The initial contact angles showed the lowest statistical variations after MW treatment, whereas plasma increased inhomogeneities. The final contact angles on treated specimens was lowest for PT-only specimens as well as specimens treated with plasma after MW. In contrast to the initial contact angles, the final contact angles showed the lowest variations after PT. Wetting rates were insignificantly improved by plasma, with reduced statistical variations after all treatments.

**Keywords:** Norway spruce wood, Wood drying, Microwave processing, Gliding arc plasma

**Izvleček:** Kljub temu, da je uporaba mikrovalov za modifikacijo lesa energetsko učinkovita in obetavna enostopenjska rešitev za številne probleme, povezane z obdelavo lesa, je ta tehnologija v raziskavah lesa ostala zelo omejena in se v lesni industriji po vsem svetu praktično ne uporablja. Dosedanje raziskave na tem področju osvetljujejo predvsem potencial te metode pri izboljšanju permeabilnosti lesa, kurativne zaščite lesa in enakomernega sušenja lesa. Medtem ko se obdelava z mikrovalovi večinoma uporablja na svežem lesu, izkazuje tehnika obdelave lesa s plazmo potencialne koristi za sinergistično izboljšanje učinkov obdelave z mikrovalovi, vendar se doslej plazma še ni uporabljala za obdelavo svežega lesa. Ta študija je prvi korak k raziskovanju učinkov plazemske obdelave na vzorcih svežega lesa ter kombinacije mikrovalovne in plazemske obdelave. Kot preliminarna študija se osredotoča na meritve stičnega kota kapljic vode, saj se metoda uporablja kot indikator učinkovitosti površinske obdelave tudi v industriji. Na raziskanih vzorcih smrekovine (*Picea abies* Karst.) je bila ugotovljena eksponentna odvisnost stičnega kota od trajanja omočenja. Začetni stični kot po nanosu kapljic vode se je povečal zaradi sušenja lesa in migracij organskih molekul na površino lesa. V primerjavi z navedbami iz literature je bil učinek plazme na svežih vzorcih lesa bistveno manj izrazit. Začetni stični kot je imel najmanjšo statistično variabilnost po obdelavi z mikrovalovi, medtem ko je plazma povečala nehomogenost. Končni stični kot je bil najnižji pri vzorcih, ki so bili obdelani samo s plazmo, kot tudi pri vzorcih, obdelanih s kombinacijo mikrovalov in plazme. V nasprotju z začetnim stičnim kotom je končni stični kot pokazal najmanjšo spremembo po obdelavi s plazmo. Stopnja omočitve je bila s plazmo neznatno izboljšana, z zmanjšano statistično variabilnostjo po vseh obdelavah.

**Ključne besede:** les navadne smreke, sušenje lesa, mikrovalovna obdelava, drsna obločna plazma

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## 1 INTRODUCTION
## 1 UVOD

Frequent availability, versatility, aesthetics and many other favourable characteristics made wood the most important biomass derived material for a varied range of end uses. The diverse nature of several wood species has resulted in their specific uses, which in the present day is difficult to maintain due to the depleting supplies of timber resources worldwide. Wood modification is the most suitable solution to this, where using various methods secondary and non-durable wood can be made suitable for specific uses and its sustainability thus ensured for longer periods. Among these techniques, thermal and chemical modifications of timber have become relatively common (Hansmann et al., 2005; Despot et al., 2008; Esteves & Pereira, 2009; Sefc et al., 2009; Hom et al., 2020; Treu et al., 2020) across the globe due to their ease of processing and satisfactory commercial and laboratory performance. However, extensive energy consumption and the utilisation of non-environmentally-friendly chemicals are the two most common drawbacks associated with these methods. In the last few decades treating and modifying wood with microwaves (MW) has become more popular (Kol & Çayır, 2021) in the niche research domain of wood modification with a variety of use cases (Oloyede & Groombridge, 2000; Torgovnikov & Vonden, 2010; Balboni et al., 2018; Samani et al., 2019; Weng et al., 2021). For timber drying, for example, it has been shown to have a lower level of energy consumption (Brodie, 2010; Sethy et al., 2016) in comparison to the conventional methods, with an increased drying rate of timber and subsequently reduced the drying time (Awoyemi, 2004; Gamben et al., 2005). Several studies demonstrated strongly increased liquid permeability and preservative uptake in refractory and moderately refractory timber species after several levels of MW modification and pre-treatment (Liu et al., 2005; Treu & Gjolsjo, 2008; Torgovnikov & Vinden, 2009; Ramezanpour et al., 2015; Xu et al. 2015; Hermoso & Vega, 2016; Samani et al., 2019; Ganguly et al., 2021a). This was shown to originate primarily in the rupture of weak anatomical elements leading to the generation of micropores and microcracks (Mekhtiev & Torgovnikov, 2004; Terziev et al., 2020; Weng et al., 2020; Ganguly et al., 2021b) as well as an increment in pore diameters along with destruction of pit membranes and damage to cell walls. However, this change in the wood microstructure upon exposure to higher intensities of MW often compromises the strength of timber (He et al., 2014), although there have been cases where the reduction is not statistically significant (Kol & Çayır, 2021). During the MW treatment of wooden materials, the build-up of internal gas pressure from vapourized water and volatile organic compounds needs to be considered, as it can increase permeability through microcracks in the cell walls (Hong-Hai et al., 2005) or negatively impact the workpiece's mechanical properties through material cracking (Ouertani et al., 2015) and other issues related to inhomogeneous rises in temperature (Bartoli et al., 2019). As such, the optimization of the treatment parameters is of utmost importance when using MW for wood modification.

Another kind of modification technique with the potential to increase the permeability of wood and uptake of liquids is the use of non-thermal plasmas (Žigon et al., 2018; Žigon et al., 2020a; Wascher et al., 2021). Plasma treatments (PT) have been used to improve wetting and uptake of both water and non-polar liquids (Rehn et al., 2003; Žigon et al., 2018; Haase et al., 2019), to improve coatings' adhesion strengths (Riedl et al., 2014) and to enhance the performance of adhesive joints (Žigon et al., 2020b; Krapež Tomec et al., 2021). Moreover, PT have been reported to remove volatile organic compounds (VOCs) and moisture from the parts of wooden specimens near the surface (Avramidis et al., 2016; Dahle et al., 2020a). The main impact of PT, however, is yielded through chemical modification of the wood's lignin and cellulose (Klarhöfer et al., 2010). In contrast to MW treatments depositing a larger proportion of energy inside the workpiece due to the drying gradient, PT almost exclusively modifies the workpiece's surface, reportedly with a maximum modification depth of ca. 3300 nm (Král et al., 2015). While the majority of research on the PT of wood over the past two decades utilizes dielectric barrier discharge (DBD) plasma devices (Žigon et al., 2018), the gliding arc jet plasma technology is much more widespread across most industrial fields and has been successfully utilized on wood and wood-based materials before (Melamies, 2014; Hämäläinen & Kärki, 2013). Therefore, this study focuses on gliding arc jet PT.

In this study, we provide the first results on the combined effects of MW and PT to modify wetting and liquid uptake. In addition to this, the study expands the insights from the PT of dried or conditioned wood specimens to green (i.e. wet) wood, which is most commonly used for MW modifications. The impact of combined modifications of green Norway spruce wood using PT either before or after MW is evaluated against the effect of single modifications of only PT or MW, as well as unmodified control specimens. These relations are investigated through water contact angle (WCA) measurements, as these are the most common indicator for applied surface processes that represent surface functionality, chemistry, morphology, roughness and so on (Kalnins et al., 1988; Boehme & Hora, 1996; Papp & Csiha, 2017).

## 2 MATERIALS AND METHODS
## 2 MATERIALI IN METODE

Norway spruce (*Picea abies* Karst.) wood, 35 years old, cut in the north-eastern part of Slovenia, was used in this study. Conversion of the logs to planks was performed at a local sawmill. Processing (sawing, planing) of the samples was done three weeks after cutting the tree. Separate sets of samples, mostly semi-radial in orientation, were prepared from the heartwood portions. The initial moisture content (IMC) was determined by the gravimetric method according to the EN 13183-1 standard for 10 randomly selected parallel heartwood samples, yielding an IMC of 27–38 %. The dimensions of the samples were l×w×h = 5×2.5×1.5 $cm^3$ for all treatment combinations.

The treatment of wood with microwaves (MW) was carried out using a MW oven (Model: M020MW, Gorenje, Velenje, Slovenia) with a frequency of 2.45 GHz and a maximum output of 700 W at a power consumption of 1500 W. One energy level of 1260 $MJ/m^3$ was applied to the wood samples for all the treatments involving MW in the study. The duration of MW irradiation was 36 s. The parameters of MW treatments were defined based on MW power and volume of the samples as proposed by (Kol & Çayir, 2021) and our preliminary experiments. After MW treatment, the samples were cooled in a desiccator at room temperature and subjected to further analysis.

A gliding arc plasma jet device was used to treat the specimens' surfaces as depicted in Figure 1. The device consists of a computerized numerical control (CNC) positioning system (SainSmart Genmitsu CNC Router 3018 DIY, Vastmind LLC, Delaware, USA) moving the head with attached plasma jet in three directions. Copper electrodes (ROLOT 605, Rothen-berger Werkzeuge GmbH, Kelkheim, Germany) are mounted to a 42 mm diameter cylindrical epoxy (Herpelin Epoksi 1000, Amal d.o.o., Ljubljana, Slovenia) nozzle with an 8 mm diameter centred hole as the gas channel. The gas is supplied from an internal compressor (Hailea ACO 208, Guangdong Hailea Group Co., Ltd., Guangdong, China) at a flow rate of approx. 35 l/min. The plasma discharge is generated between the two electrodes within the nozzle using a commercial high voltage module (ZVS_Driver_20A_kit_AC, Voltagezone Electronics e.U., Graz, Austria) operated at an input voltage (20 V) and current (5 A) from a combination of a commercial switch-mode power supply (Joylit S-240-24, Shenzhen Zhaolan Photoelectric Technology Ltd., Shenzhen, China) and a digital power supply control unit (RD DPS5020 BT/USB, Hang-zhou Ruideng Technology Co., Ltd., Zhejiang, China). The afterglow of the gliding arc jet extends approx. 2 cm out below the nozzle. During the treatment process, the specimens were placed on the stage with a gap distance between the nozzle outlet and sample surface of 10 mm. The entire surfaces of the samples were treated by the plasma jet scanning in seven lines of 80 mm length, offset by 5 mm, thus covering an area of 80 mm × 30 mm at a moving speed of 60 mm/min. The power consumption of the plasma device is 57 W for the electronic components (SainSmart controller, Raspberry Pi 4B with touchscreen, and power supply standby consumption), up to 14 W for the stepper motor movements, 18 W for the compressor unit and up to 100 W for the plasma discharge, yielding an overall power consumption during the treatment of up to 189 W. The entire construction details are available in Dahle et al. (2020b). The G-code file is provided together with the raw and analysed data of this publication in Dahle et al. (2021).

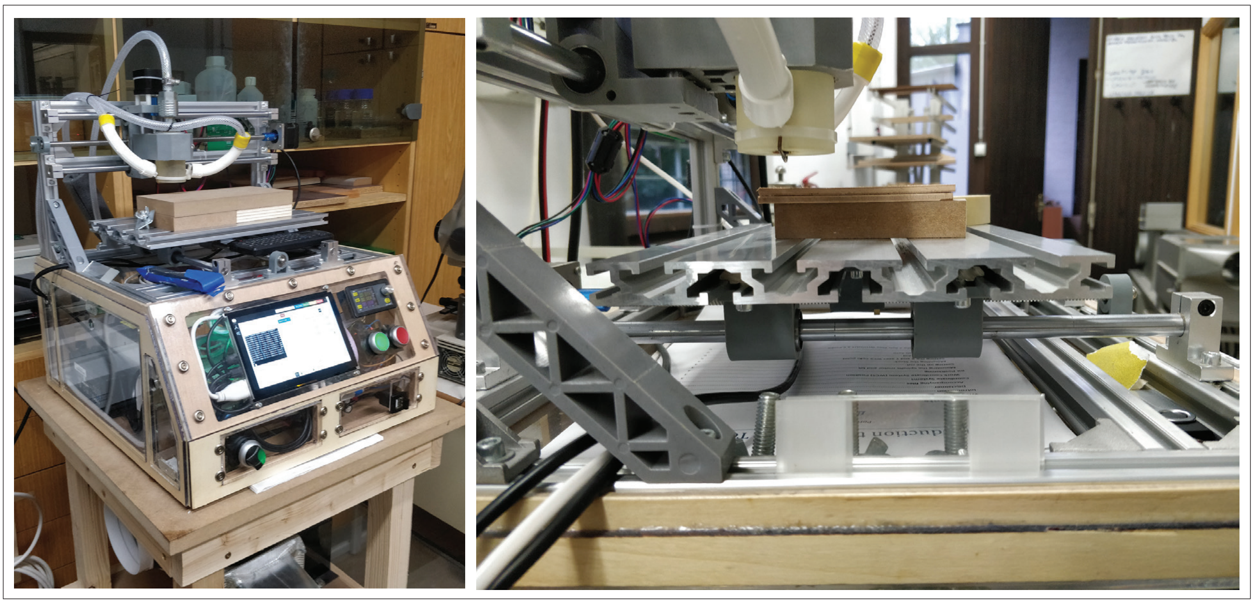

*Figure 1. Gliding arc jet device on a CNC positioning stage.*
*Slika 1. Naprava z drsnim obločnim plazemskim curkom in računalniško krmiljeno pozicionirno mizico.*

WCA were measured using a Theta optical goniometer (Biolin Scientific Oy, Espoo, Finland). Apparent WCAs were evaluated by Young–Laplace analysis using proprietary software (OneAttension version 2.4 [r4931], Biolin Scientific). For each sample type, three specimens were prepared. On each specimen, three droplets were automatically analysed within 63 s, with 1.7 images per second. The measurement started immediately after the first contact of the drop with the surface of the sample. No stable drop shape or equilibrium was achieved within a reasonable time, as opposed to typical findings on dried wood samples (c.f. Kalnins et al., 1988), thus an analysis of time-dependent data was carried out. The data was analysed using OriginPro 2018G 64bit SR1 (OriginLab Corp., Northampton, MA, USA) by numerical fitting a first order exponential decay according to eq. (1), thus yielding the initial contact angles and the subsequent wetting rates. The function was chosen empirically due to the measured data strongly deviating from other published slopes, such as the linear regression with the square root of time reported by Boehme and Hora (1996).

$$y = A_1 * exp\left(-x / t_1\right) + y_0 \qquad (1)$$

Figure 2 depicts the curve according to eq. 1 as a bold black line, with the corresponding parameters indicated by coloured lines. Parameter $y_0$, shown as dashed blue horizontal line, represents the value after indefinitely long equilibration times ($t \rightarrow \infty$). Due to penetration into the wood, this value cannot be physically reached on real specimens, but is suitable as a quantitative indicator for the

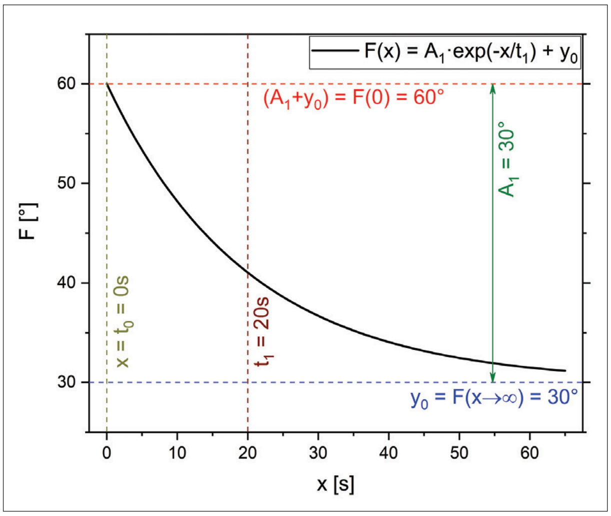


*Figure 2. Example curve with exponential decay according to equation 1.*
*Slika 2. Primer eksponentno pojemajoče krivulje po enačbi 1.*

droplet's behaviour and this is referred to as the final WCA. Parameter $A_1$, shown as vertical green arrow line, represents the difference between the contact angle at $t_0$ = 0s and the value for $t \rightarrow \infty$. The intuitive value discussed in the manuscript, however, is the WCA directly upon the first contact of the droplet with the specimen's surface ($t_0$ = 0s), which is given by $A_1+y_0$, shown as dashed red horizontal line, and this is referred to as the initial WCA. The third parameter $t_1$, shown as a dashed brown vertical line, represents the time, after which the value of the function has fallen from $F(0s)=(A_1+y_0)$ down to $F(t_1)=(A_1/e+y_0)$, i.e. by approx. 63.2% of $A_1$. The smaller parameter $t_1$, the faster the droplet spreads across the surface, i.e. the higher the wetting rate. Parameter $t_1$ is thus referred to as the time constant of wetting.

## 3 RESULTS AND DISCUSSION
## 3 REZULTATI IN RAZPRAVA

Figure 3 shows the measurement data and corresponding fits of exponential decay functions (eq. 1) on different spots of two MW-treated spruce specimens. The time-dependent progression of the measured WCAs varied strongly between individual points on all specimens regarding the initial contact angles directly after droplet deposition and the slope of the curve (i.e. time-dependent wetting), as well as the final WCA. Moreover, all measurements show deviations from the continuous curvature beyond the statistical variations of individual values.

Figure 4 shows a statistical analysis of the final WCA as given by parameter $y_0$ in eq. 1. The results are displayed such that the small square marks indicate the statistical mean value averaged over all measurements and specimens of the corresponding sample type, the coloured boxes enclose the 25%-75% medians, and the metering lines indicate 1.5 IQR (interquartile range), whereas outliers are marked by black diamonds.

It is noteworthy that the smallest final WCAs were determined for the untreated control specimens with an average of 33.1°, which can be accounted for by the high IMC. Both MW and PT yield a drying of the specimens or their surfaces, thereby reducing the proportion of polar molecules within the surface. The highest final WCAs were found for specimens directly after MW treatment with an average of 48.4° and 48.6° for MW-only and MW after PT, respectively. This effect might be due to the drying, which is known to change the character of wood from hydrophilic to hydrophobic, with surfaces becoming more hydrophobic particularly at increased temperatures due to the migration of extractives to the surface (c.f. Šernek, 2002). Directly after PT, the average final WCA amounts to 33.1° and 44.5° for plasma-only and PT after MW, respec-

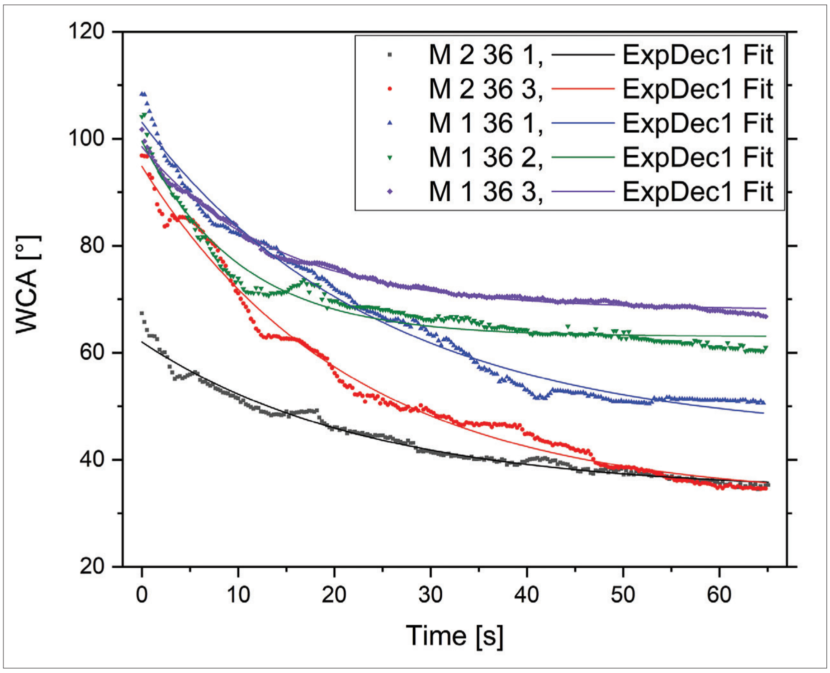


*Figure 3. Example WCA measurement and exponential curve fit for MW-treated specimens.*

*Slika 3. Primeri časovnega spreminjanja izmerjenega stičnega kota vode in prilagojene eksponentne krivulje za vzorce, obdelane z mikrovalovi.*

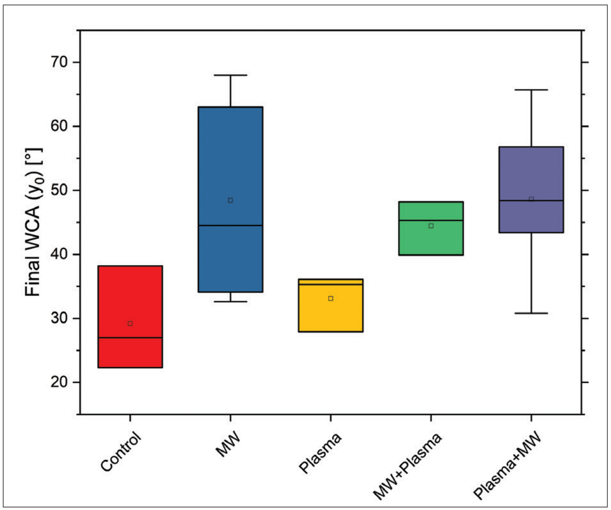


*Figure 4. Statistical analysis of final WCA on differently treated specimens.*

*Slika 4. Statistična analiza končnega stičnega kota vode s površino lesa na različno obdelanih vzorcih.*

tively. Although this presents a reduction of the WCA by PT again after MW, the WCA does increase in all cases compared to the untreated control specimens. This indicates a stronger effect of drying and evaporation of VOCs towards larger WCAs than the impact of PT through the chemical production of additional polar groups on the structural constituents of the wood material. This increase of WCA on green wood after PT is contrary to most published literature (c.f. Žigon et al., 2018) and might not only be related to a drying of the outermost surface layers through enhanced evaporation of moisture induced by the plasma, but it might well relate further to a change in plasma-chemical reactions due to the water vapour above the wood surface. From various literature sources, it is well known that the plasma chemistry that occurs here changes with varying humidity levels in non-thermal air plasmas. In particular, the amount of oxygen radicals and consequently the production rate of ozone is reduced with the increasing presence of water molecules in the atmosphere, mainly via two effects: on the one hand, oxygen radical production and hydroxyl radical generation are competitive processes, both originating from dissociation after resonant energy transfer from metastable excited nitrogen molecules. On the other hand, water molecules act as scavengers for oxygen radicals to form hydroxyl radicals. Both mechanisms lead to the reduced production of ozone and the correspondingly increased production of hydroxyl radicals, which further contribute to the formation of nitric oxide (Herron & Green, 2001; Prysiazhnyi et al., 2012). Moreover, these effects increase with the gas temperature (Sakiyama et al., 2012) and are thus more pronounced, for example, in gliding arc plasmas than in DBDs. These effects need to be further verified, e.g. by future measurements with Optical Emission Spectroscopy and X-ray Photoelectron Spectroscopy, in order to provide insights into both the plasma discharge and their effect on the yielded chemical changes on the surface. From the final WCA results, it is also noteworthy that the PT decreases statistical variations compared to the control specimens in terms of standard deviation, 1.5 IQR, and the ranges from first to third quartile medians. In contrast to that, standard deviation and 1.5 IQR are increased significantly by MW treatment, both on the untreated control and the previously plasma-treated specimens.

Figure 5 shows a statistical analysis of the initial WCA after droplet deposition as given by ($y_0$+$A_1$) with parameters from eq. 1. The untreated control specimens exhibit the lowest initial WCA upon contact with the water droplet with an average of 48.7°. Both MW and PT increased the initial WCA, yielding average values of 91.6° and 89.5° for MW-only and PT-only, respectively. Notably MW-only significant-

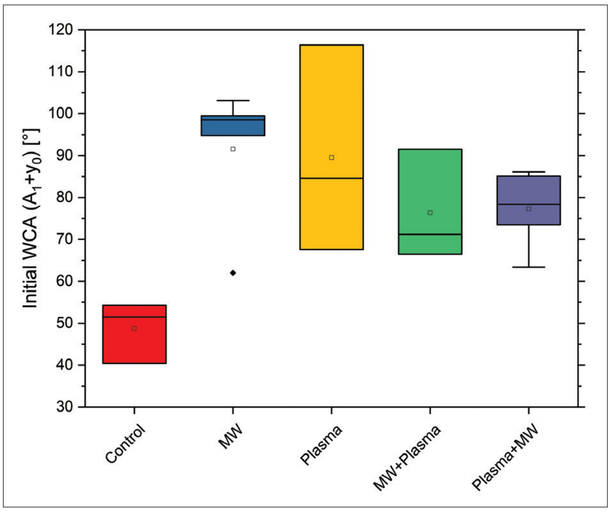


*Figure 5. Statistical analysis of initial WCA on differently treated specimens.*

*Slika 5. Statistična analiza začetnega stičnega kota vode s površino lesa na različno obdelanih vzorcih.*

ly reduced the standard deviation, but increased the 1.5 IQR, whereas PT-only strongly increased both indicators of statistical variation. This can likely be attributed to the heterogeneous nature of the PT due to its filamentary structure and a higher intensity for denser or more conductive parts of the surface, such as on latewood as compared to earlywood. Combined treatments yield comparable initial WCAs with average values of 77.3° and 74.1° for PT+MW and MW+PT, respectively. For both combined treatments, the standard deviation and 1.5 IQR are comparable, whereas the range between first and third quartile medians is lower for the specimens treated first with plasma and subsequently by MW.

Figure 6 shows a statistical analysis of the time constant $t_1$ as defined in eq. 1, representing the change in WCA over time as the droplet spreads across the sample surface, i.e. the wetting rate, whereas the concurrent penetration does not impact the measured WCA. The MW-only treated specimens exhibit an average wetting time of 19 s, equal to the untreated control specimens. The PT-only specimens show a lower wetting time of 15 s, whereas the combined treatments yield increased wetting times of 20 s and 23 s for PT following MW and MW after PT, respectively. However, all these values fall into each other's standard deviation and 1.5 IQR, and thus are not statistically significant. It does appear, though, that all MW-treated specimens show the lowest ranges between first and third quartile medians, whereas PT further reduces the 1.5 IQR. This might be an indication that the high degree of variation could be correlated with the moisture content at the wood surface.

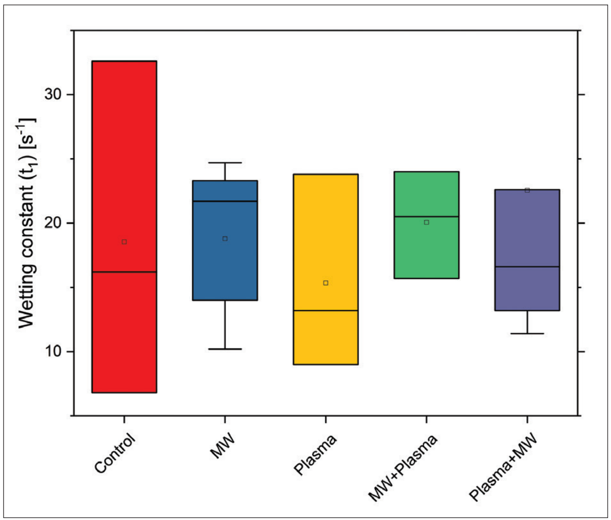


*Figure 6. Statistical analysis of the time constant for wetting on differently treated specimens.*
*Slika 6. Statistična analiza časovne konstante omočenja na različno obdelanih vzorcih.*

## 4 CONCLUSIONS
## 4 SKLEPI

The study confirmed the effects of PT and MW as well as combinations of the two on the initial WCA after droplet deposition, the final WCA, and the time constant of the wetting process for green or wet wood.

The final WCA was negatively affected by all treatments, yielding highest values for MW-only treatments. It was theorized that this negative effect is due to drying of the specimens, which is more strongly pronounced for MW than for PT treatments. Additional measurements are foreseen in the future to investigate the relation of the different methods' drying effects with regard to the measured contact angles. Combinations of MW and PT combinations might indicate a negative effect of MW on a previously plasma-treated surface, and hence suggests an ideal treatment procedure of first MW followed by PT to utilize complementary or synergistic effects.

The initial WCA upon droplet contact was increased after all of the treatments as compared to the untreated control specimens. Among the different treatment procedures, both combined treatments showed ideal and generally comparable results, regardless of the sequence of PT and MW.

The change in wetting time constants was not statistically significant, but might indicate PT as the preferred final step of surface preparation, which should be conducted only after other bulk techniques such as modification via MW.

Overall, the MW and PT results indicate synergistic effects, but experiments using complementary spectroscopic techniques will be required for further insights.

The specific utilization of electric energy per specimen volume amounted to 2700 MJ/m³ for MW, 5645 MJ/m³ for PT and 8345 MJ/m³ for combined MW+PT and PT+MW treatments. It should be noted that the use of PT as a surface treatment strongly increases the energy efficiency for larger specimen volumes, thus being more favourable for industrial applications than small-scale laboratory experiments seem to indicate. However, the results clearly show that PT is not energy-efficient in terms of drying wood material, but its energy utilization and $CO_2$ equivalent should be evaluated in comparison to the surface finishing techniques and the corresponding materials replaced by PT.

## SUMMARY

## 5 POVZETEK

Stični kot kapljic vode s površino lesa ni pokazal odvisnosti v obliki kvadratnega korena od trajanja omočitve, kot sta prej poročala Boehme in Hora (1996), tako da je bila za prileganje izmerjenim podatkom uporabljena eksponentno pojemajoča funkcija (glej eq. 1). Navidezni končni ali ravnotežni stični kot ($y_0$) se je močno povečal pri vzorcih, obdelanih z mikrovalovi, ob hkratnem povečanju variabilnosti. Obdelava s plazmo je povzročila rahlo povečanje stičnega kota, vendar zmanjšanje statistične variabilnosti. Kombinacije obdelave lesa s plazmo in mikrovalovi so povzročile manjše povečanje stičnega kota. V primerih, ko se je obdelava s plazmo izvajala na koncu, pa se je statistična variabilnost ponovno zmanjšala. Zmanjšan učinek obdelave s plazmo v primerjavi z navedbami v literaturi je verjetno posledica spremenjene kemijske sestave površine lesa. Iz svežega lesa pri obdelavi s plazmo prihaja do izhajanja vodne pare, kar vodi do zmanjšane tvorbe ozona in kisikovih radikalov, vendar povečane tvorbe hidroksilnih radikalov in dušikovega oksida.

Začetni stični koti vode neposredno po odlaganju kapljice na površino lesa ($A_1$+$y_0$) so bili največji v primerih, ko smo les posamično obdelali bodisi z mikrovalovi ali plazmo. Kombinacija obdelave z mikrovalovi in plazmo je povzročila nižji začetni stični kot, ki je bil primerljiv ne glede na vrstni red obdelave. Statistična variabilnost začetnega stičnega kota je bila zmanjšana na vzorcih, obdelanih le z mikrovalovi, obdelava s plazmo pa je le-to močno povečala. To je verjetno posledica dejstva, da smo les obdelali z mikrovalovi v surovem, t.j. svežem stanju, kar je s tokom vlage na površino lesa omogočilo tudi migracijo organskih snovi, zlasti sladkorjev in ekstraktivov, s tem pa povzročilo homogenizacijo površine. V nasprotju s tem plazemska obdelava deluje heterogeno z večjo intenzivnostjo na območjih z večjo gostoto ali višjo električno prevodnostjo, kot je kasni les v primerjavi z ranim lesom, tako da plazma prispeva k povečanju kemične nehomogenosti površin.

Časovno konstanto omočenja, določeno v eksponentno pojemajočem modelu, najmočneje poveča obdelava z mikrovalovi, medtem ko so vzorci, obdelani s plazmo, pokazali krajši čas omočenja. Časovna konstanta omočenja je podobna za neobdelane vzorce in vzorce, obdelane z mikrovalovi, medtem ko so vzorci, obdelani s plazmo, pokazali krajši čas omočenja, vzorci, obdelani s kombinacijo plazme in mikrovalov, pa so imeli podaljšan čas omočenja. Statistična variabilnost je bila najnižja pri vzorcih, obdelanih najprej z mikrovalovi in nato s palzmo. Vendar pa je bila statistična variabilnost na splošno tako velika, da ugotovljene razlike v konstanti omočenja niso statistično značilne.

Na splošno rezultati obdelave z mikrovalovi in plazmo kažejo na sinergijske učinke, vendar bodo za podrobnejšo interpretacijo rezultatov potrebni poskusi z uporabo komplementarnih spektroskopskih tehnik.

Specifična poraba električne energije na prostornino vzorca znaša 2700 MJ/m³ za mikrovalovno obdelavo, 5645 MJ/m³ za plazemsko obdelavo in 8345 MJ/m³ za kombinirano obdelavo z mikrovalovi in plazmo. Treba je opozoriti, da plazma kot površinska obdelava močno poveča energijsko učinkovitost za večje količine vzorcev, kar je ugodno za industrijsko uporabo, kot nakazujejo majhni laboratorijski poskusi. Poleg tega rezultati jasno kažejo, da plazemska obdelava ni energetsko učinkovita za sušenje lesnega materiala, vendar je treba izrabo energije in ekvivalent $CO_2$ ovrednotiti v primerjavi s tehnikami površinske obdelave in ustreznimi materiali, ki jih plazma nadomesti.

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## SUPPLEMENTAL INFORMATION AND RAW DATA
## DODATNE INFORMACIJE IN PODATKI

Supplemental material and all raw data can be accessed openly via the Zenodo at https://doi.org/10.5281/zenodo.5835738, and cited as Dahle et al., 2021.

Raziskovalni podatki, obravnavani v članku, ki so na voljo v odprtem dostopu prek Zenodo na povezavi https://doi.org/10.5281/zenodo.5835738, naj bodo citirani kot Dahle et al., 2021.

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# SORPTION PROPERTIES OF WOOD IMPREGNATED WITH THE FIRE RETARDANT BURNBLOCK

# SORPCIJSKE LASTNOSTI LESA, IMPREGNIRANEGA Z OGNJEZADRŽEVALNIM SREDSTVOM BURNBLOCK

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## Abstract / Izvleček

**Abstract:** The sorption properties of wood have a characteristic influence on some of its properties, such as the mechanical properties and susceptibility to fungal decay. Moist wood is more susceptible to fungal decay, and wood is often impregnated in order to protect it from fungal decomposition, photodegradation or fire. In particular, inorganic salts affect the sorption properties of wood. For this purpose, the sorption properties of Norway spruce wood impregnated with Burnblock refractory (uptake 38 kg/m³) were investigated. The microscopic analysis confirmed the presence of crystals of this in the cell lumina of wood tissue. Sorption properties were determined using an instrument capable of dynamic vapour sorption (DVS) assessment. DVS analysis confirmed that the sorption properties of impregnated spruce wood are comparable to those of non-impregnated spruce wood. However, the higher hysteresis at higher relative humidity is probably due to the presence of crystals in the cell lumina.

**Keywords:** fire retardants, Burnblock, wood, sorption properties, laser confocal microscopy

**Izvleček:** Sorpcijske lastnosti lesa imajo značilen vpliv na nekatere njegove lastnosti. V največji meri vplivajo na mehanske lastnosti in dovzetnost lesa na glivni razkroj. Vlažnejši les je bolj dovzeten za glivni razkroj, zato ga pred glivnim razkrojem, fotodegradacijo ali gorenjem pogosto impregniramo. Predvsem anorganske soli vplivajo na sorpcijske lastnosti lesa. S tem namenom smo preiskali sorpcijske lastnosti smrekovine, impregnirane z ognjezadrževalnim sredstvom Burnblock (navzem 38 kg/m³). Mikroskopska analiza je potrdila prisotnost kristalov sredstva v celičnih lumnih lesnega tkiva. Sorpcijske lastnosti smo določili z opremo, ki vrednoti dinamično sorpcijo vodne pare (DVS). DVS analiza je potrdila, da so sorpcijske lastnosti impregniranega smrekovega lesa primerljive s sorpcijskimi lastnostmi neimpregnirane smrekovine. Večja histereza pri višjih relativnih zračnih vlažnostih je verjetno posledica prisotnosti kristalov anarganskih soli, ki so se izločili v celičnih lumnih po impregnaciji.

**Ključne besede:** ognjezadrževalna sredstva, Burnblock, les, sorpcijske lastnosti, laserska konfokalna mikroskopija

## 1 INTRODUCTION
## 1 UVOD

Wood is hygroscopic due to its specific chemical composition and large internal surface area. Therefore, the moisture content of wood oscillates depending on the varying climatic conditions. Under stable conditions, wood reaches hygroscopic equilibrium or equilibrium moisture content (EMC). The interactions between wood and water have been studied scientifically for more than a century (Engelund et al., 2013). The moisture content of wood has a significant effect on some relevant properties, especially the mechanical properties (Gerhards, 1982) and service life of wood used outdoors (Meyer et al., 2016). Fungi can decompose wood if the moisture content is above a certain limit. The moisture content of wood must be high enough to promote the flow path for the reaction products of the enzymes, but low enough to prevent waterlogging. A wide variety of data on this is available in the literature. In the first set of data, it is indicated that the MC limits for fungal decay de-

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pend predominantly on the fungal species. For example, Schmidt (2006) reported that the minimum MC of wood was 25% for *Coniophora puteana* and *Serpula lacrymans* and 30% for *Fibroporia vaillantii* and *Gloeophyllum trabeum*. However, recent data suggest that the limiting moisture content for fungal growth depends on the wood species and fungal species studied. For example, the limiting moisture content for *C. puteana* growth on thermally modified Scots pine (*Pinus sylvestris* L.) sapwood is 12.1%, while the limiting moisture content for fungal decay on the same species of wood is 24.4% (Meyer et al., 2016).

Similar to wood-decaying fungi, MC has a significant effect on the growth and development of sap stain fungi. Sap stain fungi are mainly associated with *Ascomycetes* and *Fungi imperfecti*, and are characterised by the pigmentation of the hyphae walls, which leads to discolouration of the wood. Suitable conditions for the growth and reproduction of the various mould and sap stain fungi vary. Some thrive at relatively low air relative humidity (RH = 75%), while most fungi require higher RH levels and consequently higher wood MC for optimal growth. Different building materials have different susceptibilities to mould growth (Isaksson et al., 2010).

The relationship between the EMC and RH is expressed by sorption isotherms obtained by progressive equilibration in the adsorption or desorption process. Differences in hygroscopic and sorption isotherms result from the wood species, chemical composition of the wood, the amount of microcracks in the cell walls, density, possible hydrothermal and chemical treatment, and stress conditions (Hartley et al., 1992; Willems, 2018).

Sorption isotherms can be divided into three regions. The first represents the EMC from an absolutely dry state to the equilibrium state reached at RH between 20% and 30% (Mitchell, 2018). In this interval, the adsorption of water molecules continues gradually until the outer surface of the cell wall is completely covered by a water monolayer. The wood MC changes more rapidly in the upper part of the region, but slows as it approaches a dry state (Lesar et al., 2009). The second region begins when the first layer is saturated. The adsorption of water molecules on the first layer and the resulting formation of additional layers is a feature of this region, and the isotherms here are quasi-linear (Mangel, 2000). In the third region, capillary condensation of water occurs in microcapillaries. Water molecules form large groups, while the bonds between hydroxyl groups and the first layer of water molecules become weaker, and thus the water molecules can move in clusters (Khali & Rawat, 2000). The water concentration in this region is sufficient for liquid water to form in the pores by capillary condensation, so the microcapillary water forms a continuous phase. In the third hygroscopic region, sorption properties are also influenced by low-molecular secondary heartwood compounds such as polyphenols (flavonoids, lignans, tannins), biocides (boric acids) and fire retardants (Blahovec & Yanniotis, 2008).

As mentioned earlier, the hygroscopic properties of wood can be affected by various treatment processes, like the use of wood biocides and fire retardants. Wood impregnated with various inorganic salts is usually more hygroscopic than untreated wood, especially at high RH. The increase in EMC of such wood depends on the chemicals used, retention, and wood species (White & Dietenberger, 2010). The EMC of impregnated wood and the effects of preservative retention on the equilibrium point are still unknown. High EMC is problematic because it promotes leaching of active ingredients, corrosion of metals, and the creation of favourable conditions for the growth of fungi and especially moulds, and presents difficulties in surface treatment and gluing of moist wood (Lesar et al., 2009). In this study, the sorption properties of wood impregnated with the fire retardant Burnblock were investigated throughout the hygroscopic range during the adsorption and desorption process.

## 2 MATERIAL AND METHODS
## 2 MATERIALI IN METODE

The analysis was carried out on wood treated with fire-retardant, and specifically on Norway spruce (*Picea abies* (L.) Karst.) planks treated with Burnblock (Burnblock, København, Denmark) in a commercial impregnation plant using the full cell impregnation method. Five planks were delivered. The cross-section of the planks was approximately 23 mm × 100 mm and length 200 mm. The retention of Burnblock was 38 kg/m$^3$. Burnblock is made


*Figure 1. Cross-section of spruce wood plank, used for microscopic and sorption analysis.*
*Slika 1. Prečni prerez deske, uporabljene za mikroskopsko in sorpcijsko analizo.*

of ingredients that can be found in nature and are considered environmentally friendly. Treated wood is biodegradable and has no adverse environmental effects (Medved et al., 2019). Five parallel samples were conditioned at laboratory conditions (21 °C; RH 65%), then measured and weighted. The nominal density of the wood was then calculated.

Microscopic analysis was performed on cross-sections of the treated wood. The outer 6 mm of the wood that was fully impregnated with the fire retardant was analysed. Microscopic analysis was performed using a confocal laser scanning microscope (Olympus OLS50-BSW, Tokyo, Japan) and a digital microscope (Olympus DSX1000, Tokyo, Japan). The surface was planed with a stainless steel blade. The MC of the wood was approximately 12%.

Dynamic water vapour sorption of treated and native (i.e. reference, non-treated) samples was performed using a gravimetric dynamic sorption analyser (DVS Intrinsic, Surface Measurement Systems Ltd., London, UK). Samples were ground and homogenised into fractions smaller than 1 mm prior to analysis using a SM 2000 mill (Retsch GmbH, Haan, Germany) and a perforated sieve with a perforation of 1 mm (Conidur˝). The ground samples were conditioned at 20 °C and 1 ± 1% RH. A small amount of the ground sample (≈400 mg) was used. The measurement was performed at a constant temperature of 25 ± 0.2 °C. A total of two sorption and desorption cycles were measured from 0% RH to 95% RH, and vice versa.

## 3 RESULTS AND DISCUSSION
## 3 REZULTATI IN RAZPRAVA

The cross-sections of the wood planks indicate the typical structure of Norway spruce wood. The annual rings are about 2 mm to 3 mm wide (Figure 1). The anatomical structure (Figure 2) shows the gradual transition between earlywood and latewood cells. The respective resin canals are bordered by 8 to 12 or more thick-walled epithelial cells (Wagenführ, 2014). The density of the air-dry planks examined was 420 kg/m$^3$ (st. dev. 17 kg/m$^3$). This is in line with the data in the literature (Gryc et al., 2011; Humar, 2013).

As seen from microscopic analysis (Figure 3, Figure 4), Burnblock crystals are seen in the cell lumina. The presence of the crystals was confirmed using two independent microscopy techniques, confocal laser scanning microscopy and digital microscopy. The presence of the crystals in the cell lumina is not surprising, as the retention of Burnblock and other fire-retardants is higher than the retention of wood preservatives. For example, the retention of typical copper-ethanol wood preservatives is about 20 kg/m$^3$ (Nordic Wood Preservation Council 2021) (for in-ground use), while the retention of classical CCA barely reaches 12 kg/m$^3$ (Willeitner, 2001). The crystals in the cell lumina are rather significant. It can be assumed that they were at least partially damaged during cutting.

In the graphs (Figure 5), the sorption curves of the untreated and treated spruce wood are plotted. As can be seen, both the untreated and


*Figure 2. Annual ring of spruce wood plank*
*Slika 2. Branika v lesu smrekove deske*

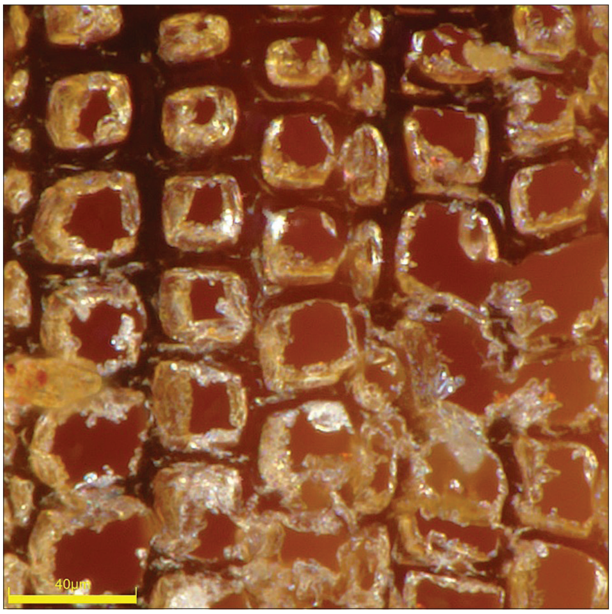

*Figure 3. Cell lumina of all cells are filled with crystals of the fire retardant Burnblock. Microscopy was performed with a digital microscope. Colours are not always representative.*
*Slika 3. Mikroskopska slika, ki prikazuje zapolnjenost vseh celičnih lumnov z ognjezadrževalnim sredstvom Burnblock. Slika je posneta z digitalnim mikroskopom. Barve niso vedno reprezentativne.*

Burnblock-treated wood show typical sorption isotherms of type II. The differences between the EMC at 95% RH of untreated and treated wood are negligible. For example, in the first sorption cycle, the EMC of untreated spruce wood (23.09%) is slightly higher than the EMC of Burnblock-treated

*Figure 4. Cell lumina filled with crystals of the fire retardant Burnblock. The image was obtained with confocal scanning laser microscope (field of view 128 µm × 128 µm). Colours are not always representative.*
*Slika 4. Celični lumni, zapolnjeni s kristali ognjezadrževalnega sredstva Burnblock. Slika je bila posneta s konfokalnim laserskim vrstičnim mikroskopom (vidno polje = 128 µm × 128 µm). Barve niso vedno reprezentativne.*

wood (22.79%). However, in the second sorption cycle, the EMC of Burnblock-treated wood was slightly higher (23.70%) than that of untreated spruce wood (22.47%). Normally, the second EMC at 95% RH for lignocellulosic materials is lower than the first (Glass et al., 2018), but in this case it was slightly higher for the Burnblock-treated sample. As DVS analysis was performed in controlled conditions it enables a reliable comparison, but statistical analysis was not performed due to low number of measurements (Glass et al., 2018).

The interpretation of the sorption curves is that the surfaces of the analysed wood samples are more polar than water molecules, and therefore

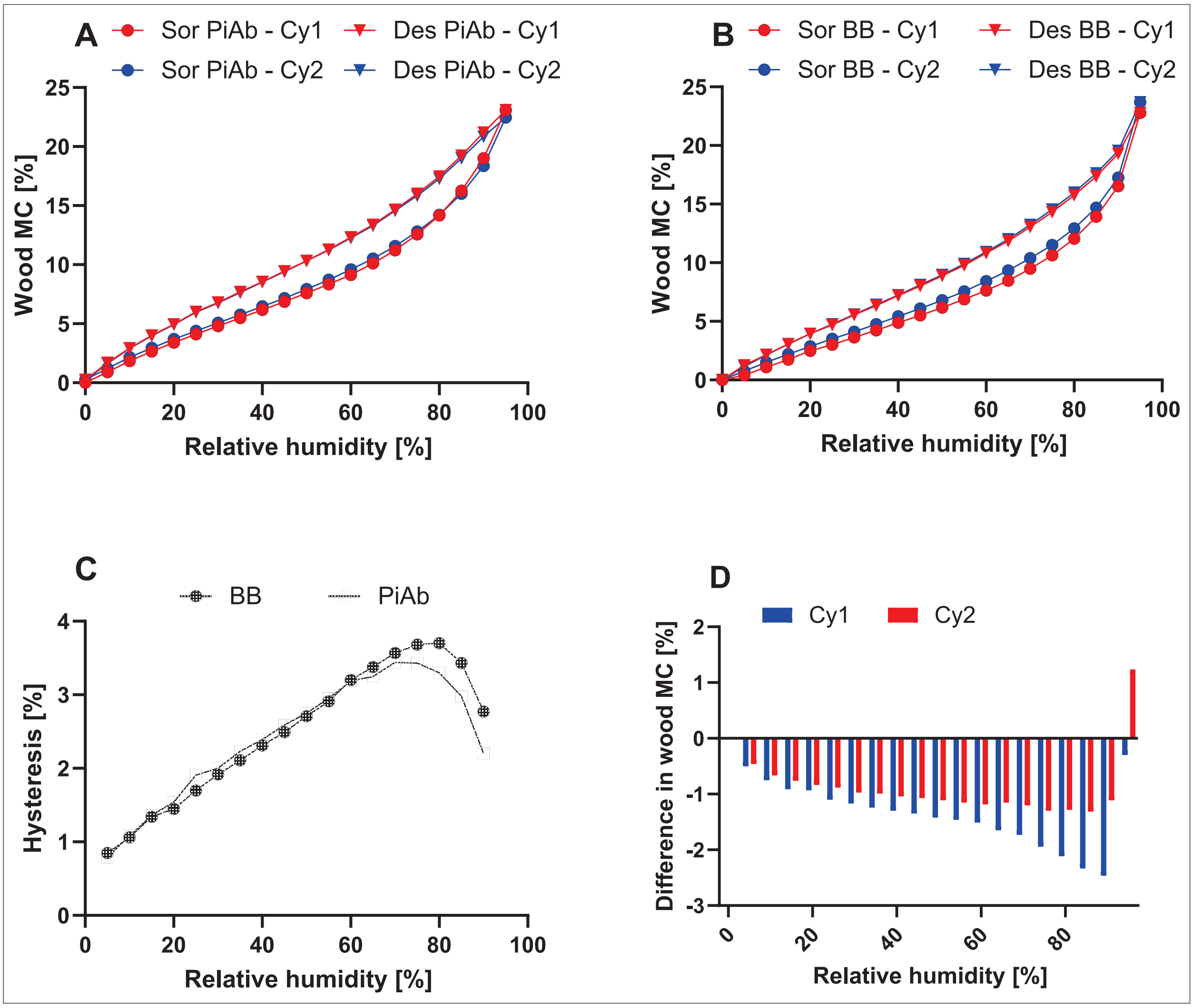


*Figure 5. Results of the sorption analysis of the (A) reference Norway spruce wood (PiAb), (B) Burnblock-impregnated Norway spruce wood (BB) in two sorption and desorption cycles. In graph (C), hysteresis is plotted. In contrast, in graph (D) differences in equilibrium wood moisture content between treated and untreated spruce in the first and second sorption cycles are presented. Negative values indicate that the MC of treated wood was lower than that of untreated wood.*

*Slika 5. Rezultati sorpcijske analize (A) referenčne smrekovine (PiAb), (B) smrekovine, impregnirane z ognjezadrževalnim sredstvom Burnblock v dveh sorpcijskih in desorpcijskih ciklih. Slika C prikazuje histerezo, slika D pa razliko v vlažnosti med impregnirano in neimpregnirano smrekovino v prvem in drugem sorpcijskem ciklu. Negativne vrednosti nakazujejo, da je bila vlažnost impregniranega lesa nižja od vlažnosti neimpregniranega lesa.*

show increased water uptake at low RH (0 to 10%). Once a single (mono-)layer of water has formed, additional adsorption increasingly resembles the condensation of water. At high RH, i.e. above 70%, adsorption is enhanced due to the presence of tiny surface pores (mesopores, with pore diameters of 2 to 50 nm). These attract water molecules on more than one side, i.e. by capillary condensation. This leads to hysteresis in this humidity region caused by the reluctant release of the adsorbed water (Mangel, 2000).

As the hysteresis between the sorption and desorption curves for Burnblock-treated wood increases at the higher sorption range (Figure 5), this indicates that there are more condensation sites present in the Burnblock-treated wood than in the reference spruce wood. This can be ascribed to the presence of crystals in cell lumina, as clearly seen from microscopic analysis (Figure 4).

## 4 CONCLUSIONS
## 4 ZAKLJUČKI

The sorption properties of Burnblock-treated wood are comparable to those of untreated wood, while the moisture content of Burnblock-treated wood is comparable to that of untreated Norway spruce. The only difference can be found in the hysteresis between the sorption and desorption curves at higher relative humidities. This can be ascribed to the presence of the crystals of Burnblock in the wood cell lumina. It can be thus presumed that the Burnblock-treated wood with retentions up to 38 kg/m$^3$, exhibits the same susceptibility towards staining fungi.

## 5 SUMMARY
## 5 POVZETEK

Sorpcijske lastnosti lesa imajo značilen vpliv na nekatere lastnosti lesa. V največji meri vplivajo na mehanske lastnosti in dovzetnost lesa na glivni razkroj. Vlažnejši les je bolj dovzeten za glivni razkroj. Mejna vrednost za glivni razkroj je tri do pet odstotnih točk pod točko nasičenja celičnih sten. Po drugi strani dovzetnost lesa na pojav gliv plesni in gliv modrivk pogosto opišemo s kritično relativno zračno vlažnostjo, pri kateri se pojavijo plesni. Za večino lesnih vrst ta meja znaša okoli 75 %. V primeru bolj higroskopnih lesnih vrst je ta meja lahko tudi nižja. Za zaščito pred glivnim razkrojem, fotodegradacijo ali gorenjem les pogosto impregniramo. Predvsem anorganske soli vplivajo na sorpcijske lastnosti lesa. To je še posebej značilno za les, impregniran z borovo kislino, boraksom ali natrijevim kloridom. Zelo higroskopna so tudi nekatera ognjezadrževalna sredstva. S tem namenom smo preiskali sorpcijske lastnosti smrekovine, impregnirane z ognjezadrževalnim sredstvom Burnblock (navzem 38 kg/m$^3$). Burnblock sodi med okolju prijazne rešitve, brez znanih negativnih vplivov na okolje. Impregniran les smo preiskali z dvema mikroskopskima tehnikama (konfokalna laserska vrstična mikroskopija in digitalna mikroskopija).

Mikroskopska analiza je potrdila prisotnost kristalov v celičnih lumnih. Sorpcijske lastnosti smo določili z opremo, ki omogoča dinamično sorpcijo vodne pare (DVS). Les smo zmleli in homogenizirali ter ga izpostavili dvema cikloma navlaževanja in sušenja v območju med 0 % in 95 % relativne zračne vlažnosti pri 25 °C. Rezultati so pokazali, da so sorpcijske lastnosti lesa, obdelanega z Burnblockom, primerljive s sorpcijskimi lastnostmi neobdelanega lesa smreke. Vsebnost vlage v lesu, impregniranim z Burnblockom, je primerljiva z vsebnostjo vlage neobdelane smrekovine. Edina razlika je v histerezi med sorpcijskimi in desorpcijskimi krivuljami pri višji relativni vlažnosti. To lahko pripišemo prisotnosti kristalov v lumnih lesnih celic. Tako lahko domnevamo, da je les, obdelan z Burnblock, z navzemom do 38 kg/m$^3$, primerljivo dovzeten za delovanje gliv modrivk in plesni kot neimpregniran les.

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# PHYSICAL AND CHEMICAL PROPERTIES OF THREE WILD ALMOND WOOD SPECIES GROWN IN ZAGROS FORESTS

# FIZIKALNE IN KEMIJSKE LASTNOSTI LESA TREH DIVJIH VRST MANDLJEVCA, KI RASTEJO V GOZDOVIH ZAGROSA

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## Abstract / Izvleček

**Abstract:** In this study, the physical properties (oven-dry density, basic density, volumetric shrinkage, and swelling) and structural components (cellulose, lignin, and extractives content) of three wild almond wood species from southwestern Iran, namely *Amygdalus arabica*, *Amygdalus eburna*, and *Amygdalus scoparia*, were investigated. Wild almond is a valuable wood species in the Zagros forests of Iran, but there is a lack of data on their wood properties. Three adult trees of each species were chosen, and samples were prepared from the breast height diameter to measure the focal properties. Results of analysis of variance (ANOVA) showed that the wood species had a significant effect on the wood density and volumetric shrinkage. Maximum oven-dry density and volumetric shrinkage of wood were identified in *Amygdalus scoparia*. The highest and lowest content of structural components were found in *Amygdalus* scoparia and *Amygdalus arabica* wood species, respectively. A deep understanding of the almond wood characteristics will provide a fresh insight into the relationship between the properties and conservation of these special, as well as applications of their wood.

**Keywords:** *Amygdalus*, wood, density, volume shrinkage, cellulose, lignin, Zagros, Iran

**Izvleček:** V študiji so bile raziskane fizikalne lastnosti (gostota, osnovna gostota, prostorninsko krčenje in nabrekanje) in kemijska sestava (celuloza, lignin in ekstraktivne snovi) lesa treh divjih vrst mandljev, in sicer *Amygdalus arabica*, *Amygdalus eburna* in *Amygdalus scoparia* iz jugozahodnega Irana. Divji mandelj je dragocena drevesna vrsta v gozdovih Zagrosa v Iranu, podatki o lastnostih lesa pa so pomanjkljivi. Izbrana so bila tri sestojna drevesa vsake vrste, za merjenje omenjenih lastnosti pa so bili pripravljeni vzorci iz nivoja v prsni višini. Rezultati analize variance (ANOVA) so pokazali, da vrsta pomembno vpliva na gostoto lesa in prostorninsko krčenje. Največje vrednosti gostote absolutno suhega lesa in prostorninskega skrčka so bile ugotovljene pri vrsti *Amygdalus scoparia*. Najvišje in najnižje vsebnosti kemijskih komponent pa so bile ugotovljene pri lesnih vrstah *Amygdalus scoparia* in *Amygdalus arabica*. Poglobljeno razumevanje značilnosti mandljevega lesa bo omogočilo nov vpogled v povezavo med lastnostmi in ohranjanjem ter uporabo lesa raziskanih vrst.

**Ključne besede:** *Amygdalus,* les, gostota, prostorninsko krčenje, celuloza, lignin, Zagros, Iran

## 1 INTRODUCTION
## 1 UVOD

Almond species grow with a large geographical distribution from the southwest to the centre of Asia (Gradziel, 2011). Iran is thought to be the centre of the origin and growth of almonds (Zohary & Hopf, 2000). As such, Iran is an important region for the world almond gene pool and the distribution of wild almond species due to its suitable climate (Balvardi et al., 2015; Nikoumanesh et al., 2011). Twen-

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ty-one wild species and seven inter-specific hybrids have been identified and reported in Iran, of which ten are supposed to be endemic (Khatamsaz, 1998). Zagros forests represent about 40% of Iranian forests and are the most extensive forest area (Sagheb-Talebi et al., 2004). Almonds are deciduous shrubs and small trees adapted to arid or semi-arid environments. They exist as a relatively pure and dominant stand or scattered between 600 to 2200 meters above sea level. This plant is a shrub with a height of 2 m that sometimes reaches a height of 6 m and a stem diameter of 5-20 cm.

Most studies regarding almond tree species are related to their distribution, ecological requirements, and morphological properties, while wood properties such as the physical and chemical properties have not been reported yet. Fundamental studies in these cases can reveal the possibility of the wood's use in various applications or lead to creating a database of different wood species (Bahmani et al., 2020; Dong et al., 2021). Kiaei and Samariha (2011) investigated the physical properties of five important hardwood plants from the forests of north Iran and found that the highest wood density was determined for hornbeam, beech, ash, and oak. Most studies regarding Zagros forest wood species are related to their distribution and ecological factors, and few have examined the wood properties. For example, Bahmani et al. (2018) investigated oak wood's physical and chemical properties in Zagros forests. They found that the density of the Persian oak (*Quercus brantii* Lindl.) is between 0.85 g·cm$^{-3}$ and 1.01 g·cm$^{-3}$, and volumetric shrinkage ranges from 11.32% to 14.15%. Moreover, their results indicated that the cellulose content increased with increasing diameter, and the lignin content decreased. Nazari et al. (2020) studied the geographic variations of wood density and fibre dimensions of Persian oak wood, and reported a significant statistical effect of altitude and slope on the volumetric swelling of this material. In another study by Nazari et al. (2021), the influence of site conditions on the physical and morphological properties of hawthorn (*Crataegus azarolus*) wood grown in the Zagros forests of Iran was investigated. They reported that there were statistically significant differences in the oven-dry density of hawthorn wood at various altitudes, while no significant differences were found between the values of volumetric shrinkage. Understanding the properties of almond wood is important for utilizing wood resources. To the best of our knowledge, there are no published studies relevant to the properties of wild almond wood. Considering the valuable position of these wood species in the Zagros forests of Iran, this study aims to investigate the physical properties, including dry-density, basic density, volumetric shrinkage and swelling, and chemical components (cellulose, lignin, extractives, and ash) of three wild almond wood species.

## 2 MATERIALS AND METHODS
## 2 MATERIALI IN METODE
### 2.1 MATERIALS
### 2.1 MATERIALI

This research was done on three wild almond wood species – desert almond (*Amygdalus arabica*; H=2.58 m, DBH=6 cm), grey almond (*Amygdalus eburna*; H=2.45 m, DBH=7.5 cm), and mountain almond *(Amygdalus scoparia*; H=2.00m, DBH=4.7cm), which were all cut from natural forests in Karebass and choliche-Charmahal and Bakhtiari province in the southwest of Iran. The research area is located between 31°31′36″ N and 31°33′55″ N and between 51°1920″ E and 51°12′15″. Disks from each sampled tree were cut at breast height. The annual rainfall and annual average temperatures were 555 mm and 16.7 °C, respectively. December and November are high-rain months and June and July are low-rain months. The temperature reaches its maximum level in June, July and August. The altitude of this site was 1580 m.

### 2.2 PHYSICAL PROPERTIES
### 2.2 FIZIKALNE LASTNOSTI

Discs, 5 cm in thickness, were taken from logs to determine physical properties such as oven-dry density, basic density, volumetric shrinkage, and volumetric swelling. Determination of wood density was carried out based on the ISO-3131 (2016) standard. For determining the physical properties, testing samples were obtained following ASTM-D143 (2000) and used for measuring the oven-dry and basic density, volumetric swelling and volumetric shrinkage. For this propose, thirty samples were prepared from different parts of the disks (10 samples from each disk) with the dimensions of

30 × 20 × 20 mm³ (tangential × radial × longitudinal). The samples were oven dried at 103 °C to reach a constant weight. Dimensions and dry weight were then measured, with the former Measured in all three principal directions with a digital caliper to the nearest 0.001 mm. The digital balance used for the measurement had an accuracy of 0.01 g. Afterwards the samples were immersed in water (one week) and the weight and dimensions of the samples were re-measured.

The physical properties of the samples were calculated using the following equations:

$\rho_0 = m_0 / V_0$

$R = m_0 / V_s$

$\alpha_v = (V_g - V_0)/V_0$

$\beta_v = (V_g - V_0)/V_s$

where: $\rho_0$ is oven dry density ($g \cdot cm^{-3}$),
R is basic density ($g \cdot cm^{-3}$),
β- volumetric shrinkage (%),
α is volumetric swelling (%),
$V_g$ - volume in green state ($g \cdot cm^{-3}$),
$V_0$ - volume in oven-dry state ($g \cdot cm^{-3}$),
$m_0$ - weight in oven-dry state (g),
$m_g$ - weight in green state.

### 2.3 CHEMICAL PROPERTIES
### 2.3 KEMIJSKE LASTNOSTI

The chemical components were determined according to the TAPPI Tests Methods: Cellulose (T 257 om-85), lignin (T 222 om-98), extractives (T 204 om-88) (Table 1). The cellulose content of almond wood was determined according to the nitric acid method (Rowell et al., 1997). All measurements were repeated three times, and the mean value was used.

### 2.4 STATISTICAL ANALYSIS
### 2.4 STATISTIČNE ANALIZE

To determine the physical properties (dry-density, basic density, volumetric shrinkage, and swelling) and chemical components (cellulose, lignin, extractives, and ash), statistical analysis was conducted using the SPPS 23 (IBM, Armonk, NY, USA) program in conjunction with the analysis of variance (ANOVA). Duncan's multiple range test (DMRT was used to test the statistical significance at the α = 0.05 level. All data were checked for normality with a Shapiro–Wilk's test.

## 3 RESULTS AND DISCUSSION
## 3 REZULTATI IN DISKUSIJA

### 3.1 MACROSCOPIC CHARACTERISTICS OF WOOD
### 3.1 MAKROSKOPSKA ZGRADBA LESA

The wood has a yellow to light brown colour with dark heartwood. Cross-sections indicate a semi-ring-porous distribution of vessels (Figure 1a, b, c). The presence of the vessels can be seen in all of the wood species investigated. As can be found from in literature (Allué et al., 2018), ray cells are uniseriate to 3- and 7-seriate depending on the species. Vessels show spiral thickenings with simple perforation plates. As seen on cross-sections, the wood is characterized by relatively wide annual rings, with clearly visible growth ring borders (Figure 1a, b, c). An attractive macroscopic appearance has made almond wood an excellent choice for furniture production in the past (Britannica Online Encyclopaedia, 2022). Due to the small dimensions of the trees, however, furniture applications are rare nowadays. The wood is now mainly used for high-quality end products such as knife handles, pencils, souvenirs and decorative boxes.

*Table 1. Applied methods for measuring the structural components of wood*
*Preglednica 1. Metode, uporabljene za določitev kemijske zgradbe lesa*

| Chemical components | Standard | Description |
|---|---|---|
| Cellulose (%) | Pettersen (1984) | Concentrated nitric acid |
| Lignin (%) | T 222 om-98 | Sulfuric acid 72% |
| Extractives (%) | T 222 om-88 | Alcohol / acetone |

a

Cross-section / Prečni prerez | Radial section / Radialni prerez | Tangential section / Tangencialni prerez

b

Cross-section / Prečni prerez | Radial section / Radialni prerez | Tangential section / Tangencialni prerez

c

Cross-section / Prečni prerez | Radial section / Radialni prerez | Tangential section / Tangencialni prerez

*Figure 1. Macroscopic appearence of wild almond wood species: (a) Amygdalus arabica, (b) Amygdalus eburna, (c) Amygdalus scoparia*

*Slika 1. Prerezi lesa divjih mandljevcev (a) Amygdalus arabica, (b) Amygdalus eburna, (c) Amygdalus scoparia*

### 3.2 PHYSICAL PROPERTIES
### 3.2 FIZIKALNE LASTNOSTI

The oven-dry density and basic density values for the three wild almond species – *Amygdalus arabica* (0.92 g·cm$^{-3}$; 0.79 g·cm$^{-3}$), *Amygdalus eburna* (0.91 g·cm$^{-3}$; 0.78 g·cm$^{-3}$), and *Amygdalus scoparia* (0.96 g·cm$^{-3}$; 0.93 g·cm$^{-3}$) – are given in Table 2. The analysis of variance (ANOVA) showed a significant difference between species regarding oven-dry density and basic wood density. The highest and lowest oven-dry density and basic density were found in *Amygdalus scoparia* and *Amygdalus arabica* wood, respectively. It is reported that there are several factors affecting the density of the wood, such as anatomical properties, e.g. vessel and fibre morphology, provenance, moisture content, and chemical composition (Wagenführ & Scheiber, 1995; Pásztory et al., 2014; Zeidler & Borůvka, 2016; Bahmani et al., 2020). Wood density classification is grouped according to Wong (2002): light (<0.5 g·cm$^{-3}$), moderately dense (between 0.5–0.8 g·cm$^{-3}$), including the almond wood species examined in this study, heavy (between 0.8–1.0 g·cm$^{-3}$), very dense >1.0 g·cm$^{-3}$).

The values of oven-dry density and basic density obtained in this study for the three species of almond were lower than those of other hardwoods, such as Persian oak and hawthorn growing in the Zagros forests, as reported by Bahmani et al. (2018) and Nazari et al. (2021). From the ANOVA test, there is a significant difference between wood species and volumetric shrinkage and volumetric swelling. Maximum and minimum volumetric shrinkage and swelling were identified in *Amygdalus scoparia* and *Amygdalus arabica*, respectively. As is well known, the relationship between wood density and volumetric shrinkage is positive (Sousa et al., 2018). Wood density is generally variable and is related to many factors such as anatomical characteristics, e.g., vessel and fibre morphology, ecological site, moisture content and chemical constitutes. Fibres are the most important elements affecting wood density, followed by vessels, as (for instance) reported by Kiaei (2012) for *Carpinus betulus*.

### 3.3 CHEMICAL COMPOSITION
### 3.3 KEMIJSKA ZGRADBA

Table 3 illustrates the mean values of the chemical constituents of the three wood species of almond.

The analysis of variance (ANOVA) showed significant differences between the tested species and the chemical components they contain. The highest and lowest content of cellulose, lignin and extractives were found in *Amygdalus scoparia* and *Amygdalus arabica* wood, respectively Such differences could be related to site, growth conditions and forest management practices (Zobel & Buijtenen, 1989; Bahmani et al., 2018). On average, hardwood comprises 40-45% cellulose, 17-25% lignin, and less than 10% extractives. Overall, the cellulose lignin content of *Amygdalus scoparia* is higher than the average of most hardwoods, whereas the lignin content does not differ significantly.

*Table 2. The average values of physical properties in the three studied species. Standard deviations are given in the parenthesis. The different letters indicate a different statistical group.*

*Preglednica 2. Povprečne vrednosti fizikalnih lastnosti preučevanih vrst lesa. V oklepajih so podani standardni odkloni. Različne črke označujejo različne statistične skupine.*

| Wood species / Lesna vrsta | Oven-dry density / Gostota lesa (g·cm$^{-3}$) | Basic density / Osnovna gostota (g·cm$^{-3}$) | Volumetric shrinkage / Prostorninski skrček (%) | Volumetric swelling / Prostorninski nabrek (%) |
|---|---|---|---|---|
| *Amygdalus arabica* | 0.92a (0.07) | 0.79a (0.09) | 0.10a (0.05) | 0.11a (0.06) |
| *Amygdalus eburna* | 0.91a (0.06) | 0.78a (0.06) | 0.12a (0.08) | 0.14a (0.01) |
| *Amygdalus scoparia* | 0.96b (0.02) | 0.93b (0.01) | 0.14b (0.02) | 0.16b (0.02) |

*Table 3. The average chemical composition in the three studied species. Standard deviations are given in parentheses. The different letters indicate different statistical groups.*
*Preglednica 3. Povprečna kemijska sestava preučevanih lesnih vrst. V oklepajih so podani standardni odkloni. Različne črke označujejo različne statistične skupine.*

| Wood species | Cellulose (%) | Lignin (%) | Extractives (%) |
|---|---|---|---|
| *Amygdalus arabica* | 39.92a<br>(2.86) | 17.96a<br>(1.6) | 3.89a<br>(0.6) |
| *Amygdalus eburna* | 43.18a<br>(3.13) | 18.52a<br>(1.8) | 4.01a<br>(0.7) |
| *Amygdalus scoparia* | 49. 83b<br>(4.18) | 25.71b<br>(2.10) | 5.12b<br>(0.9) |

## 4 CONCLUSIONS
## 4 SKLEPI

Wild almonds are valuable tree species in Iranian Zagros forests, although there is limited data on their wood properties. As such, in this study the wood properties of three wild almond species were examined. The results indicated that the studied wood is moderately heavy with a density between 0.91 g·cm$^{-3}$ to 96 g·cm$^{-3}$ and can be classified into moderate-volumetric swelling species. In addition, the cellulose content (39.92-49.83 %), lignin content (17.96-25.71 %), and extractive content (3.89-5.12 %) were determined. Considering the valuable position of tree wood species in Zagros forests, the results obtained in this study can provide basic information about the conservation and rehabilitation of almond wood. Further studies will address other properties such as the fibre dimensions and natural durability of wild almond wood against fungi, moulds, insects, and termites.

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## 5 SUMMARY
## 5 POVZETEK

Divji mandelj je dragocena lesna vrsta v gozdovih Zagrosa v Iranu, podatki o lastnostih lesa pa so pomanjkljivi. Različne vrste mandljev so geografsko razširjene od jugozahoda do osrednje Azije (Gradziel, 2011). Iran naj bi bil središče izvora mandljev (Zohary & Hopf, 2000) in je zaradi primernega podnebja pomembna regija za svetovni genski sklad in razširjenost divjih vrst mandljev (Balvardi et al., 2015; Nikoumanesh et al., 2011). Mandlji so listopadni grmi in majhna drevesa, prilagojena na sušna ali polsušna okolja. Večina študij mandljev se nanaša na njihovo razširjenost, ekološke zahteve, morfološke lastnosti, o lastnostih lesa, kot so fizikalne in kemijske lastnosti, pa še niso poročali. Temeljne raziskave bi lahko ovrednotile možnost njihove uporabe in omogočile oblikovanje podatkovne zbirke različnih vrst lesa (Bahmani et al., 2020; Dong et al., 2021). Bahmani et al. (2018) so raziskovali fizikalne in kemijske lastnosti hrastovega lesa v gozdovih Zagrosa. V literaturi ni podatkov o lastnostih lesa divjega mandlja, zato je namen te študije raziskati fizikalne in kemijske lastnosti treh divjih vrst mandljev *Amygdalus arabica*, *Amygdalus eburna* in *Amygdalus scoparia*. Les je bil posekan v naravnih gozdovih v provincah Karebass, Choliche-Charmahal in Bakhtiari na jugozahodu Irana. Raziskovalno območje se nahaja med 31°31′36″ S in 31°33′55″ S ter med 51°1920″ V in 51°12′15″.

Iz hlodov so bili odvzeti 5 cm debeli diski za določanje fizikalnih lastnosti, kot so gostota absolutno suhega lesa, osnovna gostota, ter prostorninsko krčenje in nabrekanje. Gostota lesa je bila določena na podlagi standarda ISO-3131 (2016). Za določanje fizikalnih lastnosti so bili po standardu ASTM-D143 (2000) pridobljeni preskusni vzorci z dimenzijami 2 × 2 × 2 cm$^3$, ki so bili uporabljeni za merjenje gostote absolutno suhega lesa in osnovne gostote ter prostorninskega krčenja.

Kemične sestavine so bile določene v skladu s preskusnimi metodami TAPPI: Celuloza (T 257 cm-85), lignin (T 222 om-98), pepel (T 211 om-93) in

topnost v alkohol-acetonu (T 204 cm-88). Vsebnost celuloze v mandljevem lesu je bila določena po metodi z dušikovo kislino (Rowell et al., 1997). Vse meritve so bile ponovljene trikrat, uporabljena pa je bila povprečna vrednost.

Za ugotavljanje razlik med vrstami mandljevca z vidika fizikalnih in kemijskih lastnosti je bila opravljena statistična analiza variance (ANOVA) s programom SPPS 23 (IBM, Armonk, NY, ZDA). Za preverjanje statistične značilnosti na ravni α = 0,05 je bil uporabljen Duncanov test več razponov (DMRT).

Les je rumene do svetlo rjave barve s temno jedrovino. Prečni prerezi kažejo na (pol)venčasto porozno razporeditev trahej (slika 1a, b, c). Kot je mogoče razbrati iz literature (Allué et al., 2018), so trakovi 1-3 in 7-redni, odvisno od posamezne vrste. Traheje imajo spiralne odebelitve in enostavne perforirane ploščice. Kot je razvidno iz prečnih prerezov, so za les značilne razmeroma široke branike z jasno vidnimi mejami (slika 1a, b, c). Zaradi dekorativne teksture je bil mandljev les v preteklosti zaželen za izdelavo pohištva (Spletna enciklopedija Britannica, 2022). Zaradi manjših dimenzij dreves je danes uporaba za pohištvo redka. Les se večinoma uporablja za izdelke višjega cenovnega razreda, kot so ročaji nožev, svinčniki, spominki in okrasne škatle.

Vrednosti gostote absolutno suhega lesa in osnovne gostote so: *Amygdalus arabica* (0,92 g·cm$^{-3}$; 0,79 g·cm$^{-3}$), *Amygdalus eburna* (0,91 g·cm$^{-3}$; 0,78 g·cm$^{-3}$) in *Amygdalus scoparia* (0,96 g·cm$^{-3}$; 0,93 g·cm$^{-3}$) (Tabela 1). Analiza variance (ANOVA) je pokazala značilno razliko med vrstami za obe gostoti. Najvišja in najnižja gostota je bila ugotovljena pri lesu *Amygdalus scoparia* oziroma *Amygdalus arabica*.

Iz testa ANOVA je razvidna pomembna razlika med vrstami lesa v prostorninskem krčenju in nabrekanju. Največje in najmanjše prostorninsko krčenje in nabrekanje je bilo ugotovljeno pri vrstah *Amygdalus scoparia* in *Amygdalus arabica*.

Analiza variance (ANOVA) je pokazala pomembno razliko med vrstami in kemijskimi komponentami. Največja vsebnost celuloze, lignina in ekstraktivov je bila ugotovljena v lesu *Amygdalus scoparia* in najmanjša pri *Amygdalus arabica*. Razlike bi lahko bile povezane z rastiščem, pogoji rasti in praksami gospodarjenja z gozdom (Zobel & Buijtenen, 1989; Bahmani et al., 2018). V povprečju les listavcev vsebuje 40-45 % celuloze, 17-25 % lignina in manj kot 10 % ekstraktivnih snovi. Na splošno je vsebnost celuloze in lignina v drevesu *Amygdalus scoparia* višja od povprečja večine listavcev, medtem ko se vsebnost lignina bistveno ne razlikuje.

Rezultati so pokazali, da so preučevane vrste lesa srednje goste, z gostoto med 0,91 g·cm$^{-3}$ in 96 g·cm$^{-3}$, in jih uvrščamo med vrste z zmernim volumenskim nabrekanjem. Poleg tega je bila določena vsebnost celuloze (39,92-49,83 %), lignina (17,96-25,71 %) in pepela (3,89-5,12 %). Nadaljnje študije bodo obravnavale druge lastnosti, kot so dimenzije vlaken in naravno odpornost lesa divjega mandlja proti glivam, plesnim, žuželkam in termitom.

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# TREE-RING CHRONOLOGIES OF *PICEA ABIES*, *LARIX DECIDUA* AND *FAGUS SYLVATICA* ALONG ALTITUDINAL GRADIENTS

# KRONOLOGIJE ŠIRIN BRANIK DREVESNIH VRST *PICEA ABIES, LARIX DECIDUA* IN *FAGUS SYLVATICA* VZDOLŽ GRADIENTOV NADMORSKE VIŠINE

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## *Abstract / Izvleček*

**Abstract:** The dendrochronological climate signal of Norway spruce (*Picea abies*), European larch (*Larix decidua*), and European beech (*Fagus sylvatica*), among others, depends on altitude, therefore we have to collect dendrochronological data systematically for each species along altitude gradients. To this end, we established local tree-ring chronologies for the three species along two elevation gradients: (1) Kokra – Jezersko with sites at 750, 780, 950, 1200, 1250, 1380, 1600 m a.s.l., and (2) Bled – Radovna – Krma with sites at 518, 550, 580, 700, 750, 900, 950, 1000, 1200, 1400, 1600, 1760, 1900, 2040 m a.s.l. We present the main characteristics of the chronologies and the results of the dendroclimatological analyses, which show how the climatic factors influence the variation of the tree rings in dependence of altitude and species. We also present the agreement of the different chronologies in terms of standard dendrochronological parameters such as the t-value and discuss the potential use of the presented database.

**Keywords:** Norway spruce (*Picea abies*), European larch (*Larix decidua*), European beech (*Fagus sylvatica*), tree rings, dendrochronology, altitudinal gradients, climate, Slovenia

**Izvleček:** Dendrokronološki signal navadne smreke (*Picea abies*), evropskega macesna (*Larix decidua*) in navadne bukve (*Fagus sylvatica*) je med drugim odvisen od nadmorske višine, zato moramo za razumevanje dendrokronološkega signala sistematično zbirati podatke o posamezni vrsti vzdolž višinskih gradientov. V ta namen smo zbrali vzorce lesa in sestavili lokalne kronologije širin branik omenjenih treh vrst z območij vzdolž dveh gradientov: (1) Kokra – Jezersko, ki vključuje lokacije na 750, 780, 950, 1200, 1250, 1380 in 1600 m nadmorske višine, in (2) Bled – Radovna – Krma z rastišči na 518, 550, 580, 700, 750, 900, 950, 1000, 1200, 1400, 1600, 1760, 1900, 2040 m n. v. Predstavljamo glavne značilnosti kronologij in rezultate dendroklimatoloških analiz, ki so potrdile, da se podnebni dejavniki, ki vplivajo na variiranje širin branik, spreminjajo z nadmorsko višino in da je to spreminjanje širin branik različno pri posamezni drevesni vrsti. Predstavljamo tudi ujemanje različnih kronologij z vidika standardnih dendrokronoloških parametrov, kot je t-vrednost, in razpravljamo o možni uporabi predstavljene podatkovne baze.

**Ključne besede:** navadna smreka (*Picea abies*), evropski macesen (*Larix decidua*), navadna bukev (*Fagus sylvatica*), branike, dendrokronologija, gradient nadmorske višine, podnebje, Slovenija

## 1 INTRODUCTION
## 1 UVOD

Norway spruce (*Picea abies*), European larch (*Larix decidua*), and European beech (*Fagus sylvatica*) in Slovenia have particular species-specific tree-ring characteristics in relation to climatic factors which affect the importance of each species in dendrochronology. Since the climatic signal depends on altitude, the construction of local chronologies of trees from known sites along altitudi-

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nal gradients represents an important step in their dendrochronological characterization, including the issues of teleconnection (similarity of the dendrochronological signal of the same species over longer distances) and heteroconnection (similarity of the dendrochronological signal between different species).

European beech has a wide natural range and grows on a great variety of sites (Euforgen, 2022), and is important for dendroecological studies throughout Europe (e.g., Di Filippo et al., 2007; Martinez del Castillo et al., 2022). The dendrochronological signal of beech shows a variable response to climatic factors depending on altitude and latitude (Čufar et al., 2008; Di Filippo et al., 2007; Martinez del Castillo et al., 2018). In temperate zones of Central Europe, including Slovenia, lowland beech responds mainly negatively to hot and dry late spring and early summer (May, June, July) weather, while at higher elevations and cold sites it responds positively to summer temperatures (e.g., Čufar et al., 2008; Di Filippo et al., 2007). Numerous studies have also shown that frequent climatic extremes such as ice storms, late frosts, and excessive summer heat negatively affect beech growth (Bascietto et al., 2018; Decuyper et al., 2020; Gazol et al., 2019; Martinez del Castillo et al., 2022; Roženbergar et al., 2020). It is thus assumed that beech might decline at numerous sites as climate change progresses (Martinez del Castillo et al., 2022).

Beech is currently the most common forest tree species in Slovenia, accounting for 32.9% in the wood stock (ZGS, 2021). Its wood is a highly valued industrial timber for numerous uses (Čufar et al., 2017). Despite this, it is rarely found in historical objects, and we do not have long composed regional chronologies for dating (Čufar et al., 2012).

Norway spruce is one of the most important coniferous tree species with a wide distribution area (Euforgen, 2022). Basically, it is a species of cold environments, which has been widely artificially spread in Central Europe (including Austria, Germany, Czech Republic, Switzerland, Slovenia) even in lowland areas (e.g., Caudullo et al., 2016; Jansen et al., 2017; Kolář et al., 2020; Marincek et al., 2003). It is an important wood species for various uses (Straže et al., 2022), wood formation and dendroecology (e.g., Kolář et al., 2020; Martinez del Castillo et al., 2018), dendrochronology and for dating historical objects, including musical instruments (Bernabei et al., 2017; Cherubini, 2021; Wilson et al., 2004).

Norway spruce is currently the second most common forest tree species in Slovenia, accounting for 30.2% of the wood stock (ZGS, 2021). Its natural range in Slovenia is restricted to high altitudes, mainly in the Alps and the Dinaric Mountains (Brus, 2012; ZGS, 2022). Since the early 19$^{th}$ century, the species has been artificially spread throughout Slovenia, including the lowlands (ZGS, 2022), where it is currently severely affected by climate change and associated bark beetle infestations (e.g., de Groot et al., 2021).

The dendroclimatological signal of spruce is strongly influenced by local climatic conditions and varies considerably with altitude. This variability is particularly high in Slovenia, where we lack an adequate collection of chronologies for dating historical objects (e.g., Bernabei et al., 2018; Čufar et al., 2020). In Slovenia, spruce is found in numerous objects that are often difficult to date, therefore the knowledge of its signal along elevational gradients is particularly important.

European larch is a pioneer tree species, able to colonize open land on disturbed soils; it can tolerate very cold temperatures during winter. Its natural range is mainly limited to high mountains, especially the Alps (Euforgen, 2022). In Slovenia it has a share of 1.2% in the wood stock (ZGS, 2021). Its wood is highly valued for its high density and durable heartwood (Čufar, 2006; Gričar & Prislan, 2021). It is and was valued for modern and historical constructions, and can be found in many prominent buildings of the Venetian Republic (Levanič et al., 2001). Therefore, long composed tree-ring chronologies have been constructed for this species, based on wood from trees and historical constructions (Bebber, 1990; Nicolussi, 1995; Siebenlist-Kerner, 1984). Using subfossil stems preserved in bogs and glaciers it was possible to construct one of the longest multimillennial chronologies of conifers, including *Larix decidua*, *Pinus cembra* and *Picea abies*, spanning 9,111 years (7109 BC to AD 2002) (Nicolussi et al., 2009).

Larch from high elevation shows excellent teleconnection over larger areas (Levanič, 2005a; Levanič et al., 2001). However, detailed studies in Slovenia have shown that its dendrochronological signal also depends on altitude (Levanič, 2005b) which often makes the dating of historical objects made of "lowland larch" extremely difficult.

The main objective of this study is to present the tree-ring chronologies of Norway spruce (*Picea abies*), European larch (*Larix decidua*), and European beech (*Fagus sylvatica*) along two altitudinal gradients in the Kamnik-Savinja Alps and the Julian Alps, starting from the lowlands to the altitudinal limit of species distribution in the studied areas. We present (1) the constructed tree-ring chronologies and their main characteristics, (2) how climatic

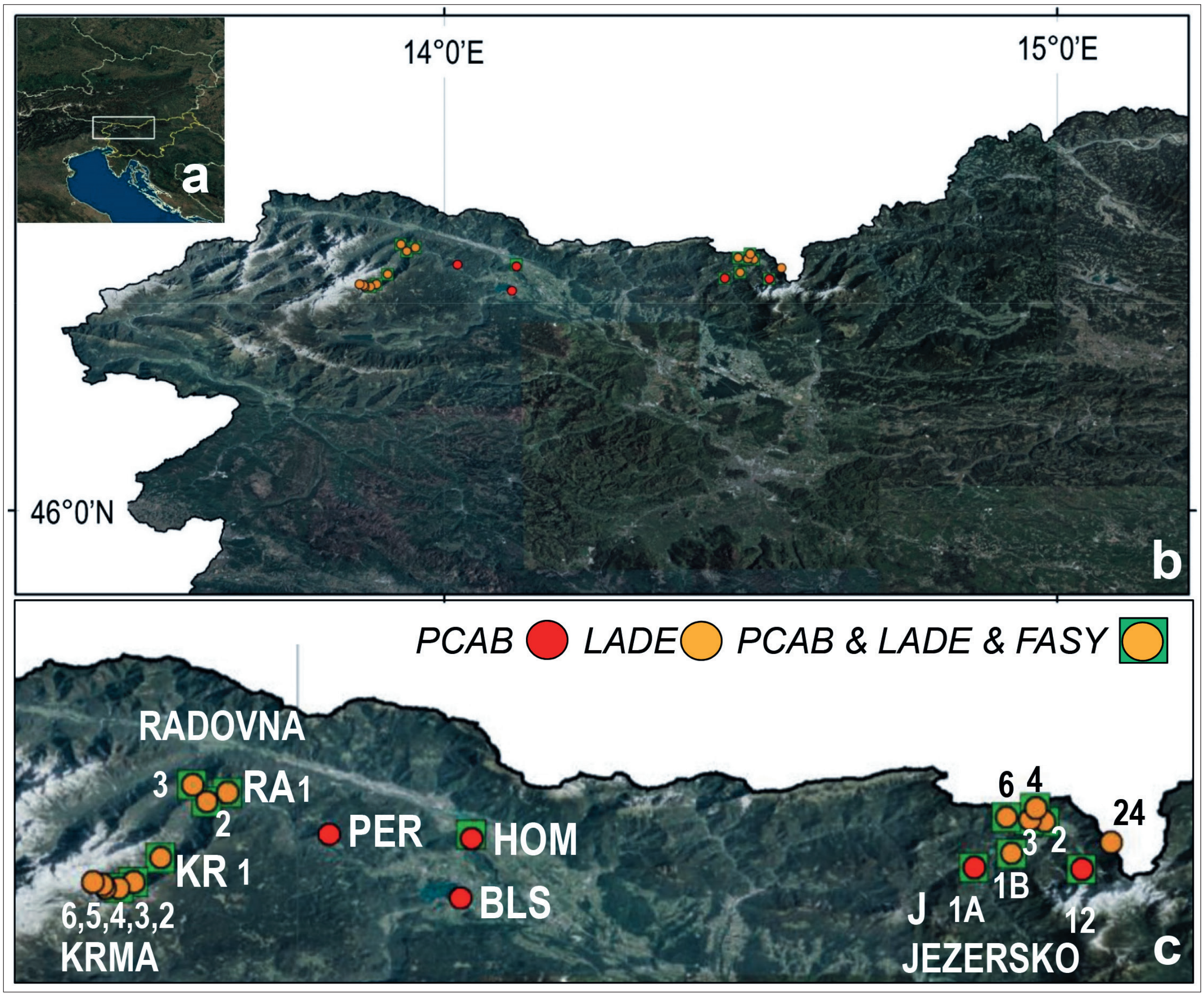



*Figure 1. Sampling areas with (a) map of Slovenia, (b) Kokra – Jezersko, and Bled – Radovna – Krma with sampling locations and (c) detailed views of site locations along the altitudinal gradients for three tree species: European beech (Fagus sylvatica) – FASY; European larch (Larix decidua) – LADE; and Norway spruce – (Picea abies), PCAB. For details see Tables 1 and 2.*
*Source of maps: Esri, DigitalGlobe, GeoEye, Earthstar Geographics, CNES/Airbus DS, USDA, USGS, AeroGrid, IGN, and the GIS User Community.*
*Slika 1. Mesta vzorčenja (a) zemljevid Slovenije, (b) Kokra – Jezersko in Bled – Radovna – Krma z oznakami lokacij vzorčenja ter (c) mesta vzorčenja vzdolž višinskih gradientov za tri vrste: navadno bukev (Fagus sylvatica)–FASY, evropski macesen (Larix decidua)–LADE in navadno smreko (Picea abies)–PCAB. Za podrobnosti primerjajte preglednici 1 in 2.*
*Vir zemljevidov: Esri, DigitalGlobe, GeoEye, Earthstar Geographics, CNES/Airbus DS, USDA, USGS, AeroGrid, IGN, and the GIS User Community.*


*Figure 2. Selected locations from two areas with similar site characteristics: (a) Kokra – Jezersko with sampling locations J02, J03, J04 and J06, and (b) Krma with sampling locations KR1, KR2, KR3 and KR4.*
*Slika 2. Izbrane lokacije z dveh območij s podobnimi rastiščnimi razmerami: (a) Kokra – Jezersko z lokacijami vzorčenja J02, J03, J04 in J06 in (b) Krma z lokacijami vzorčenja KR1, KR2, KR3 and KR4.*

factors influence tree-ring variations, (3) the variation of their dendrochronological signal, and (4) the potential of the database for future studies addressing various issues related to ecology, climate, and cultural heritage.

## 2 MATERIALS AND METHODS
## 2 MATERIAL IN METODE
### 2.1 STUDY SITES AND TREES
### 2.1 RAZISKOVALNE PLOSKVE IN DREVESA

The experimental design was based on a selection of mature dominant or codominant trees of Norway spruce, European larch, and European beech felled in the areas of Kokra – Jezersko, between the Karawanks and Kamnik-Savinja Alps, and Bled – Radovna – Krma, Julian Alps in northwestern Slovenia (Figure 1).

The sampling area Kokra – Jezersko is oriented south-east in the Kamnik-Savinja Alps. The sampling was performed on localities at 750, 780, 950, 1200, 1250, 1380, 1600 m a. s. l.) (Figures 1, 2a, Table 1).

The area Bled – Radovna – Krma included localities on 518, 550, 580, 700, 750, 900, 950, 1000, 1200, 1400, 1600, 1760, 1900, 2040 m a. s. l. (Figures 1, 2b, Table 2).

### 2.2 SAMPLE COLLECTION AND PREPARATION
### 2.2 VZORČENJE IN OBDELAVA VZORCEV

The fieldwork with the collection of samples was carried out from March until November 2019 in cooperation with forest owners, the Slovenia Forest Service, local foresters, the Triglav National Park in the area Bled – Radovna – Krma, and the Municipality of Jezersko in the area Kokra – Jezersko with corresponding permissions. We aimed to collect 15 trees per site. After regular felling we collected discs at the lower part of the trees (mainly 4 m above ground level). If the number of felled trees was not sufficient, additional samples were collected by coring from the nearby living trees. For this purpose, two cores per tree were extracted at the breast height, perpendicular to the tree axis from the bark to the pith using a Haglöf increment corer combined with a Haglöf increment borer chuck and a cordless drilling machine (Milwaukee, M18 FDD2-502X FUEL-135 Nm).

The samples were labelled with the identifying system of the codes, which contained information on sampling site, tree species, tree number and the radius.

The sampled discs and cores fixed on wooden supports were transported to the workshop and air dried. Their transversal surfaces were sanded with the belt sander using progressively finer sandpaper, from 80, 120, 180, 220, 280, and 360 grit until the tree rings and individual cells in the wood on the transversal section were perfectly visible under a stereo microscope.

The wood surface was scanned with a Mustek S-series 2400 Plus flatbed scanner with the resolution set at 1200 dpi and the images were processed with Adobe Photoshop Elements 2020. In the case of extremely narrow rings the structure of wood was additionally checked under an Olympus stereo microscope S2 11 or images were obtained with the help of confocal laser scanning microscope CLSM (Balzano et al., 2019).

### 2.3 DATA ACQUISITION AND PROCESSING
### 2.3 ZAJEM IN OBDELAVA PODATKOV

Tree-ring widths were measured using calibrated high-resolution digital photos along two radii of each tree, to the nearest 0.01 mm using the CDendro / CooRecorder 9.5 image analysis program (Cybis Elektronik, 2022 http://www.cybis.se/forfun/dendro/helpcoorecorder7/index.php). The TSAP-Win program (Frank Rinn, Heidelberg, Germany) and R Studio program using the dplR library (Bunn, 2010) were used for visual and statistical cross-dating and verification.

Cross-dated tree-ring series were assembled into local chronologies using R Studio and the dplR package (Bunn, 2008).

### 2.4 TREE RING CHRONOLOGIES AND CLIMATE
### 2.4 KRONOLOGIJE ŠIRIN BRANIK IN KLIMA

The climatic influence on tree growth was analysed using the residual version of each chronology with R Studio. For this purpose, the original tree-ring width series were standardized in a two-step procedure. First, the long-term trend was removed by fitting a negative exponential function (regression line) to each tree-ring series. Second, more flexible detrending was carried out by applying a cubic smoothing spline with a 50% frequency response of 30 years to further reduce non-climatic variance. Subsequently, autoregressive modelling of the residuals and bi-weight robust estimation of the mean were applied (Cook & Peters, 1997).

Local climatic data for calculation were obtained from the SLOCLIM data base (Škrk et al., 2021) which is a publicly available modelled climatic database which contains a daily gridded dataset of maximum and minimum temperature and precipitation data with 1×1 km spatial resolution covering the entire territory of Slovenia from 1950 to 2018. The data are available on zenodo (Škrk et al., 2020, 2021) and on the web page www.sloclim.eu. For each sampling location we extracted the climatic data of the nearest grid point and aggregated the daily data into monthly mean values.

Pearson correlation function coefficients (CFC) were calculated by using the residual version of each tree-ring chronology as a dependent variable and the regressors monthly minimum and maximum temperatures and the monthly sums of precipitation for each biological year from the previous January to current December, as well as for the past and current spring, summer, autumn and current winter for the period 1950-2018. The climate and growth relationships were calculated using the program packages library("dplR"), library("stringr"), and library("plyr"). The CFC values were considered statistically significant when $p<0.05$.

### 2.5 TELECONNECTION AND HETEROCONNECTION
### 2.5 TELEKONEKCIJA IN HETEROKONEKCIJA

To test the potential of the chronologies with regard to establishing regional chronologies for dating purposes, we made basic comparisons among the chronologies by calculating standard statistical values, including the t-value after Baillie and Pilcher (tBP) and sign test (Gleichläufigkeit–Glk) using the TSAP-Win program.

We also tested the chronologies for teleconnection (agreement between the chronologies of the same species from different sites) and for heteroconnection (agreement between different tree species from the same site).

## 3 RESULTS AND DISCUSSION
## 3 REZULTATI IN RAZPRAVA

### 3.1 TREE-RING DATA AND THE CHRONOLOGIES
### 3.1 KRONOLOGIJE ŠIRIN BRANIK

The database consists of 47 chronologies of three species along two altitudinal gradients in the Alps of Slovenia. We present their locations and time spans (Table 1 and 2). The local chronologies had average lengths of 156 (69-296) years for *European beech*, 139 (57-355) years for *Norway spruce*, and 191 (56-378) years for *European larch.*

At Kokra – Jezersko the corresponding averages (minimum-maximum) for seven *beech* chronologies were 133 (69-296) years, for seven *spruce* chronologies 135 (57-246) years, and for six *larch* chronologies 156 (56-214) years (Table 1).

At Bled – Radovna – Krma the averages (minimum-maximum) for seven *beech* chronologies were 179 (69-296) years, for 11 *spruce* chronologies 142 (81-355) years, and for nine larch chronologies 215 (84-378) years. The oldest trees were sampled in Krma (KR*) (Table 2).

### 3.2 TREE-RINGS AND CLIMATE
### 3.2 ŠIRINE BRANIK IN KLIMA

Correlation function coefficients (CFCs) for residual chronologies and monthly minimum (Tmin) and maximum temperatures (Tmax) and precipitation (PCP) (Figures 3 and 4) from the previous January to current December and from the previous and current spring, summer, autumn and current winter show that each of the species has a unique response to climate and that the response varies with elevation.

For example, in Kokra – Jezersko (Figure 3) beech shows a negative response to June temperatures (especially Tmax) and a positive response to June precipitation. The values of the CFCs generally decrease from lower to higher altitude, while at 1600 m a.s.l. we observe a positive response to temperatures in July, August and September. Spruce shows a negative response to temperatures in July and August and a positive response to precipitation in July at the same gradient, while at 1600 m a.s.l. a positive response to temperatures in May and August is observed. Larch shows a negative response to Tmax in March and a positive response to summer temperatures, while at altitudes above 1250 m a.s.l. we observe a positive response to May temperatures, especially Tmax, and a pos-

| Code / Koda | Species / Drevesna vrsta | Altitude / Nadmorska višina | Latitude / Zemljepisna širina | Longitude / Zemljepisna dolžina | Number of trees / Število dreves | Chronology Length / Kronologija Razpon | Start / Začetek | End / Konec |
|---|---|---|---|---|---|---|---|---|
| | | m | N / S | E / V | | Years / leta | Year / leto | Year / leto |
| J01A | FASY | 750 | 46.380118° | 14.457731° | 7 | 121 | 1899 | 2019 |
| J01B | FASY | 780 | 46.389231° | 14.483326° | 10 | 122 | 1898 | 2019 |
| J02 | FASY | 950 | 46.410676° | 14.505766° | 12 | 78 | 1941 | 2018 |
| J12 | FASY | 1200 | 46.378934° | 14.530259° | 18 | 130 | 1890 | 2019 |
| J03 | FASY | 1250 | 46.411992° | 14.494944° | 8 | 151 | 1869 | 2019 |
| J04 | FASY | 1380 | 46.419799° | 14.499313° | 11 | 169 | 1850 | 2018 |
| J06 | FASY | 1600 | 46.413493° | 14.479713° | 10 | 160 | 1860 | 2019 |
| J01B | LADE | 780 | 46.389231° | 14.483326° | 10 | 162 | 1858 | 2019 |
| J02 | LADE | 950 | 46.410676° | 14.505766° | 5 | 163 | 1856 | 2018 |
| J03 | LADE | 1250 | 46.411992° | 14.494944° | 4 | 153 | 1866 | 2018 |
| J04 | LADE | 1380 | 46.419799° | 14.499313° | 10 | 214 | 1805 | 2018 |
| J06 | LADE | 1600 | 46.413493° | 14.479713° | 13 | 189 | 1832 | 2020 |
| J24 | LADE | 1600 | 46.396635° | 14.550305° | 10 | 56 | 1964 | 2019 |
| J01A | PCAB | 750 | 46.380118° | 14.457731° | 18 | 145 | 1874 | 2018 |
| J01B | PCAB | 780 | 46.389231° | 14.483326° | 15 | 174 | 1846 | 2019 |
| J02 | PCAB | 950 | 46.410676° | 14.505766° | 30 | 89 | 1930 | 2018 |
| J12 | PCAB | 1200 | 46.378934° | 14.530259° | 17 | 138 | 1882 | 2019 |
| J03 | PCAB | 1250 | 46.411992° | 14.494944° | 14 | 133 | 1887 | 2019 |
| J04 | PCAB | 1380 | 46.419799° | 14.499313° | 15 | 97 | 1922 | 2018 |
| J06 | PCAB | 1600 | 46.413493° | 14.479713° | 12 | 246 | 1774 | 2019 |

*Table 1. Kokra – Jezersko, basic information on the sites and chronologies along the gradient (short code, species, altitude, latitude, longitude, number of trees, useful length of the chronology and its start and end date) for three tree species: European beech (Fagus sylvatica) – FASY: European larch (Larix decidua) – LADE, and Norway spruce (Picea abies) – PCAB.*

*Preglednica 1. Kokra – Jezersko, osnovni podatki o rastiščih in kronologijah vzdolž gradienta nadmorske višine (kratka koda, drevesna vrsta, nadmorska višina, zemljepisna širina, zemljepisna dolžina, število dreves, uporabna dolžina kronologije ter prvo in zadnje leto) za tri drevesne vrste: navadna bukev (Fagus sylvatica)–FASY, evropski macesen (Larix decidua)–LADE in navadna smreka (Picea abies)–PCAB.*

itive response to August temperatures with lower values of correlation coefficients.

Species responses along the Bled – Radovna – Krma slope differ from those at Jezersko. Beech shows a negative response to June temperatures in Hom (518 a.s.l.), while the Radovna and Krma sites respond mainly positively to May temperatures and negatively to March temperatures. Spruce at lower elevations shows a positive influence of January, February and March temperatures, while at elevations above 1000 m a positive influence of May temperatures is observed. Larch shows a negative response to Tmax in March and a positive response to temperatures in May.

| Code / Koda | Species / Drevesna vrsta | Altitude / Nadmorska višina | Latitude / Zemljepisna širina | Longitude / Zemljepisna dolžina | Number of trees / Število dreves | Chronology Length / Kronologija Razpon | Start / Začetek | End / Konec |
|---|---|---|---|---|---|---|---|---|
| | | m | N / S | E / V | | Years / leta | Year / leto | Year / leto |
| HOM | FASY | 550 | 46.401975° | 14.117478° | 15 | 146 | 1874 | 2019 |
| RA01 | FASY | 700 | 46.430571° | 13.952667° | 14 | 69 | 1951 | 2019 |
| RA02 | FASY | 750 | 46.423824° | 13.938677° | 15 | 115 | 1905 | 2019 |
| RA03 | FASY | 900 | 46.435654° | 13.928797° | 20 | 188 | 1833 | 2020 |
| KR01 | FASY | 1000 | 46.386683° | 13.907620° | 18 | 239 | 1780 | 2018 |
| KR02 | FASY | 1200 | 46.370348° | 13.888781° | 10 | 198 | 1822 | 2019 |
| KR03 | FASY | 1400 | 46.366540° | 13.879257° | 10 | 296 | 1724 | 2019 |
| RA01 | LADE | 700 | 46.430571° | 13.952667° | 20 | 174 | 1846 | 2019 |
| RA02 | LADE | 750 | 46.423824° | 13.938677° | 12 | 195 | 1824 | 2018 |
| RA03 | LADE | 900 | 46.435654° | 13.928797° | 7 | 84 | 1936 | 2019 |
| KR01 | LADE | 1000 | 46.386683° | 13.907620° | 15 | 181 | 1833 | 2013 |
| KR02 | LADE | 1200 | 46.370348° | 13.888781° | 10 | 154 | 1866 | 2019 |
| KR03 | LADE | 1400 | 46.366540° | 13.879257° | 16 | 141 | 1880 | 2020 |
| KR04 | LADE | 1600 | 46.366383° | 13.869982° | 7 | 328 | 1692 | 2019 |
| KR05 | LADE | 1760 | 46.368392° | 13.867702° | 12 | 378 | 1642 | 2019 |
| KR06 | LADE | 1900-2040 | 46.370559° | 13.861593° | 12 | 303 | 1717 | 2019 |
| HOM | PCAB | 518 | 46.359262° | 14.110083° | 11 | 110 | 1910 | 2019 |
| BLS | PCAB | 580 | 46.399197° | 14.117837° | 12 | 98 | 1922 | 2019 |
| RA01 | PCAB | 700 | 46.430571° | 13.952667° | 20 | 107 | 1913 | 2019 |
| RA02 | PCAB | 750 | 46.423824° | 13.938677° | 23 | 155 | 1864 | 2018 |
| RA03 | PCAB | 900 | 46.435654° | 13.928797° | 12 | 110 | 1909 | 2018 |
| PER | PCAB | 950 | 46.402138° | 14.021151° | 15 | 81 | 1939 | 2019 |
| KR01 | PCAB | 1000 | 46.386683° | 13.907620° | 19 | 164 | 1855 | 2018 |
| KR02 | PCAB | 1200 | 46.370348° | 13.888781° | 9 | 172 | 1848 | 2019 |
| KR03 | PCAB | 1400 | 46.366540° | 13.879257° | 15 | 116 | 1904 | 2019 |
| KR04 | PCAB | 1600 | 46.366383° | 13.869982° | 11 | 355 | 1665 | 2019 |
| KR05 | PCAB | 1760 | 46.368392° | 13.867702° | 4 | 95 | 1925 | 2019 |

*Table 2. Bled – Radovna – Krma, basic information on the sites and chronologies along the gradient (short code, species, altitude, latitude, longitude, number of trees, useful length of the chronology and its start and end date) for three tree species: European beech (Fagus sylvatica) – FASY, European larch (Larix decidua) – LADE, and Norway spruce (Picea abies) – PCAB.*

*Preglednica 2. Bled – Radovna – Krma, osnovni podatki o rastiščih in kronologijah vzdolž gradienta nadmorske višine (kratka koda, drevesna vrsta, nadmorska višina, zemljepisna širina, zemljepisna dolžina, število dreves, uporabna dolžina kronologije ter prvo in zadnje leto) za tri drevesne vrste: navadna bukev (Fagus sylvatica)–FASY, evropski macesen (Larix decidua)–LADE in navadna smreka (Picea abies)–PCAB.*

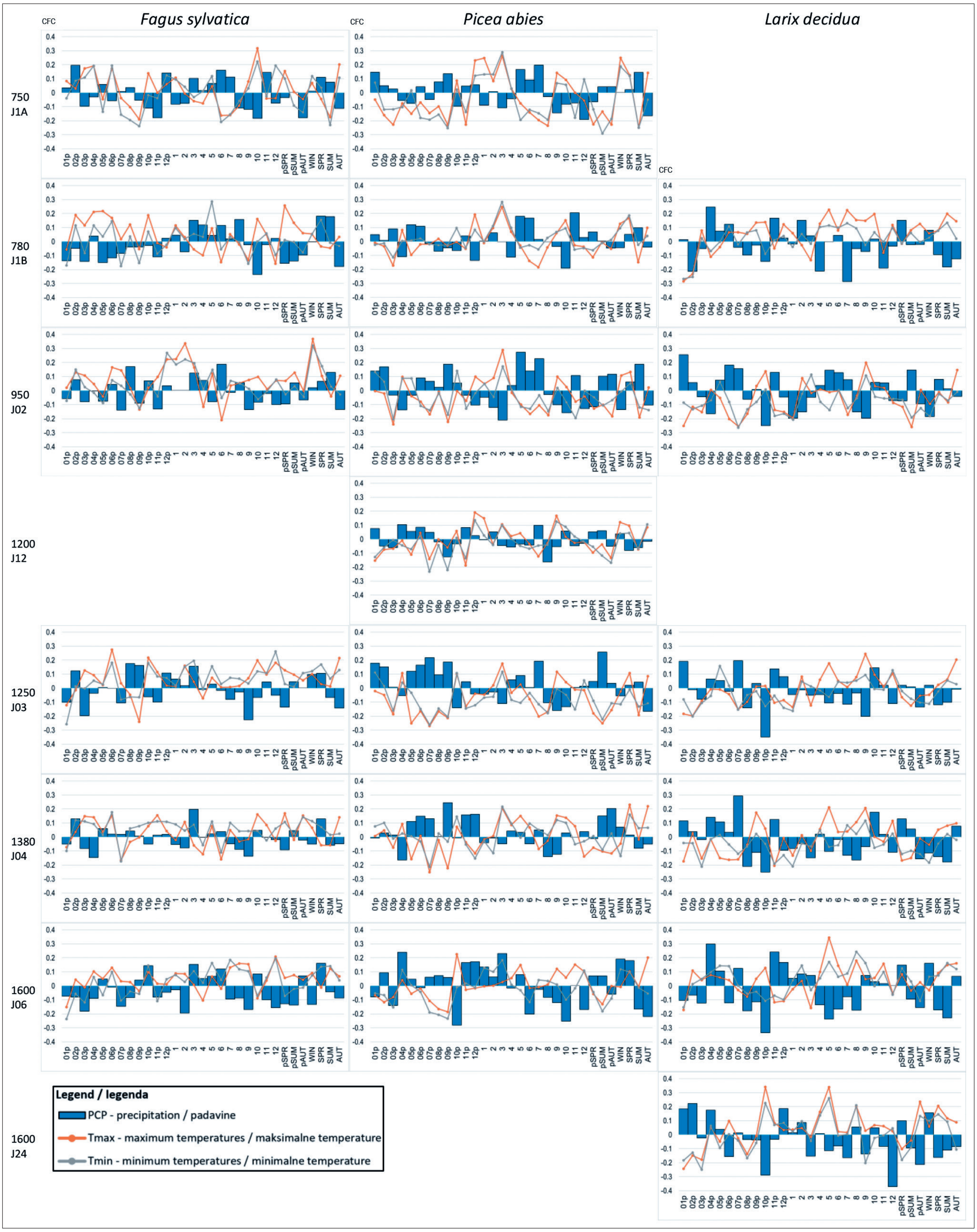
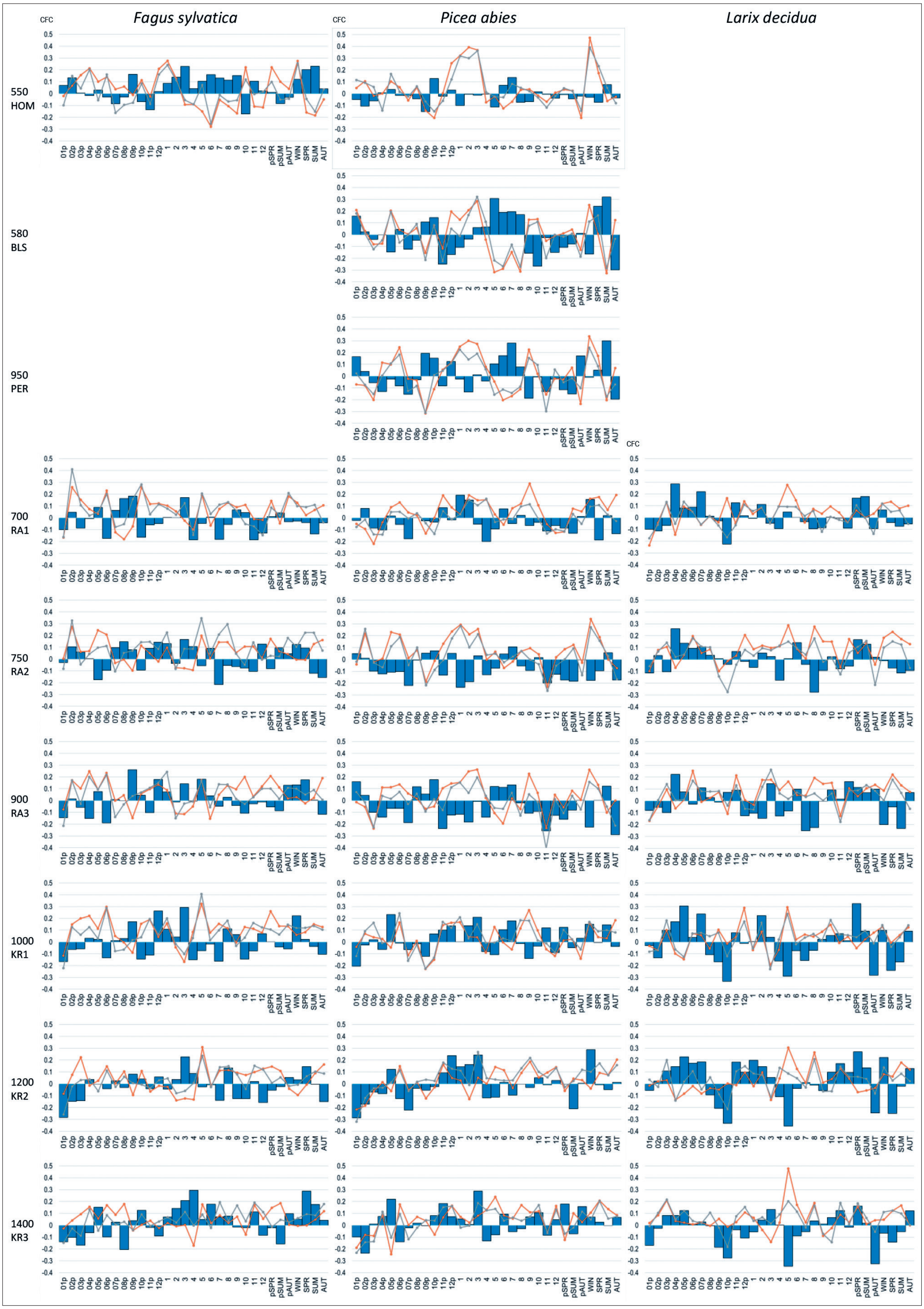



*Figure 3. Kokra-Jezersko correlation function coefficients between tree-ring width indices and climate variables (for details, see Figure 4 caption).*

*Slika 3. Kokra – Jezersko korelacijski koeficienti med indeksi širin branik in klimatskimi spremenljivkami (za podrobnosti glejte napis pod sliko 4).*


Fagus sylvatica
Picea abies
Larix decidua
CFC
550
HOM
580
BLS
950
PER
700
RA1
750
RA2
900
RA3
1000
KR1
1200
KR2
1400
KR3

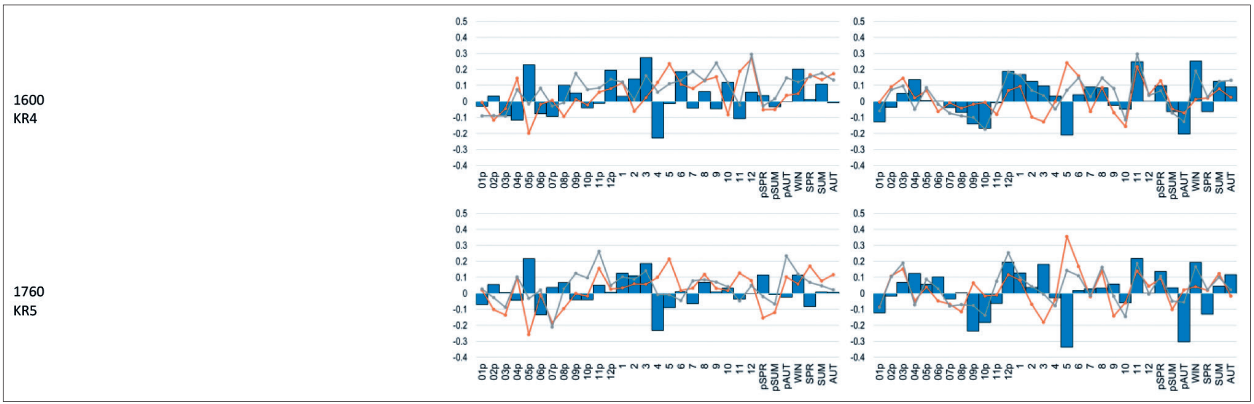



*Figure 4. Bled – Radovna – Krma: correlation function coefficients for the residual chronologies of European beech (FASY), Norway spruce (PCAB), European larch (LADE) from various elevations and monthly minimum (Tmin, gray lines) and maximum temperatures (Tmax, orange lines) and precipitation (PCP, bars) from the previous January (01p) to current December (12) and the past and current spring (pSPR, SPR), summer (pSUM, SUM), autumn (pAUT, AUT) and current winter (WIN) for the period 1950-2018. CFC values are statistically significant (p<0.05) if >0.2084 or <-0.2084 (for legend, see Figure 3).*

*Slika 4. Bled – Krma: korelacijski koeficienti med rezidualnimi verzijami kronologij za navadno bukev (FASY), navadno smreko (PCAB) in evropski macesen (LADE) ter mesečnimi minimalnimi (Tmin, sive črte) in maksimalnimi temperaturami (Tmax, oranžne črte) ter padavinami (PCP, stolpci) od preteklega januarja (01p) do decembra (12) tekočega leta, ter za preteklo in tekočo pomlad (pSPR, SPR), poletje (pSUM, SUM), jesen (pAUT, AUT) in zimo (WIN) za obdobje 1950–2018.Vrednosti koeficientov so statistično značilne (p<0,05), če so manjše od -0,2084, ali večje od 0,2084 (za legendo glejte sliko 3).*

The presented complex response to climatic parameters with similarities and differences among species and sites requires confirmation with further studies using principal component analysis (e.g., Čufar et al., 2014, 2008) or other methods.

### 3.2 COMPARISONS OF CHRONOLOGIES
### 3.2 PRIMERJAVA KRONOLOGIJ

The comparison of chronologies using tBP values shows that at the Kokra – Jezersko gradient most of the chronologies of the same species showed tBP≥4, which is considered to indicate statistically significant similarity (Table 3). Comparison of the chronologies J2, J3, J4 and J6 from locations on the same slope (Figure 2a) showed the highest similarity between nearby altitudes and smallest between the two extreme altitudes at 950 and 1600 m a.s.l. Heteroconnection, i.e. similarity between the chronologies of different species, is observed only occasionally (Table 3).

The cross-correlations at the Bled – Radovna – Krma gradient of the same species generally show some similarity between nearby locations and no similarity between lowest and highest elevations (Table 4). Highest similarity between the chronologies KR1, KR2, KR3 and KR4 could be partly explained by the location of the sites in the valley (Figure 2b) Heteroconnection is observed only occasionally (Table 4).

## 4 CONCLUSIONS
## 4 ZAKLJUČKI

The 47 tree-ring chronologies of Norway spruce (*Picea abies*), European larch (*Larix decidua*), and European beech (*Fagus sylvatica*) for two altitudinal ranges in the Kamnik-Savinja Alps and the Julian Alps, starting from the lowlands to the altitudinal limit of species distribution, show variability in tree-ring response to climate.

Correlation function coefficients (CFCs) for residual chronologies and monthly minimum (Tmin) and maximum temperatures (Tmax) and precipitation from the previous January to current December and from the previous and current spring, summer, autumn and current winter vary along the altitudinal gradient.

*Table 3. Cross-correlation values of tBP (t-value after Baillie and Picher) between Kokra – Jezersko raw chronologies (J1A-J6, maximum overlap) of Norway spruce, Picea abies, PCAB, European larch, Larix decidua, LADE, and European beech, Fagus sylvatica, FASY, from different altitudes. Values tBP≥4 with statistically significant similarity are marked.*

*Preglednica 3. Korelacijske vrednosti tBP (t-vrednost po Baillieju in Picherju) med surovimi kronologijami Kokra – Jezersko (J1A-J6, maksimalno prekrivanje) smreke, Picea abies, PCAB, evropskega macesna, Larix decidua, LADE in evropske bukve, Fagus sylvatica, FASY, z različnih nadmorskih višin. Vrednosti tBP≥4 so statistično značilne in so označene.*

| | J1A | J1B | J02 | J12 | J03 | J04 | J06 | J1B | J02 | J03 | J04 | J06 | J24 | J1A | J1B | J02 | J12 | J03 | J04 | J06 | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| J1A | 100 | | | | | | | | | | | | | | | | | | | | 750 PCAB |
| J1B | 6.7 | 100 | | | | | | | | | | | | | | | | | | | 780 PCAB |
| J02 | 8.5 | 3.1 | 100 | | | | | | | | | | | | | | | | | | 950 PCAB |
| J12 | 6.5 | 6.1 | 4.8 | 100 | | | | | | | | | | | | | | | | | 1200 PCAB |
| J03 | 7.5 | 7.7 | 6.9 | 6.3 | 100 | | | | | | | | | | | | | | | | 1250 PCAB |
| J04 | 6.4 | 3.9 | 6.5 | 4.6 | 5.3 | 100 | | | | | | | | | | | | | | | 1380 PCAB |
| J06 | 6.4 | 5.6 | 1.7 | 6.7 | 5.5 | 4.0 | 100 | | | | | | | | | | | | | | 1600 PCAB |
| J1B | 2.9 | 1.7 | 0.9 | 2.4 | 2.0 | 1.6 | 5.9 | 100 | | | | | | | | | | | | | 780 LADE |
| J02 | 6.6 | 4.8 | 3.6 | 4.2 | 6.5 | 4.5 | 5.5 | 5.8 | 100 | | | | | | | | | | | | 950 LADE |
| J03 | 4.5 | 2.3 | 0.9 | 3.5 | 3.4 | 4.1 | 4.6 | 6.5 | 7.7 | 100 | | | | | | | | | | | 1250 LADE |
| J04 | 3.4 | 3.6 | 1.0 | 3.8 | 3.3 | 3.9 | 4.1 | 6.0 | 10.5 | 9.8 | 100 | | | | | | | | | | 1380 LADE |
| J06 | 1.7 | 0.9 | 1.4 | 4.4 | 1.1 | 2.2 | 6.0 | 4.3 | 3.5 | 5.5 | 4.9 | 100 | | | | | | | | | 1600 LADE |
| J24 | 1.7 | 0.9 | 0.7 | 2.1 | 1.9 | 2.8 | 3.9 | 1.6 | 1.0 | 2.8 | 2.5 | 4.2 | 100 | | | | | | | | 1600 LADE |
| J1A | 3.1 | 2.1 | 2.7 | 1.1 | 1.8 | 3.5 | 2.5 | 1.6 | 3.0 | 1.0 | 0.9 | 1.0 | 0.0 | 100 | | | | | | | 750 FASY |
| J1B | 2.2 | 2.4 | 0.7 | 0.4 | 2.1 | 2.9 | 3.1 | 1.8 | 2.4 | 1.1 | 0.8 | 1.5 | 0.0 | 5.3 | 100 | | | | | | 780 FASY |
| J02 | 3.2 | 1.2 | 2.4 | 1.6 | 2.4 | 1.8 | 2.5 | 1.2 | 1.5 | 0.6 | 0.5 | 0.1 | 1.4 | 4.7 | 4.9 | 100 | | | | | 950 FASY |
| J12 | 2.5 | 3.1 | 0.1 | 2.1 | 1.5 | 2.2 | 5.5 | 2.5 | 1.5 | 1.2 | 0.6 | 3.0 | 1.5 | 3.5 | 6.6 | 4.0 | 100 | | | | 1200 FASY |
| J03 | 3.8 | 6.2 | 2.2 | 2.6 | 2.7 | 2.7 | 6.5 | 2.5 | 4.5 | 1.5 | 3.2 | 1.4 | 1.5 | 8.1 | 4.4 | 8.5 | 6.3 | 100 | | | 1250 FASY |
| J04 | 3.3 | 1.9 | 0.7 | 2.3 | 1.9 | 3.7 | 2.8 | 2.8 | 4.5 | 1.4 | 2.9 | 1.3 | 0.8 | 6.7 | 9.2 | 7.6 | 8.2 | 7.2 | 100 | | 1380 FASY |
| J06 | 1.5 | 1.7 | 0.7 | 0.7 | 1.6 | 2.6 | 2.9 | 3.6 | 0.9 | 2.4 | 2.1 | 2.6 | 1.8 | 2.8 | 4.8 | 4.9 | 6.4 | 3.2 | 4.4 | 100 | 1600 FASY |
| | 750 PCAB | 780 PCAB | 950 PCAB | 1200 PCAB | 1250 PCAB | 1380 PCAB | 1600 PCAB | 780 LADE | 950 LADE | 1250 LADE | 1380 LADE | 1600 LADE | 1600 LADE | 750 FASY | 780 FASY | 950 FASY | 1200 FASY | 1250 FASY | 1380 FASY | 1600 FASY | |

Standard dendrochronological parameters (tBP) calculated between the chronologies from the Kokra–Jezersko gradient showed that most of the chronologies of the same species along the gradient showed similarity (tBP≥4). In the subset of chronologies from locations on the same slope the greatest similarity was found between the nearby altitudes and smallest between the two extreme altitudes at 950 and 1600 m a.s.l. Heteroconnection, i.e. similarity between the chronologies of different species, was observed only occasionally. The relationships between the Bled – Radovna – Krma chronologies seem to be more complex, and require a detailed study.

The presented results show that the relationship between tree growth and climate is not only affected by altitude and the corresponding climatic conditions. The complex relationships need to be further investigated with an appropriate methodology, such as principal component analysis.

The database shows great potential for future studies of *spruce*, *larch*, and *beech* from cold environments in the southern Alps in a time of changing climate. The local chronologies with average lengths of 156 (69-296) years for *beech*, 139 (57-355) years for *spruce*, and 191 (56-378) years for *larch* also provide a basis for the construction of master chronologies for dating cultural heritage objects. In Slovenia and in the surrounding areas such

*Table 4. Cross-correlation values of tBP (t-value after Baillie and Picher) between Bled – Radovna – Krma raw chronologies (HOM-KR6, common period 1900-2020) of Norway spruce, Picea abies, PCAB, European larch, Larix decidua, LADE, and European beech, Fagus sylvatica, FASY, from different altitudes. Values of tBP≥4 with statistically significant similarity are marked.*

*Preglednica 4. Korelacijske vrednosti tBP (t-vrednost po Baillieju in Picherju) med surovimi kronologijami Bled – Radovna – Krma (HOM-KR6, skupno obdobje 1900–2020) smreke, Picea abies, PCAB, evropskega macesna, Larix decidua, LADE, in evropske bukve, Fagus sylvatica, FASY, z različnih nadmorskih višin. Vrednosti tBP≥4 so statistično značilne in so označene.*

| | HOM | BLS | RA1 | RA2 | RA3 | PER | KR1 | KR2 | KR3 | KR4 | KR5 | RA1 | RA2 | RA3 | KR1 | KR2 | KR3 | KR4 | KR5 | KR6 | HOM | RA1 | RA2 | RA3 | KR1 | KR2 | KR3 | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| HOM | 100 | | | | | | | | | | | | | | | | | | | | | | | | | | | 518 PCAB |
| BLS | 7.5 | 100 | | | | | | | | | | | | | | | | | | | | | | | | | | 580 PCAB |
| RA1 | 3.4 | 3.7 | 100 | | | | | | | | | | | | | | | | | | | | | | | | | 700 PCAB |
| RA2 | 4.0 | 4.9 | 5.0 | 100 | | | | | | | | | | | | | | | | | | | | | | | | 750 PCAB |
| RA3 | 5.0 | 6.5 | 2.8 | 5.5 | 100 | | | | | | | | | | | | | | | | | | | | | | | 900 PCAB |
| PER | 5.0 | 6.3 | 5.7 | 8.5 | 9.4 | 100 | | | | | | | | | | | | | | | | | | | | | | 950 PCAB |
| KR1 | 2.1 | 2.5 | 3.2 | 7.3 | 2.3 | 4.7 | 100 | | | | | | | | | | | | | | | | | | | | | 1000 PCAB |
| KR2 | 0.6 | 0.4 | 1.6 | 3.8 | 0.1 | 3.0 | 8.8 | 100 | | | | | | | | | | | | | | | | | | | | 1200 PCAB |
| KR3 | 1.4 | 0.5 | 0.9 | 1.8 | 1.3 | 1.0 | 4.5 | 2.9 | 100 | | | | | | | | | | | | | | | | | | | 1400 PCAB |
| KR4 | 0.0 | 0.0 | 2.6 | 0.7 | 0.7 | 2.1 | 2.4 | 5.9 | 2.6 | 100 | | | | | | | | | | | | | | | | | | 1600 PCAB |
| KR5 | 1.9 | 0.5 | 3.3 | 1.5 | 0.9 | 4.0 | 2.1 | 3.9 | 2.8 | 6.7 | 100 | | | | | | | | | | | | | | | | | 1760 PCAB |
| RA1 | 1.8 | 0.9 | 0.7 | 1.8 | 0.5 | 0.1 | 3.4 | 2.1 | 0.8 | 0.1 | 1.5 | 100 | | | | | | | | | | | | | | | | 700 LADE |
| RA2 | 2.5 | 2.9 | 3.9 | 6.3 | 3.2 | 3.2 | 5.2 | 2.4 | 0.8 | 0.4 | 1.2 | 6.7 | 100 | | | | | | | | | | | | | | | 750 LADE |
| RA3 | 3.2 | 2.0 | 3.1 | 2.6 | 2.4 | 2.6 | 2.2 | 3.0 | 1.2 | 1.6 | 2.3 | 4.7 | 5.6 | 100 | | | | | | | | | | | | | | 900 LADE |
| KR1 | 0.2 | 1.7 | 0.3 | 1.3 | 0.9 | 0.5 | 3.6 | 2.6 | 2.1 | 1.8 | 1.1 | 4.3 | 3.7 | 2.6 | 100 | | | | | | | | | | | | | 1000 LADE |
| KR2 | 1.0 | 0.8 | 1.1 | 0.1 | 0.8 | 0.7 | 4.6 | 4.3 | 3.5 | 3.6 | 2.2 | 4.3 | 3.1 | 2.2 | 5.7 | 100 | | | | | | | | | | | | 1200 LADE |
| KR3 | 3.3 | 3.1 | 0.6 | 0.6 | 1.3 | 1.6 | 3.3 | 3.1 | 3.6 | 3.2 | 0.4 | 2.3 | 0.8 | 1.0 | 3.9 | 7.7 | 100 | | | | | | | | | | | 1400 LADE |
| KR4 | 1.7 | 1.7 | 0.4 | 0.7 | 1.7 | 0.7 | 3.0 | 4.9 | 1.9 | 4.5 | 1.1 | 2.0 | 2.3 | 0.2 | 3.5 | 8.9 | 11.4 | 100 | | | | | | | | | | 1600 LADE |
| KR5 | 2.0 | 0.8 | 0.2 | 0.0 | 2.2 | 0.3 | 2.9 | 5.4 | 2.3 | 3.4 | 1.4 | 2.3 | 2.5 | 0.7 | 2.6 | 8.2 | 9.0 | 16.9 | 100 | | | | | | | | | 1760 LADE |
| KR6 | 2.4 | 1.5 | 1.6 | 3.2 | 1.7 | 1.3 | 0.9 | 3.2 | 1.3 | 3.6 | 1.7 | 0.3 | 0.1 | 1.2 | 0.8 | 4.9 | 5.3 | 8.7 | 9.0 | 100 | | | | | | | | 1950 LADE |
| HOM | 2.9 | 4.3 | 0.1 | 3.1 | 2.2 | 2.8 | 2.5 | 0.7 | 1.0 | 0.4 | 1.2 | 1.1 | 1.2 | 0.4 | 2.4 | 0.3 | 1.0 | 0.5 | 0.0 | 0.6 | 100 | | | | | | | 550 FASY |
| RA1 | 2.4 | 1.7 | 2.9 | 3.8 | 2.3 | 2.1 | 1.5 | 2.0 | 0.7 | 2.0 | 2.3 | 0.8 | 1.8 | 2.2 | 1.1 | 0.6 | 0.3 | 0.4 | 1.0 | 0.3 | 2.0 | 100 | | | | | | 700 FASY |
| RA2 | 0.1 | 0.1 | 0.7 | 3.0 | 1.2 | 1.1 | 2.1 | 2.6 | 0.6 | 1.3 | 0.2 | 0.7 | 1.3 | 1.3 | 0.5 | 2.1 | 1.0 | 2.4 | 2.2 | 0.5 | 3.2 | 5.4 | 100 | | | | | 750 FASY |
| RA3 | 0.4 | 0.4 | 0.1 | 2.5 | 0.9 | 0.9 | 2.7 | 2.5 | 0.8 | 1.6 | 0.6 | 0.8 | 2.3 | 2.5 | 1.6 | 1.8 | 2.1 | 2.6 | 2.4 | 1.0 | 2.4 | 1.9 | 5.0 | 100 | | | | 900 FASY |
| KR1 | 0.8 | 1.3 | 0.7 | 2.6 | 0.8 | 0.4 | 2.9 | 3.0 | 1.1 | 1.9 | 1.2 | 1.5 | 1.7 | 0.8 | 4.0 | 2.4 | 2.8 | 2.9 | 2.4 | 1.5 | 3.5 | 3.4 | 5.4 | 7.7 | 100 | | | 1000 FASY |
| KR2 | 1.2 | 0.7 | 0.8 | 0.0 | 1.3 | 0.9 | 3.5 | 4.7 | 2.2 | 3.2 | 1.5 | 1.5 | 0.5 | 0.6 | 3.9 | 4.7 | 4.7 | 4.6 | 4.4 | 5.8 | 2.4 | 1.5 | 3.4 | 4.0 | 6.3 | 100 | | 1200 FASY |
| KR3 | 0.1 | 1.2 | 0.4 | 0.2 | 1.3 | 1.0 | 3.0 | 4.7 | 1.4 | 4.6 | 2.4 | 2.1 | 0.4 | 1.1 | 2.6 | 3.4 | 3.6 | 5.4 | 5.1 | 5.7 | 1.4 | 2.5 | 2.2 | 1.6 | 3.1 | 9.4 | 100 | 1400 FASY |
| | 518 PCAB | 580 PCAB | 700 PCAB | 750 PCAB | 900 PCAB | 950 PCAB | 1000 PCAB | 1200 PCAB | 1400 PCAB | 1600 PCAB | 1760 PCAB | 700 LADE | 750 LADE | 900 LADE | 1000 LADE | 1200 LADE | 1400 LADE | 1600 LADE | 1760 LADE | 1950 LADE | 550 FASY | 700 FASY | 750 FASY | 900 FASY | 1000 FASY | 1200 FASY | 1400 FASY | |

chronologies are particularly needed for spruce (e.g., Bernabei et al., 2017; Čufar et al., 2020).

The sampling material and data are stored in the archive of the Chair for Wood Science at the Department of Wood Science and Technology, Biotechnical Faculty, University of Ljubljana.

## 5 SUMMARY

## 5 POVZETEK

Predstavljamo mrežo lokalnih kronologij navadne smreke (*Picea abies*), evropskega macesna (*Larix decidua*) in navadne bukve (*Fagus sylvatica*) vzdolž dveh gradientov nadmorskih višin na območju Kokra – Jezersko v Kamniško Savinjskih Alpah in Bled – Radovna – Krma v Julijskih Alpah v Sloveniji. Vsaka od izbranih vrst ima svoje posebnosti z vidika dendrokronologije. Bukev, ki je v Evropi in Sloveniji zelo razširjena, pogosto uporabljamo kot modelno vrsto v dendroekologiji ter za proučevanje učinkov klimatskih sprememb na vegetacijo (npr. Čufar et al., 2008; Di Filippo et al., 2007; Martínez del Castillo et al., 2019, 2022). Redkeje se srečamo z bukovimi predmeti iz preteklih obdobij, ki bi jih želeli dendrokronološko datirati (Čufar et al., 2012). Smreka je prav tako zelo razširjena v Evropi in Sloveniji. V splošnem je to vrsta hladnih okolij, ki so jo v zadnjih dvesto letih razširili tudi na manj primerna rastišča na nižjih nadmorskih višinah. Pogosto jo proučujemo kot modelno drevesno vrsto v dendroekologiji (npr. Martinez del Castillo et al., 2018). Smreka je pogosta v zgodovinskih konstrukcijah, predmetih in glasbenih inštrumentih. Dendrokronološki signal smreke po Sloveniji zelo variira, ker je v veliki meri odvisen od nadmorske višine, zato za Slovenijo še nismo uspeli sestaviti dobrih referenčnih kronologij za datiranje (npr. Bernabei et al., 2017; Čufar et al., 2020). Macesen je v Sloveniji manj razširjen kot bukev in smreka (ZGS, 2021), a je pomemben z vidika uporabe lesa, ter tudi za dendrokronološke raziskave. Kot cenjena lesna vrsta je pogost tudi v predmetih kulturne dediščine. Macesen z visokih nadmorskih višin, ki je bil uporabljen za prestižne konstrukcije Benečanov, ima dobro telekonekcijo (Levanič et al., 2001). Tudi dendrokronološki signal macesna je zelo odvisen od nadmorske višine, zato za nižje nadmorske višine še nimamo ustreznih kronologij za datiranje.

Cilj te študije je bil predstaviti (1) kronologije in njihove glavne značilnosti, (2) kako podnebni dejavniki vplivajo na variiranje širin branik, (3) kako se dendrokronološki signal posamezne vrste spreminja z nadmorsko višino in (4) kakšen potencial ima predstavljena podatkovna zbirka za bodoče raziskave na področju ekologije in kulturne dediščine.

Vzorce lesa za raziskave smo pridobili na različnih nadmorskih višinah: (1) Kokra-Jezersko, na 750, 780, 950, 1200, 1250, 1380 in 1600 m in (2) Bled – Radovna – Krma z rastišči na 518, 550, 580, 700, 750, 900, 950, 1000, 1200, 1400, 1600, 1760, 1900, 2040 m n. v.

Na vsakem rastišču smo v času redne sečnje iz posekanih dreves na nivoju 4 m od baze drevesa odžagali kolut. V kolikor število posekanih dreves ni bilo zadostno, smo iz rastočih dreves na posameznem rastišču odvzeli izvrtke.

Prečne prereze vzorcev smo gladko zbrusili in jih skenirali pri ločljivosti 1200 dpi. Na slikah smo izmerili širine branik s programom CDendro / Cooo Recorder 9.5 (Cybis Elektronik, 2022 http://www.cybis.se/forfun/dendro/helpcoorecorder7/index.php). Za vizualno in statistično sinhronizacijo smo uporabili program TSAP-Win (Frank Rinn, Heidelberg, Nemčija) in paket dplR v programu R Studio (Bunn, 2010).

Sinhronizirana in datirana zaporedja širin branik smo uporabili za sestavo lokalnih kronologij s programom R Studio z uporabo paketa dplR (Bunn, 2008). Izračunali smo tri različice kronologij: kronologijo širin branik ter standardno in rezidualno kronologijo. Za proučevanje vpliva klime na rast dreves smo uporabili rezidualno kronologijo z uporabo programa R Studio. Lokalni vremenski podatki za izračune so bili pridobljeni iz podatkovne baze SLOCLIM (Škrk et al., 2021).

Opravili smo osnovne primerjave med kronologijami z izračunom standardnih statističnih vrednosti (predvsem t-vrednost Baillie in Pilcher, tBP) s programom TSAP-Win.

Kronologije smo testirali tudi z vidika telekonekcije (ujemanje med kronologijami iste vrste z različnih lokacij) in heterokonekcije (ujemanje med kronologijami različnih drevesnih vrst z istega območja).

Za vseh 47 kronologij za 3 drevesne vrste smo predstavili natančne zemljepisne koordinate, število dreves, uporabno dolžino ter prvo in zadnje leto kronologije (preglednica 1 in 2).

Za vseh 47 kronologij 3 drevesnih vrst smo predstavili korelacijske koeficiente (CFC) med indeksi širin branik (rezidualne kronologije) ter mesečnimi minimalnimi (Tmin) in maksimalnimi temperaturami (Tmax), padavinami (PCP) od preteklega januarja do decembra tekočega leta, ter za preteklo in tekočo pomlad, poletje, jesen in zimo za obdobje 1950–2018.

Rezultati za Kokro – Jezersko (slika 3) kažejo, da se bukev negativno odziva na junijske temperature (zlasti Tmax), pozitivno pa na junijske padavine. Vrednosti korelacijskih koeficientov (CFC) se na splošno zmanjšujejo od nižje proti višji nadmorski višini, medtem ko na nadmorski višini 1600 m opažamo pozitiven odziv na temperature v juliju, avgustu in septembru. Smreka se negativno odziva na temperature julija in avgusta ter pozitivno na padavine julija na večini gradienta, medtem ko je na 1600 m nadmorske višine opazen pozitiven odziv na temperature v maju in avgustu. Macesen kaže negativen odziv na Tmax v marcu in pozitiven odziv na poletne temperature, medtem ko na nadmorski višini nad 1250 m opažamo pozitiven odziv na majske temperature, zlasti Tmax, in pozitiven odziv na avgustovske temperature z nižjimi vrednostmi korelacijskih koeficientov.

Rezultati na območju Bled – Radovna – Krma se razlikujejo od rezultatov na Jezerskem. Bukev kaže negativen odziv na junijske temperature na Homu (518 m n. v.), medtem ko se na rastiščih v Radovni in Krmi drevesa odzivajo večinoma pozitivno na majske temperature in negativno na marčevske.

Smreka na nižjih nadmorskih višinah kaže pozitiven odziv na januarske, februarske in marčevske temperature, medtem ko je na nadmorskih višinah nad 1000 m opazen pozitiven vpliv majskih temperatur. Macesen kaže negativen odziv na Tmax v marcu in pozitiven odziv na temperature v maju.

Ker je odziv različnih vrst na različnih nadmorskih višinah na dveh območjih zelo kompleksen, bi bilo dobljene podatke treba analizirati še z drugimi metodami, na primer z analizo glavnih komponent (npr. Čufar et al., 2008, 2014).

Navzkrižne korelacije z izračunom parametra tBP (kjer tBP≥4 pomeni statistično značilno podobnost), kažejo, da je na Jezerskem večina kronologij vsaj v določeni meri podobnih (tBP≥4) (preglednica 3). Primerjava kronologij J2, J3, J4 in J6 z lokacij z enako ekspozicijo (slika 2a) je pokazala največjo podobnost med bližnjimi nadmorskimi višinami in najmanjšo med dvema skrajnima nadmorskima višinama na 950 in 1600 m. Heterokonekcija, tj. primerjava kronologij različnih vrst, je pokazala, da imajo različne vrste na isti lokaciji podoben dendrokronološki signal samo v posameznih primerih (preglednica 3).

Korelacije med kronologijami vzdolž gradienta Bled – Radovna – Krma kažejo nekaj podobnosti iste vrste na bližnjih lokacijah. Med kronologijami z najnižjih in najvišjih nadmorskih višin pa ni bilo podobnosti v dendrokronološkem signalu (preglednica 4). Najbolj so si bile podobne kronologije KR1, KR2, KR3 in KR4, kar bi lahko delno pojasnili z lego rastišč v isti dolini (slika 2b). Podobnost dendrokronoloških signalov med vrstami (heterokonekcija) je bila zabeležena le v nekaj primerih (preglednica 4).

Predstavljeni rezultati kažejo, da na rast (variiranje širin branik) ne vplivajo le nadmorska višina in pripadajoče podnebne razmere. Kompleksne odnose med kronologijami bi bilo treba dodatno raziskati.

Prikazani rezultati kažejo na velik potencial podatkovne zbirke za prihodnje študije dendrokronoloških posebnosti vrst navadne smreke (*Picea abies)*, evropskega macesna (*Larix decidua)* in navadne bukve (*Fagus sylvatica)* iz hladnih okolij v južnih Alpah v spreminjajočem se podnebju. Lokalne kronologije, ki so v povprečju dolge 156 (69-296) let za bukev, 139 (57-355) za smreko in 191 (56-378) let za macesen, predstavljajo tudi osnovo za izdelavo sestavljenih referenčnih kronologij, ki jih zlasti za smreko potrebujemo za datiranje lesenih predmetov kulturne dediščine (prim. Čufar et al., 2020).

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# CRITICAL STEPS AND TROUBLESHOOTING IN SAMPLE PREPARATION FOR WOOD AND PHLOEM FORMATION: FROM SAMPLING TO MICROSCOPIC OBSERVATION

# KRITIČNI KORAKI IN REŠEVANJE TEŽAV PRI PRIPRAVI VZORCEV ZA SPREMLJANJE NASTAJANJA LESA IN FLOEMA: OD VZORČENJA DO OPAZOVANJA POD MIKROSKOPOM

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## Abstract / Izvleček

**Abstract:** We present a technical note that supplements published procedures on optimal sample preparation for performing wood and phloem formation analyses. Before beginning sampling, it is important to learn about the characteristics of the tree or shrub species to be investigated. Some tips are given how to use the Trephor tool in the best way, how to remove the outer hard bark (periderm), how microcores should be handled after removal from the tree, and how they should be oriented for embedding in paraffin, and cutting thin sections for microscopy. Possible defects that may result from improper handling are illustrated and discussed. We also present optimal images to accurately identify different cell development stages in phloem and xylem, which is particularly challenging in hardwoods and Mediterranean tree and shrub species.

**Keywords:** tissue sampling, tissue preparation, microscopic slides, microscopy, xylogenesis, phloem formation, troubleshooting

**Izvleček:** Predstavljamo tehnična navodila, ki dopolnjujejo objavljene postopke za optimalno pripravo vzorcev za izvajanje analiz nastajanja lesa in floema. Pred začetkom vzorčenja je potrebno poznati značilnosti drevesne ali grmovne vrste, ki jo želimo preiskovati. Podanih je nekaj nasvetov, kako najbolje uporabiti orodje Trephor, kako odstraniti zunanjo trdo skorjo (periderm), kako ravnati z mikro izvrtki po odvzemu iz debla in kako jih orientirati za vklapljanje v parafin in rezanje tankih rezin za mikroskopske preiskave. Prikazane in razložene so možne napake, ki lahko nastanejo zaradi neustreznega ravnanja v različnih korakih postopka. Predstavljamo tudi optimalne slike tkiv za prepoznavanje različnih razvojnih faz celic v floemu in ksilemu, ki je še posebej zahtevno pri listavcih ter sredozemskih drevesnih in grmovnih vrstah.

**Ključne besede:** odvzem vzorcev tkiva, priprava vzorcev, mikroskopski preparati, mikroskopiranje, nastajanje lesa, nastajanje floema, odpravljanje težav

## 1 INTRODUCTION
## 1 UVOD

The microcoring technique is increasingly used to obtain samples for monitoring tree responses to changing environmental conditions and assessing tree plasticity in the context of climate change. Analysis of microscopic specimens makes it possible to monitor cambial activity and wood and phloem formation in real time, and to distinguish the different phases of cell division, differentiation, and maturation and determine how and when they occur. To this end, samples (usually microcores) containing phloem, cambium, and xylem are taken weekly or biweekly from living trees, for at least one calendar year, with particular emphasis on the growing season. The most effective tool for sampling micro-

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cores is the Trephor tool (Rossi et al., 2006), which was developed for this purpose and first used in 2004. Since its first use, several protocols for sampling of microcores and sample preparation have been developed to monitor the different stages of wood and phloem formation (e.g., Rossi et al., 2006; Deslauriers et al., 2015; Prislan et al., 2014a, b, 2022; De Micco et al., 2019; Pace, 2019).

In addition, various protocols for preparing anatomical slides (including embedding, cutting and staining) have been developed and improved to achieve high-quality slides for microscopy and quantitative anatomy, including image analysis (Deslauriers et al., 2015; De Micco et al., 2019; Prislan et al., 2014a, b, 2022). Appropriate microscopy techniques are critical to accurately identify xylem and phloem cells during differentiation. Various histological and microscopic techniques are used to analyse and identify the different stages of cell division and differentiation (e.g., Balzano et al., 2021a).

Despite the availability of detailed protocols, mistakes can still happen, especially if one is a novice and there are no experts available to help with critical steps. Many technical errors can occur at all stages of the process, from sampling to quantification errors. Errors in live tissue collection can jeopardise the entire research. Repeating systematic errors at this stage runs the risk of collecting hundreds of samples over the course of a year that later prove useless for analyses.

The quality of thin sections can also be influenced by many factors: the tissue sample (microcore) itself (density, hardness, homogeneity, etc.), its storage (appropriate solution), sample preparation (possible trimming), orientation (anatomical orientation and orientation of the sample during sectioning), type and sharpness of the blade, and last but not least, the experience and skills of the operator.

Such errors may affect the measurement of cells, especially if it is done automatically, making it difficult to count cell number and measure cell dimensions, or leading to inaccurate results and interpretations.

In our experience, methods should be slightly adjusted on a case-by-case basis, as each species is different. It is also very important to document the species under study well (density, wood and phloem anatomy…) and to perform technical tests before starting the actual experiment.

Most of the techniques developed for the study of xylogenesis have been applied to conifers growing in temperate climates, characterized by fairly regular cambial activity and relatively homogeneous wood structure. In Mediterranean and tropical species, whose wood is often characterized by high density and hardness, the sampling of microcores can be technically challenging. Precautions must also be taken, and existing protocols adapted when sampling shrubs.

The goal of this study is to improve the published protocols, help novices with critical steps, and focus on technical issues that are not necessarily addressed in detail in existing protocols. We want to report on the most common mistakes that are made and show possible solutions to overcome them, as well as share our own tips. We want to note some bad examples and explain their causes and solutions.

## 2 MATERIALS AND METHODS
## 2 MATERIALI IN METODE

The standard sample preparation protocol for wood and phloem formation analyses consists of: (1) collection of tissues (microcores) from living trees using a Trephor tool, (2) fixation of tissues with a solution of ethanol, formalin, and acetic acid, (3) storage in ethanol, (4) trimming of microcores, (5) dehydration and infiltration with paraffin, (6) embedding in paraffin, (7) trimming and cutting of (transverse) sections with a rotary microtome, (8) staining, (9) embedding of slides on objective glasses, (10) microscopy (different light modes), (11) image acquisition, (12) recognition of tissues and tissue parts, including determination of different stages of cell and cell wall development, (13) measurement of cells and cell parts (e.g. cell walls) – image analysis, (14) interpretation of results.

This article is a technical note describing the critical steps in the sample preparation protocol for wood and phloem formation analyses, and how to better handle the most critical steps in the process that often lead to poor quality microscopic slides and problems in measuring tissue elements (image analysis) and interpreting results.

# 3 RESULTS
# 3 REZULTATI
## 3.1 SAMPLING AND SAMPLE FIXATION – CRITICAL POINTS
## 3.1 VZORČENJE IN FIKSIRANJE VZORCEV – KRITIČNE TOČKE

Before taking tissue samples from trees, one should carefully study the characteristics of the tree species to be studied: its wood and bark anatomy (how dense and hard is the wood, peculiarities and thickness of the bark) as well as the time and duration of the growing season. Before starting the actual experiment, collect additional specimens for practice. Both novice and experienced scientists may otherwise collect a large number of unsuitable specimens, limiting the possibility of making suitable sections. During the experiment it is desirable to collect specimens throughout the calendar year to avoid missing crucial milestones, such as the beginning and end of cell production by the cambium.

The procedure for sampling and processing microcores is described in Prislan et al. (2022), and this article also includes some critical points and potential errors to avoid.

During sampling the use of a Trephor tool wounds the tissue, resulting in the production of wound tissue that is different from the tissues of interest. To avoid wounding effects, successive samples for wood and phloem formation should be taken in a spiral pattern along the stem, with sampling sites at least 10 cm apart. Two microcores are usually taken from each tree on a given day to have a reserve in case one of the two cores is not suitable. In the case of small trees or shrubs, such as grapevine (*Vitis vinifera*) (Figure 1), the sample is taken from a lower location and it is not possible to take two microcores because the sampling would cause too much damage. In this case, it is recommended to select several individuals for analysis and sample them in turn.

The quality of thin sections depends on the quality of the microcores, which is influenced by the sharpness of the Trephor cutting edge. A blunt cutting edge will cause damage to the microcores (e.g., compressed, partially crushed, broken, or twisted cores), which directly affects the quality of the thin sections (Figures 1, 2, 3).

Choose the correct size of Trephor depending on the species studied. A standard size cutting dimeter (1.9 mm) is appropriate for conifers and hardwoods with small vessels and homogeneous wood structure. A larger Trephor (cutting diameter

*Figure 1. Sampling tissues from the stem of grapevine (Vitis vinifera): removal of the outer bark (left) and taking microcores with a Trephor tool (right).*

*Slika 1. Vzorčenje tkiv iz stebla vinske trte (Vitis vinifera): odstranitev zunanje skorje (levo) ter odvzem mikro izvrtka z orodjem Trephor (desno).*

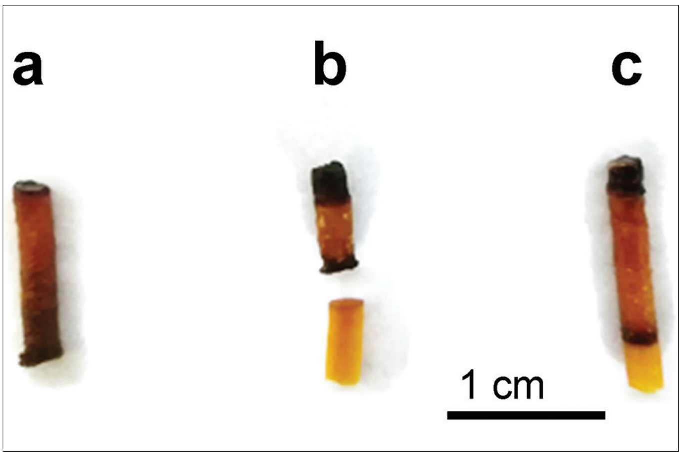


*Figure 2. Damaged microcores due to errors in sampling tissues from a living tree: (a) sample consisting only of bark (more bark tissue should be removed before sampling), (b) broken sample with crushed cambium (blunt edge of Trephor), (c) sample with too little wood tissue.*

*Slika 2. Poškodovani mikro izvrtki zaradi napak pri odvzemu vzorcev iz živih dreves: (a) vzorec vsebuje samo skorjo (pred vzorčenjem bi bilo treba odstraniti več skorje), (b) zlomljen vzorec s porušenim kambijem (topo rezilo Trephorja), (c) vzorec s premalo lesnega tkiva.*

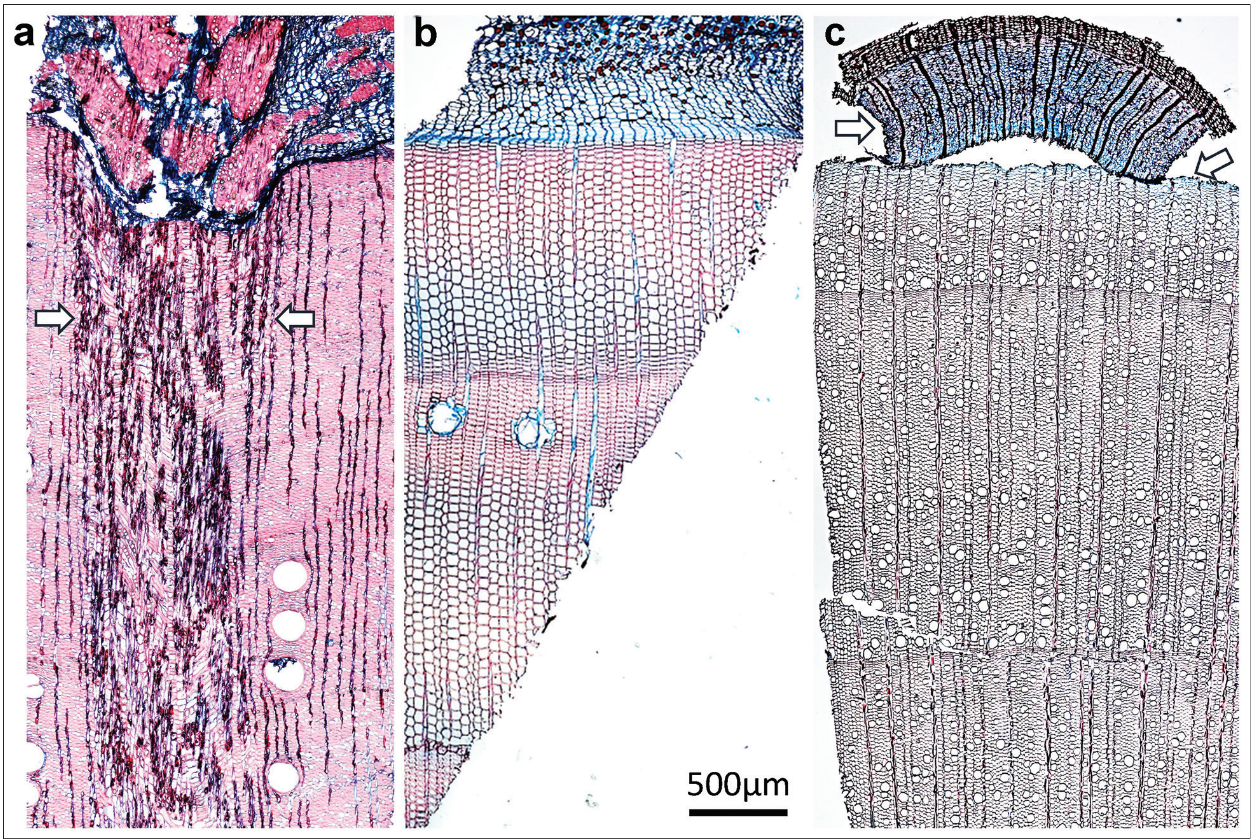


*Figure 3. Sampling errors reflected in the quality of thin sections: (a) cross-section of holm oak (Quercus ilex), whose wood and bark are mainly ray tissue (white arrows) (larger diameter Trephor should be used), (b) sample of stone pine (Pinus pinea) not taken perpendicular to the stem, (c) sample of strawberry tree (Arbutus unedo) which was not properly stored in the fixation medium (the sample was dried out, cambium is crushed, phloem is shrunken – white arrows).*

*Slika 3. Napake pri vzorčenju, ki se odražajo v kakovosti tankih rezin: (a) preparat tkiv črničevja (Quercus ilex) večinoma zavzema širok trak (beli puščici), (uporabiti bi bilo treba Trephor večjega premera), (b) vzorec pinije (Pinus pinea) ni bil odvzet pravokotno na deblo, (c) vzorec navadne jagodičnice (Arbutus unedo), po odvzemu iz drevesa ni bil pravilno shranjen v fiksacijskem mediju (vzorec se je posušil, kambij je porušen, floem se je skrčil (beli puščici)).*

> 2 mm) would help to obtain wider tissue sections for analyses. This is especially important when studying species with wider rays such as beech (*Fagus*) and oaks (*Quercus*) (Balzano et al., 2021b). Too narrow sections and large rays hinder the analyses, as in the case of *Quercus ilex* (Figure 3a).

Figures 1, 2, and 3 show sampling and common sampling errors and their effects on thin sections. Care must be taken to remove the correct amount of bark to avoid the risk of collecting only bark tissue (Figures 2a, c). The depth of the flap to be removed must be based on the depth of the bark, leaving the uncollapsed phloem and cambium intact. If the Trephor is not sharp enough, the bark and wood may separate (Figure 2b). It is very important to pierce the stem perpendicularly in the spot where the bark has been removed (Figure 3b). Hold the Trephor securely and then hammer firmly and vigorously until the entire tip of the Trephor penetrates the tissues.

It is important to place specimens in the fixation medium (FAA) immediately after collection. Since the purpose of fixation is to immobilize proteins and cellular components to maintain the structural integrity of the tissues, it is imperative that this step is done quickly. If the sample is not stored properly, the softer tissues such as the cambium and parenchyma cells will rapidly collapse and be unusable for measurements (Figure 3c).

### 3.2 SAMPLE PREPARATION AND CUTTING – CRITICAL POINTS
### 3.2 PRIPRAVA VZORCEV IN REZANJE – KRITIČNE TOČKE

Before dehydration and infiltration with paraffin, the microcores must be perfectly oriented using a scale magnifier to identify the transverse plane and mark it with a waterproof pencil. In this way, we can easily arrange the sample in the desired orientation for cutting after the paraffin embedding.

The transverse plane often appears darker than the radial plane. If there is any doubt about the orientation, it can be determined by placing a dot in the centre of the end of the microcore. In this way, the ink will follow the fibre orientation and reach the cross section. If the sample is not well oriented on the thin sections, the tissue will appear disoriented and will not match the normal structure of the cross-section, making it impossible to identify and properly measure the cells (Figure 4a). The microcore is later placed at an angle to the mould and with the bark down so that the blade encounters the least possible resistance when cutting.

There are several problems when cutting. Because of the very heterogeneous tissue, consisting of thin- and thick-walled cells with different lumen sizes, the microcore often breaks in the area of the cambium with extremely thin-walled cells, which is then destroyed. Therefore, it is extremely difficult to produce thin sections without defects such as cracked and collapsed areas.

Perfectly sharp blades for cutting are also essential to avoid the specimen splitting, collapsing, tearing off cells and leaving a blade track (Figure 4b, c, d). This is especially true for very dense and hard tissue, where it is often necessary to change the position of the blade for each cut, so that a previously unused, perfectly sharp part of the blade can be used.

To achieve perfect cuts, it is also important to properly adjust the angle of the blade correctly. The greater the angle, the less the impact on the specimen; therefore, it is advisable to increase the angle when the bark is harder. The optimum angle is usually between 5 and 10°.

Cutting speed is also an important factor when cutting. Cutting too slowly hinders the formation of the "wax ribbon", while cutting too fast can result in highly compressed sections that are difficult to expand.

A cutting thickness of 9 µm is sufficient for studying wood and phloem formation. However, if the bark or wood is too hard, it is recommended to prepare smaller cutting blocks and make thinner sections. This will provide less resistance to the knife and allow better sections to be made. A slight variation in the thickness of the section can often solve cutting problems.

When it comes to preparing the slide, we can save space by placing as many sections as possible on it. This can only be achieved if the ribbon consisting of paraffin and thin sections is straight. Before cutting, it is useful to trim away any excess wax and tissue to maximize the number of sections that can be placed on a slide. The smaller the block area, the easier it is to make a ribbon. During sectioning, water is applied with a fine brush to constantly keep both the sample and edge of the blade wet, which helps the section to slide across the blade when cutting.

The ribbons containing the cross-sections of microcore should be checked under the microscope to be sure you are selecting the best one. Although this is time consuming, it will facilitate and expedite further analyses.

Another critical step in sample preparation is the application of glycerine albumin to the slide. The key here is to apply the right amount of glycerine albumin; if we apply too much, the end result will be an opaque sample, if we apply too little, the section will be washed away from the slide in further steps. One drop from a generic disposable pipette to measure 3 ml of liquid is more or less the right amount.

Excess paraffin must be properly removed from the slides. The clearing agent (e.g., D-limonene) should be changed frequently to avoid dirty samples (Figure 4c). Also, be sure to leave the sample in the staining solution for as long as necessary (15 minutes) to avoid weak staining or overstaining. The safranin and astra blue aqueous solution is optimal for this purpose, as it allows observation in brightfield, polarized light, and epi-fluorescence modes (Balzano et al., 2021a).

During dehydration prior to embedding in Euparal, the alcohol used must not contain water, otherwise the slide will fog (Figure 4d). In addition, the Euparal embedding medium must be well dosed to avoid stains on the coverslip, which prevent observation (Figure 4e). If not enough Euparal is applied, air bubbles will form.

### 3.3 EXPECTED RESULTS
### 3.3 PRIČAKOVANI REZULTATI

Following the protocol (Prislan et al., 2022) and considering the tips suggested here should help to

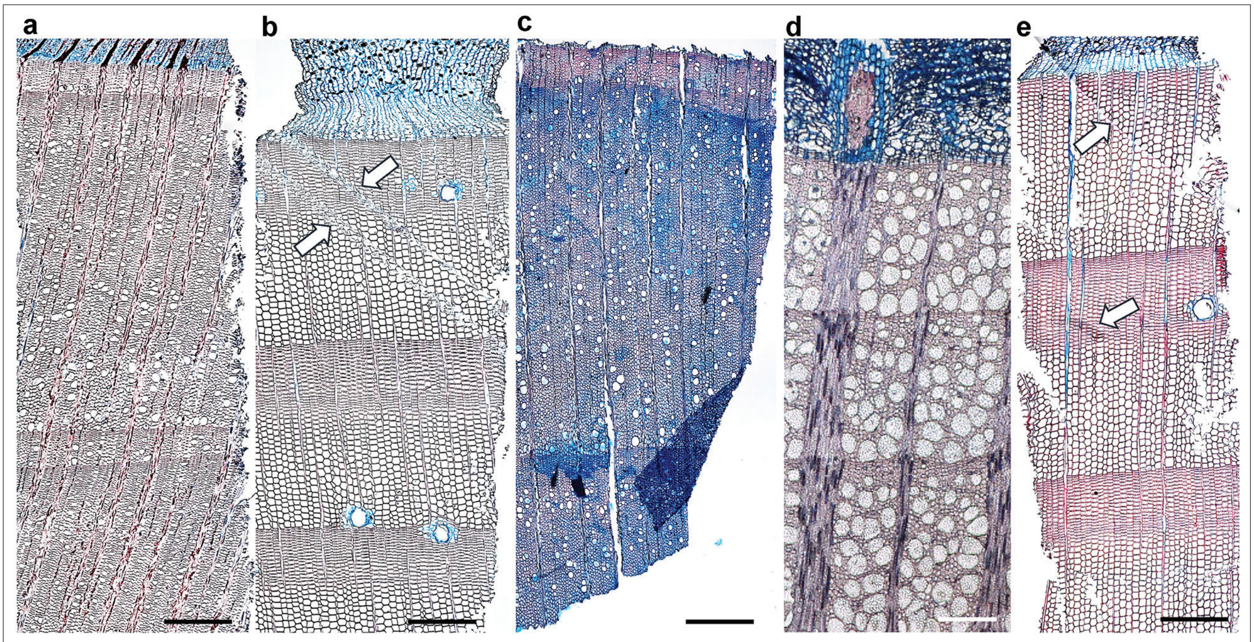


*Figure 4. Sample preparation errors: (a) microcore was not well oriented, (b) section with knife marks due to a blunt blade (white arrows), (c) section contaminated, overstained and split due to a blunt blade, (d) "foggy" slide due to presence of water in alcohol, (e) section split due to a blunt blade and traces of Euparal on the coverslip (white arrows). Black bars = 500 µm, white bar = 200 µm.*

*Slika 4. Napake pri pripravi vzorcev in preparatov: (a) mikro izvrtek ni bil dobro orientiran, (b) rezina s poškodbami zaradi topega rezila (beli puščici), (c) preparat vsebuje nečistoče, preveč obarvan in razcepljen zaradi topega rezila, (d) rezina motna zaradi nepopolne dehidracije (vode v alkoholu), (e) s poškodbami zaradi topega rezila in sledovi euparala na krovnem steklu (beli puščici). Črne merilne daljice = 500 µm, bela merilna daljica = 200 µm.*

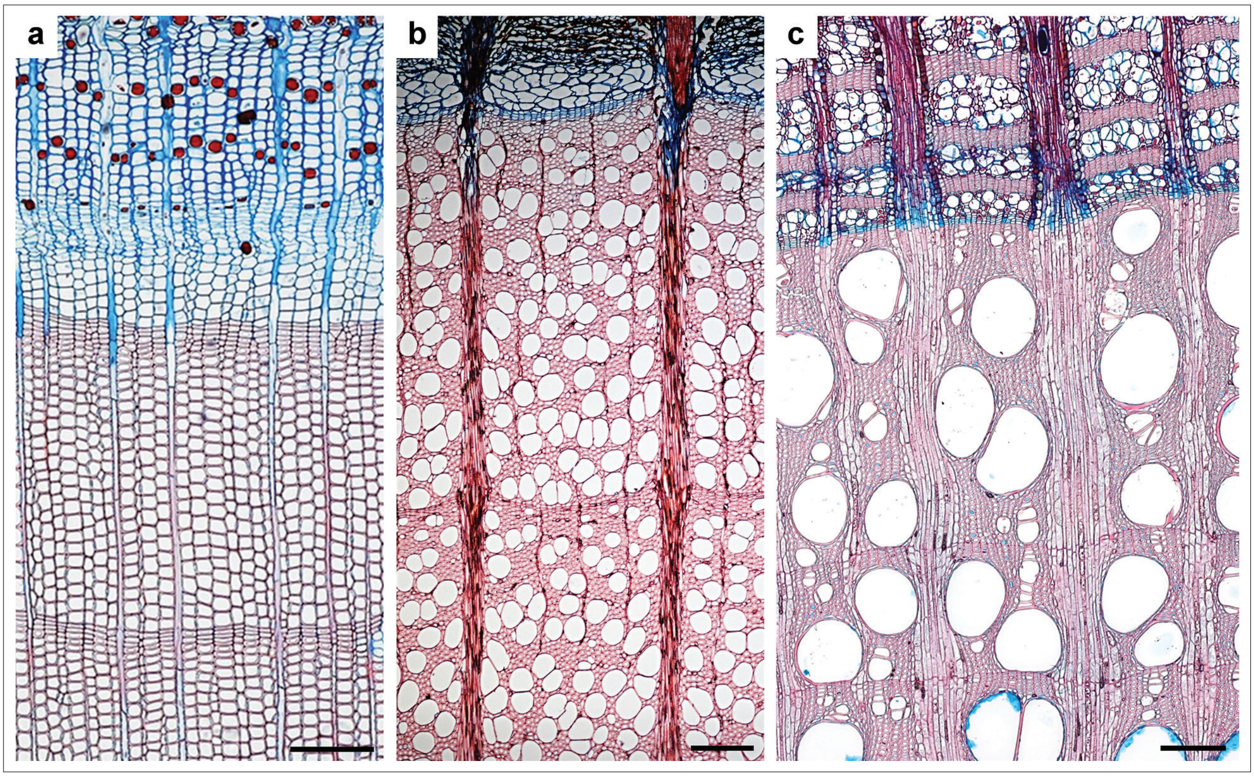


*Figure 5. Examples of high-quality cross-sections for quantitative image analyses of wood, cambium and phloem of: (a) Pinus halepensis, (b) Fagus sylvatica, and (c) Vitis vinifera. Scale bars = 200 µm.*
*Slika 5. Primeri visokokakovostnih prečnih prerezov za kvantitativno analizo slik tkiv lesa, kambija in floema pri vrstah: (a) Pinus halepensis, (b) Fagus sylvatica in (c) Vitis vinifera. Merilna daljica = 200 µm.*

obtain high-quality slides and images needed for subsequent analyses (Figure 5).

For successful analysis of wood and phloem formation, it is critical to obtain high-quality images to accurately identify the different cell development stages in phloem and xylem, which is particularly challenging in Mediterranean and tropical hardwoods. For the analyses, we need to identify the following:

(1) cambial cells and their division;

(2) postcambial cell development with the cell enlargement phase;

(3) secondary cell wall deposition and lignification; and

(4) cell maturation ending with autolysis of the protoplast and complete lignification of the cell walls.

Polarized light is commonly used to distinguish between primary and secondary cell walls and cellulose deposition (Figure 6a, b). It helps to distinguish between enlarging cells that contain only a primary cell wall and those that are in the phase of cell wall thickening with the appearance of a birefringent secondary wall.

Lignified and unlignified cell walls and tissues can be observed using epifluorescence techniques. Fluorescence imaging of sections stained with an aqueous solution of safranin and astra blue (or other combination of stains) can clearly highlight the contrast between lignified and unlignified cell walls to clearly show the boundary between postcambial cells with primary cell walls and those in the phase of secondary cell wall deposition, as well as the progression of lignin deposition in the areas of secondary wall thickening and maturation phases (Figure 6c). Fluorescence also highlight the presence of cytoplasm in the cell lumen, facilitating differentiation between mature and non-mature cells.

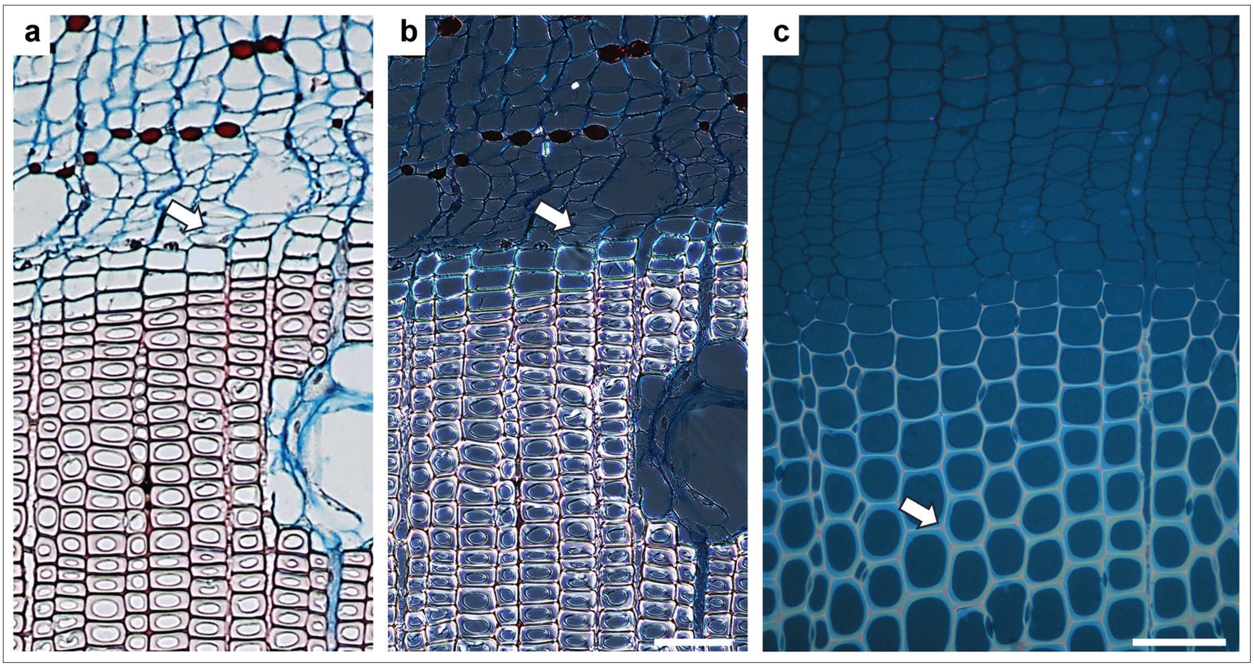


*Figure 6. Cross-section of phloem, cambium and xylem of Pinus halepensis: (a) under a light microscope, bright field mode, safranin-astra blue staining, (b) under polarized light allowing to distinguish between enlarging tracheids (white arrow) containing only a primary cell wall and tracheids in the phase of cell wall thickening, which contain a birefringent, shiny secondary wall, (c) progress of lignin deposition in the (shiny) areas of the middle lamella and secondary wall, starting from the middle lamella in the cell corners (white arrow). Scale bars = 500 µm.*

*Slika 6. Prečni prerez floema, kambija in ksilema alepskega bora (Pinus halepensis): (a) s svetlobnim mikroskopom, svetlo polje, obarvanje s safranin-astra modrim, (b) s polarizirano svetlobo, ki omogoča razlikovanje med traheidami v fazi rasti (bela puščica), ki vsebujejo le primarno celično steno, in traheidami v fazi odlaganja celične stene, ki vsebujejo optično aktivno sekundarno celično steno, ki se sveti, (c) potek odlaganja lignina (svetleča mesta) v srednji lameli in sekundarni steni, z začetkom v vogalih celic (bela puščica). Merilne daljice = 500 µm.*

## 4 CONCLUSIONS
## 4 ZAKLJUČKI

Monitoring xylogenesis and phloem formation provides useful information about xylem and phloem formation in real time, but requires specific tools, techniques, and skills for successful sampling, preparation of high-quality sections, and the ability to recognize the cells and the different stages of cell differentiation. The aforementioned skills are preferably acquired with the help of experts. If this is not possible, experience using existing protocols and technical references must be gained to master the critical steps. Many errors can occur at all stages of the process, from sampling to sectioning and quantification errors, especially if one is a novice in this field. This article is designed to share our experiences and help novices to not get discouraged and avoid the most common mistakes. First, we recommend acquiring knowledge of the species one intends to study. In our experience, protocol procedures should be adapted to it. Practicing on test samples can also help to gain the necessary experience without compromising the actual samples used in the research. It is hoped that the tips we have provided in these technical notes will help beginners to deal with some of the critical steps in the procedure that are not addressed in existing protocols.

## 5 SUMMARY

## 5 POVZETEK

Tehnika odvzema mikro izvrtkov in priprava preparatov za mikroskopijo se danes najpogosteje uporablja za spremljanje kambijeve aktivnosti ter nastajanja lesa in floema, med drugim tudi za oceno plastičnosti dreves v kontekstu podnebnih sprememb. Mikroizvrtke iz debel dreves jemljemo z orodjem Trephor (Rossi et al., 2006), ki je bilo prvič uporabljeno leta 2004. Razvitih je bilo več protokolov za vzorčenje in pripravo vzorcev mikroizvrtkov za mikroskopske analize kambija, ksilema in floema (na primer Deslauriers et al., 2015; Prislan et al., 2014a, b, 2022; De Micco et al., 2019; Pace, 2019; Balzano et al., 2021a). Namen tega prispevka je nadgraditi objavljene protokole, da bi začetnikom pomagali pri premagovanju kritičnih korakov med postopkom, ki v obstoječih protokolih niso nujno podrobno obravnavani. Predstavljamo najpogostejše napake in na podlagi naših izkušenj podajamo nasvete, kako se jim izogniti.

Najnovejši protokol Prislan et al. (2022) priporočamo kot referenco, kamor dodajamo naše nasvete. Pred začetkom novega projekta predlagamo, da skrbno preučite značilnosti drevesne vrste, ki jo želite preučiti (anatomija lesa in skorje, čas in trajanje rastne sezone ipd.). Po naših izkušnjah je treba protokol prilagoditi glede na značilnosti preiskovane vrste. Predlagamo tudi odvzem nekaj dodatnih vzorcev za preizkus metode. Da ne bi zamudili ključnih mejnikov (npr. začetek in konec kambijeve aktivnosti), je zaželen odvzem vzorcev preko celotnega koledarskega leta. Da bi se izognili učinku poškodovanj, moramo mikro izvrtke odvzemati spiralno vzdolž debla, pri čemer morajo biti mesta vzorčenja med seboj oddaljena vsaj 10 cm. Običajno se z vsakega drevesa na določen dan odvzameta dva mikro izvrtka, da imamo enega za rezervo. Pri majhnih drevesih ali grmih, kot je na primer vinska trta (slika 1), se prvi vzorec odvzame spodaj in ni priporočljivo odvzeti dveh mikroizvrtkov na vzorčenje, ker bi to povzročilo prevelike poškodbe. V tem primeru lahko za analizo izberemo več osebkov in jih vzorčimo izmenoma.

Rezilo Trephorja mora biti ostro. Topo rezilo bi povzročilo poškodbe na mikro izvrtku, kar bi posredno vplivalo tudi na kakovost tankih rezin (preparatov) (slike 1, 2, 3). Glede na vrsto, ki jo proučujemo, izberemo pravo velikost Trephorja. Večji Trephor (premer rezila > 2 mm) omogoča, da odvzamemo širši pas tkiva za analize, kar je pomembno zlasti pri preučevanju vrst s širšimi trakovi. Odstraniti moramo ustrezno količino zunanje skorje, da se izognemo tveganju, da bi mikro izvrtek vseboval predvsem skorjo in premalo drugih tkiv (lesa) (sliki 2a, c). Vbod s Trephorjem opravimo pravokotno na deblo na mestu, kjer je bil odstranjen zunanji del skorje (slika 3b). Pomembno je, da vzorce takoj po odvzemu vložimo v fiksacijsko sredstvo (FAA), da preprečimo poškodbe mehkejših tkiv (slika 3c).

Pred nadaljnjo obdelavo morajo biti mikro izvrtki pravilno orientirani, sicer bo tkivo na preparatih deorientirano, kar bo onemogočilo prepoznavanje in merjenje celic (slika 4a). Bistveno je tudi, da pri rezanju uporabimo popolnoma ostra rezila, da na preparatu ne bi prišlo do razpok, kolapsa, »izpuljenih« celic ter da preparat ne bi vseboval sledi rezila, ki jih vidimo kot pas raztrganih celic (slike 4b, c, d). Pri rezanju je pomembno, da nastavimo pravilen kot rezil (med 5° in 10°) in prilagodimo hitrost rezanja. Težave pri rezanju lahko pogosto rešimo že z manjšo spremembo debeline rezine.

Še en kritičen korak pri pripravi vzorca je nanos glicerin albumina na objektno steklo, ki »prilepi« preparat na steklo. Topila za zadnje korake pri dehidraciji tkiv je treba pogosto menjavati, da se izognemo nečistočam v preparatu (slika 4c). Pri dehidraciji uporabljeni alkohol ne sme vsebovati vode, sicer se vklopni medij zamegli (slika 4d). Poleg tega je treba vklopni medij Euparal primerno dozirati, da se izognemo nastanku madežev na objektnem ali pokrivnem steklu (slika 4e) in da se v preparat ne »ujamejo« zračni mehurčki. Z upoštevanjem protokola (Prislan et al., 2022) in tukaj predlaganih nasvetov bi morali nastati visokokakovostni preparati in slike, primerne za kvantitativno analizo slike (slika 5).

Pri analizah nastajanja lesa in skorje moramo prepoznati različne vrste celic in tkiv v lesu in skorji (floemu) ter njihovo razvojno stopnjo. Za razlikovanje med primarnimi in sekundarnimi celičnimi stenami običajno uporabljamo mikroskopijo s polarizirano svetlobo (slika6 a, b). Z epi-fluorescenčno mikroskopijo pa lahko poudarimo kontrast med lignificiranimi in nelignificiranimi celičnimi stenami, da prepoznamo potek odlaganja lignina na območjih srednje lamele in sekundarne stene ter nastanek popolnoma diferenciranih zrelih celic (slika 6c). Upamo, da bodo ob upoštevanju predstavljenih na-vodil tudi začetniki lahko premostili kritična mesta v postopku in dosegli želene rezultate.

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# DENDROKRONOLOGIJA IN ABSOLUTNO DATIRANJE KOLIŠČ NA LJUBLJANSKEM BARJU

# DENDROCHRONOLOGY AND ABSOLUTE DATING OF PILE-DWELLINGS IN LJUBLJANSKO BARJE

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## Izvleček / Abstract

**Izvleček:** Na 16 koliščih na Ljubljanskem barju v Sloveniji je bilo med leti 1995 in 2021 z arheološkimi izkopavanji zbranih več kot 8.800 vzorcev z vodo napojenega arheološkega lesa. Večina vzorcev je bilo odvzetih iz pilotov, zabitih v zemljo, na katerih so bila zgrajena bivališča. Približno 20 % vzorcev je bilo iz lesa hrasta (*Quercus* sp.) in jesena (*Fraxinus* sp.), z več kot 45 branikami, ki jih je bilo mogoče vključiti v dendrokronološke analize in sestaviti kronologije širin branik za večino najdišč. Datiranje z uporabo dendrokronologije, radiokarbonskega datiranja in metode wiggle matching ter telekonekcije z nemško-švicarsko referenčno kronologijo so omogočili na leto natančno absolutno datiranje hrasta v časovnem okviru 3771–3330 pr. Kr. (kronologija BAR-3330), medtem ko so bili natančni radiokarbonski datumi pridobljeni za kronologije, ki pokrivajo obdobji 3285–3109 ± 14 kal. pr. Kr. (SG-VO) in 2659–2417 ± 18 kal. pr. Kr. (ZA-QUSP1). Potencial kronologij jesena, zlasti tistih iz 3. tisočletja pr. Kr., kjer ta vrsta prevladuje, še ni bil v celoti izkoriščen.

**Ključne besede:** koliščarske naselbine, Ljubljansko barje, neolitik, eneolitik, arheološki les, dendrokronologija, C14 wiggle-matching, absolutno datiranje

**Abstract:** Between 1995 and 2021, archaeological excavations at 16 sites in Ljubljansko barje, Slovenia, collected more than 8,800 samples of waterlogged archaeological wood, mostly from piles driven into the ground on which dwellings were built. About 20% of the samples were from oak (*Quercus* sp.) and ash (*Fraxinus* sp.) trees with more than 45 tree-rings, which could be included in the dendrochronological analyses, and tree-ring chronologies could be established for most sites. Dating by dendrochronology, radiocarbon dating, and wiggle matching, as well as teleconnection with the German-Swiss reference chronology, allowed absolute dating of oak in the time frame 3771–3330 BC (BAR-3330 chronology), while precise 14C dates were obtained for chronologies covering the periods 3285–3109 ± 14 cal BC (SG-VO) and 2659–2417 ± 18 cal BC (ZA-QUSP1). The potential of the ash wood chronologies, especially those of the 3rd millennium BC, when this wood species was predominant, has not yet been fully exploited.

**Keywords:** pile dwellings, Ljubljansko barje, Neolithic, Eneolithic, archaeological wood, dendrochronology, radiocarbon wiggle-matching, absolute dating

## 1 UVOD
## 1 INTRODUCTION

Ljubljansko barje je edino območje z ohranjenimi arheološkimi ostanki prazgodovinskih koliščarskih bivališč, odkritih v Sloveniji. Predstavlja plitvo mokrišče v bazenu tektonskega izvora s površino 163 kvadratnih kilometrov, ki je občasno lahko poplavljeno zaradi taljenja snega ali obilnih padavin. Od poznega pleistocena do zgodnjega poznega holocena je območje prekrivalo plitvo jezero.

Prva kolišča je leta 1875 odkril Karl Deschmann v bližini vasi Studenec, zdaj Ig (Leghissa, 2021). Po

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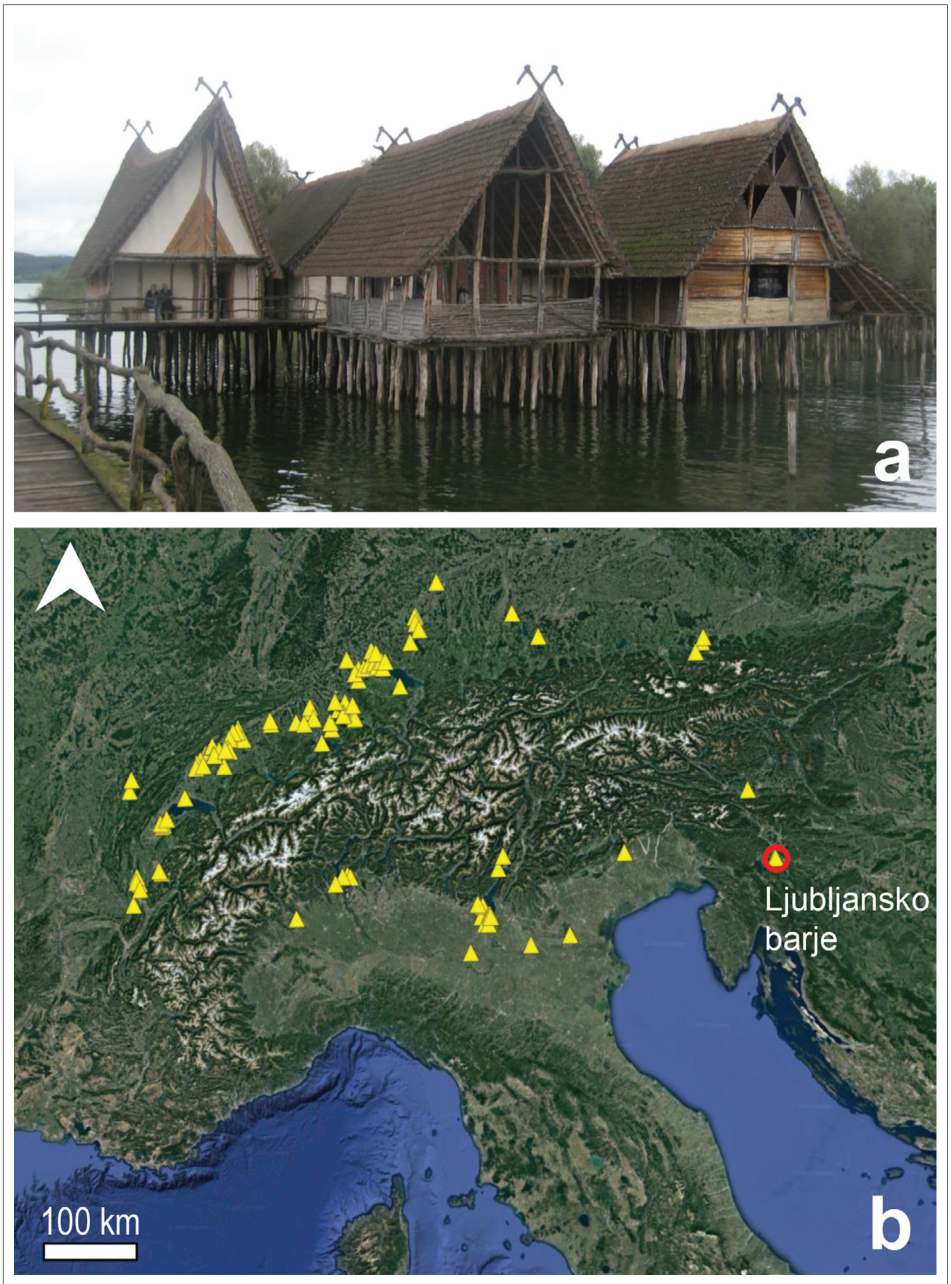
odkritju in pionirskih izkopavanjih v letih 1875–1877 je bilo izvedenih več raziskovalnih akcij, npr. 1907–1908 (W. Schmid), 1953–1989 (J. Korošec, T. Bregant) in več akcij po letu 1992 (Velušček, 2019). Leta 2011 je Unesco pomen dveh različnih skupin kolišč iz okolice Iga prepoznal z vpisom na seznam svetovne dediščine (slika 1b) (Unesco, 2022).

Inštitut za arheologijo Znanstvenoraziskovalnega centra Slovenske akademije znanosti in umetnosti (IA ZRC SAZU) je koordiniral serijo raziskav, ki so se začele leta 1995 in še vedno potekajo. V tej kampanji so bile uvedene sodobne metode vzorčenja, ki so jih razvili pri raziskavah koliščarskih naselbin na območju Alp. Tu imajo poseben pomen raziskave arheološkega lesa in uvajanje dendrokronologije, ki jo je bilo v Sloveniji potrebno vpeljati za raziskave v arheologiji (npr. Čufar et al., 1999). Interdisciplinarne raziskave so vključevale tudi razvoj palinologije, arheobotanike in arheozoologije ter sodelovanje z drugimi področji, kot so arheometrija, tekstilno inženirstvo, metalurgija, geologija in medicina (npr. Velušček 2004a, 2009).




*Slika 1. Koliščarske naselbine: (a) replika kolišča iz 2. tisočletja pr. Kr. v koliščarskem muzeju Unteruhldingen ob Bodenskem jezeru v Nemčiji in (b) lokacije 111 prazgodovinskih koliščarskih najdišč v Alpah iz obdobja od leta 5000 do 500 pr. Kr. na Unescovem seznamu svetovne dediščine. Ljubljansko barje ima najbolj jugovzhodno lego. Zemljevid: Nina Škrk, Google Earth, 2020.*

*Figure 1. Lake dwellings: (a) replica of a 2nd millennium BC pile dwelling at the Pile Dwelling Museum in Unteruhldingen on Lake Constance in Germany and (b) 111 prehistoric pile dwellings in the Alps dating from 5,000 to 500 BC that are on the World Heritage List UNESCO. Ljubljansko barje has the most southeastern location. Map: Nina Škrk, Google Earth, 2020.*


*Slika 2. Terensko delo in zbiranje arheološkega materiala na koliščarskih naselbinah na Ljubljanskem barju: (a) sonda, (b) drenažni jarek, (3) dno reke z vidnimi ostanki lesenih pilotov.*
*Figure 2. Archaeological field work on pile-dwelling settlements to collect arcaheological material and wood in Ljubljansko barje: (a) excavation probe, (b) drainage ditch, (3) riverbed with visible remains of wooden piles.*

Dendrokronološke raziskave, brez katerih si danes ne moremo zamišljati sodobnih arheoloških raziskav lesa v vlažnih okoljih, so opravili na Oddelku za lesarstvo Biotehniške fakultete Univerze v Ljubljani (OL BF) (npr. Čufar & Velušček, 2012; Čufar et al., 2013; Velušček & Čufar, 2014). Od ustanovitve dendrokronološkega laboratorija leta 1993 so se za ta namen posvetili sestavljanju referenčnih kronologij in proučevanju dendrokronološkega potenciala hrasta in drugih lesnih vrst v Sloveniji (npr. Čufar & Levanič, 1999; Čufar et al., 2008a), ter tudi temeljnim raziskavam na področju dendroekologije hrasta južno od Alp (npr. Čufar et al., 2014), dendroklimatologije (npr. Čufar et al., 2008b) ter lastnosti in možnosti konserviranja z vodo napojenega arheološkega lesa (npr. Čufar et al., 2002; Čufar et al., 2008c; Balzano et al., 2022), kar je vse prispevalo k celoviti obravnavi lesa in uporabi dendrokronologije v arheologiji.

Po 27 letih dendrokronoloških raziskav na koliščih Ljubljanskega barja je bilo doseženih nekaj pomembnih uspehov, ki še niso bili sistematično predstavljeni domači javnosti. Ob velikem raziskovalnem potencialu tega materiala ostaja tudi več odprtih izzivov. Vse to predstavljamo v tem prispevku.

## 2 MATERIAL IN METODE
## 2 MATERIALS AND METHODS

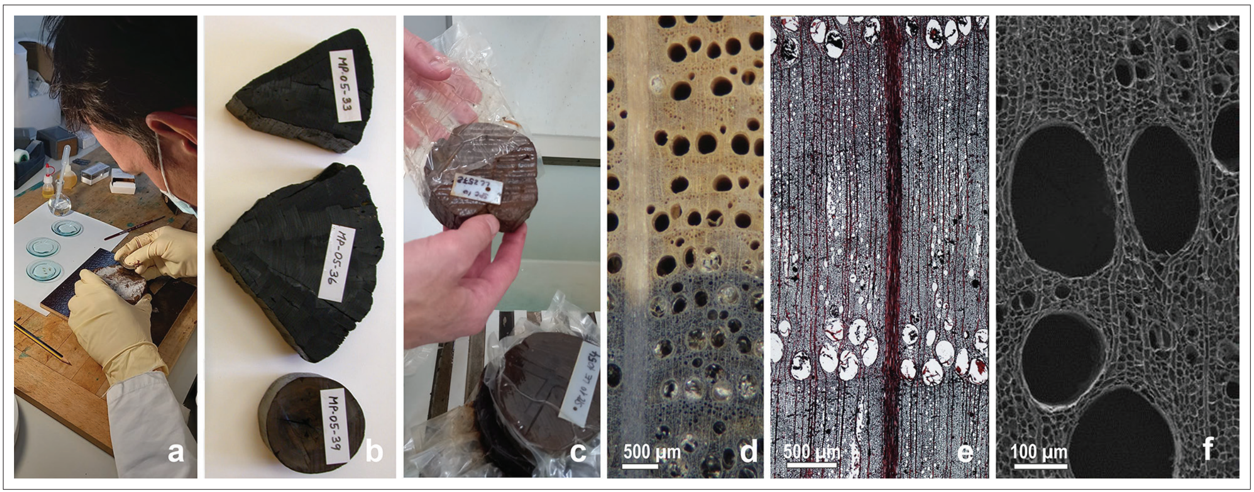
### 2.1 ARHEOLOŠKE RAZISKAVE
### 2.1 ARCHAEOLOGICAL RESEARCH

Med leti 1995 in 2021 je bilo na 16 koliščih na Ljubljanskem barju opravljenih 29 izkopavanj in terenskih pregledov, kjer so pridobili les in druge arheološke najdbe (preglednica 1). Vse raziskave, razen na koliščih Črnelnik, Špica in Veliki Otavnik Ib, je vodil Inštitut za arheologijo ZRC SAZU. Na zbranem arheološkem materialu so bile opravljene sistematične interdisciplinarne raziskave (npr. Velušček, 2004a, 2006, 2009, 2020).

Da bi za dendrokronološke analize pridobili zadostno količino lesa, je zbiranje ostankov lesa in drugih najdb potekalo na predhodno izkopanih območjih, v rečnih strugah, drenažnih jarkih in z manjšimi izkopavanji ali vkopi na različnih lokacijah (slika 2). Območje nekaterih kolišč so izkopavali v več etapah (preglednica 1).

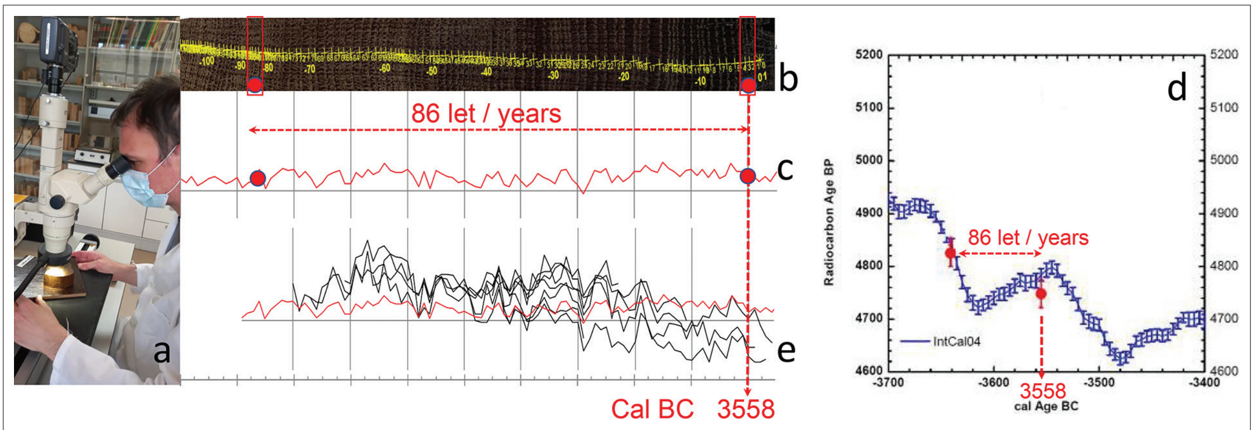
### 2.2 ANALIZE LESA–IDENTIFIKACIJA, DENDROKRONOLOGIJA, RADIOKARBONSKO DATIRANJE
### 2.2 WOOD ANALYSES–IDENTIFICATION, DENDROCHRONOLOGY, RADIOCARBON DATING

Med leti 1995 in 2021 je bilo zbranih 8829 vzorcev z vodo napojenega arheološkega lesa, večinoma iz pilotov kolišč (preglednica 1). Na Oddelku za lesarstvo smo les obdelali, tako da smo pripravili približno 10 cm debele odrezke s prečnim prerezom. Tako pripravljene vzorce smo globoko zamr-




*Slika 3. Delo z arheološkim lesom s koliščarskih naselbin: (a) obdelava površine globoko zamrznjenega vzorca – izdelava preparata za identifikacijo lesa, (b) vzorci pilotov iz celih in cepljenih debel z obdelanimi prečnimi prerezi, (c) priprava arheološkega lesa za skladiščenje v napojenem stanju in vakuumu v zavarjenih polietilenskih vrečkah. Prečni prerez lesa hrasta (Quercus sp.) pod: (d) stereo lupo, (e) svetlobnim mikroskopom in (f) elektronskim vrstičnim mikroskopom. (Foto: Andraž Benedik a-e, Angela Balzano f).*

*Figure 3. Working with archaeological wood from pile-dwelling sites: (a) surface smoothing of a frozen sample – slide preparation for wood identification, (b) pile samples of whole and split logs with smoothed cross-sections, (c) preparation of archaeological wood for storage in water-saturated state under vacuum in polyethylene bags. Cross-section of oak wood (Quercus sp.) under (d) stereomicroscope, (e) light microscope, and (f) scanning electron microscope (photos: Andraž Benedik a-e, Angela Balzano f).*




*Slika 4. Postopek datiranja: (a) merjenje širin branik, les je na merilni mizici pod stereo lupo, (b) oznake širin branik na posnetku lesa, (c) zaporedje širin branik enega kosa lesa z označenimi mesti odvzema dveh vzorcev za radiokarbonsko in wiggle-matching analizo – razlika v starosti je 86 let, (d) kalibriranje radiokarbonskih datumov z metodo wiggle matching in rezultat datacije mlajše branike, (e) zaporedje "c" (rdeča črta) in zapredja širin branik drugih vzorcev iz kolišča (črne črte) v sinhronem položaju, vsi vzorci so datirani.*

*Figure 4. Dating process: (a) measurement of tree-ring widths of wood on a measuring table under a stereomicroscope, (b) tree-ring widths on the image, (c) recording of the tree-ring series of a sample with the sampling points marked for radiocarbon and wiggle matching analysis–the age difference between two samples is 86 years, (d) the calibration of radiocarbon dates by the wiggle matching method and the result of dating of the last ring, (e) the series of 'c' (red curve) and other samples from the site (black lines) in cross-dated position – all of them are dated.*

znili pri temperaturi -22 °C in nato na zamrznjenih vzorcih zgladili prečno površino za pregled pod stereo mikroskopom in makroskopsko identifikacijo lesa (slika 3a, b, c, d). V kolikor makroskopska identifikacija lesa ni bila mogoča, smo pripravili tanke preparate za mikroskopsko identifikacijo lesa (slika 3e) in jih identificirali ob uporabi standardnih ključev za identifikacijo lesa (npr. Schoch et al., 2004; Čufar, 2006; Čufar & Merela, 2014).

Na vseh vzorcih lesa smo najprej prešteli branike in izmerili oz. ocenili premer debla. Dendrokronološko merjenje širin branik smo opravili na vzorcih hrasta, jesena, bukve in jelke, ki so imeli 45 ali več branik. Sledilo je sinhroniziranje zaporedij širin branik posameznih vzorcev in združevanje v kronologije za posamezno lesno vrsto in koliščarsko naselbino.

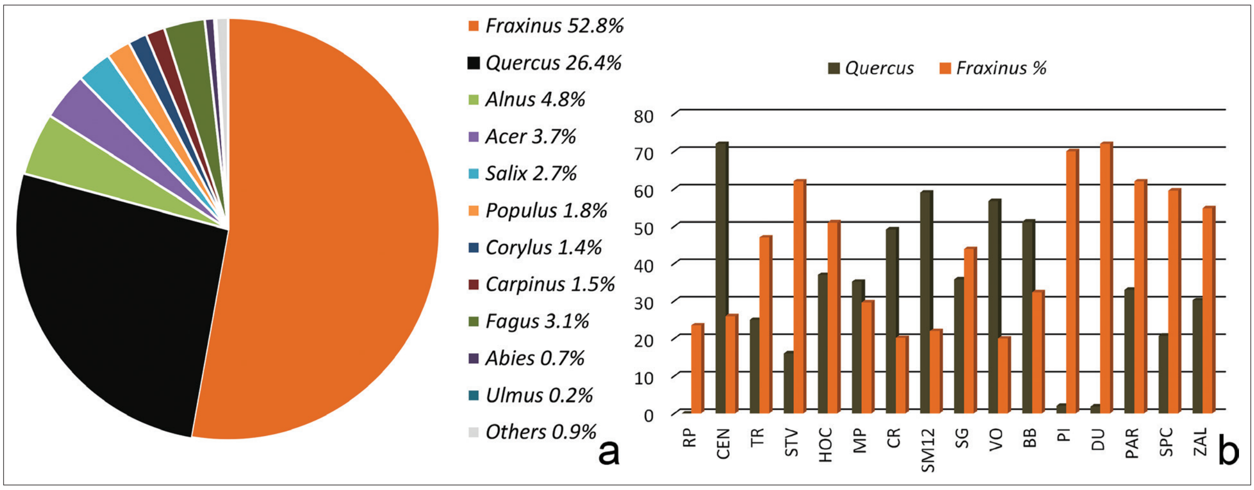
Za vsako kronologijo smo iz enega ali več reprezentativnih vzorcev odvzeli les za radiokarbonsko datiranje (Čufar & Kromer, 2004). Ob naraščanju števila radiokarbonskih datumov in dendrokronološki določitvi razlike v številu branik oz. let med radiokarbonsko datiranimi vzorci je bila možna uporaba metode wiggle-matching in bolj natančna umestitev kronologij v čas, v najboljšem primeru ± 10 let (Čufar et al., 2010).

Absolutno dendrokronološko datiranje nam je po 20 letih raziskav uspelo z dendrokronološko telekonekcijo z nemško-švicarsko referenčno kronologijo; tako smo na leto natančno datirali več kronologij iz sredine 4. tisočletja pr. Kr. (Čufar et al., 2015).

## 3 REZULTATI IN RAZPRAVA
## 3 RESULTS AND DISCUSSION

Med 8829 vzorci arheološkega lesa so bile identificirane vrste lesa veliki jesen (*Fraxinus excelsior*), hrast (*Quercus robur* in *Quercus petraea*), črna jelša (*Alnus glutinosa*), javor (*Acer* sp.), vrba (*Salix* sp.), topol (*Populus* sp.), leska (*Corylus avellana*), beli gaber (*Carpinus betulus*), bukev (*Fagus sylvatica*), jelka (*Abies alba*), brest (*Ulmus* sp.) in posamezni predstavniki drugih vrst (slika 5a) (prim. Čufar & Velušček, 2012).

Nabor in odstotni deleži uporabljenih lesnih vrst so se med kolišči zelo razlikovali (preglednica 1, slika 5b). Jesen je skupno predstavljal dobro polovico vzorcev, hrast dobro četrtino. Hrasta je najmanj na koliščih Resnikov prekop (0 %), Parte-Iščica (2 %) in Dušanovo (2 %), na ostalih koliščih je bil delež hrasta 16–72 %. Odstotni deleži jesena so znašali 20–72 % in so bili največji tam, kjer je bilo hrasta




*Slika 5. Izbor lesa na koliščarskih naselbinah: (a) deleži lesnih vrst (rodov), raziskanih med leti 1995-2021 in (b) variiranje deležev vzorcev hrasta (Quercus) in jesena (Fraxinus) na koliščarskih naselbinah (za pomen kratic in obdobja kolišč glejte preglednico 1).*

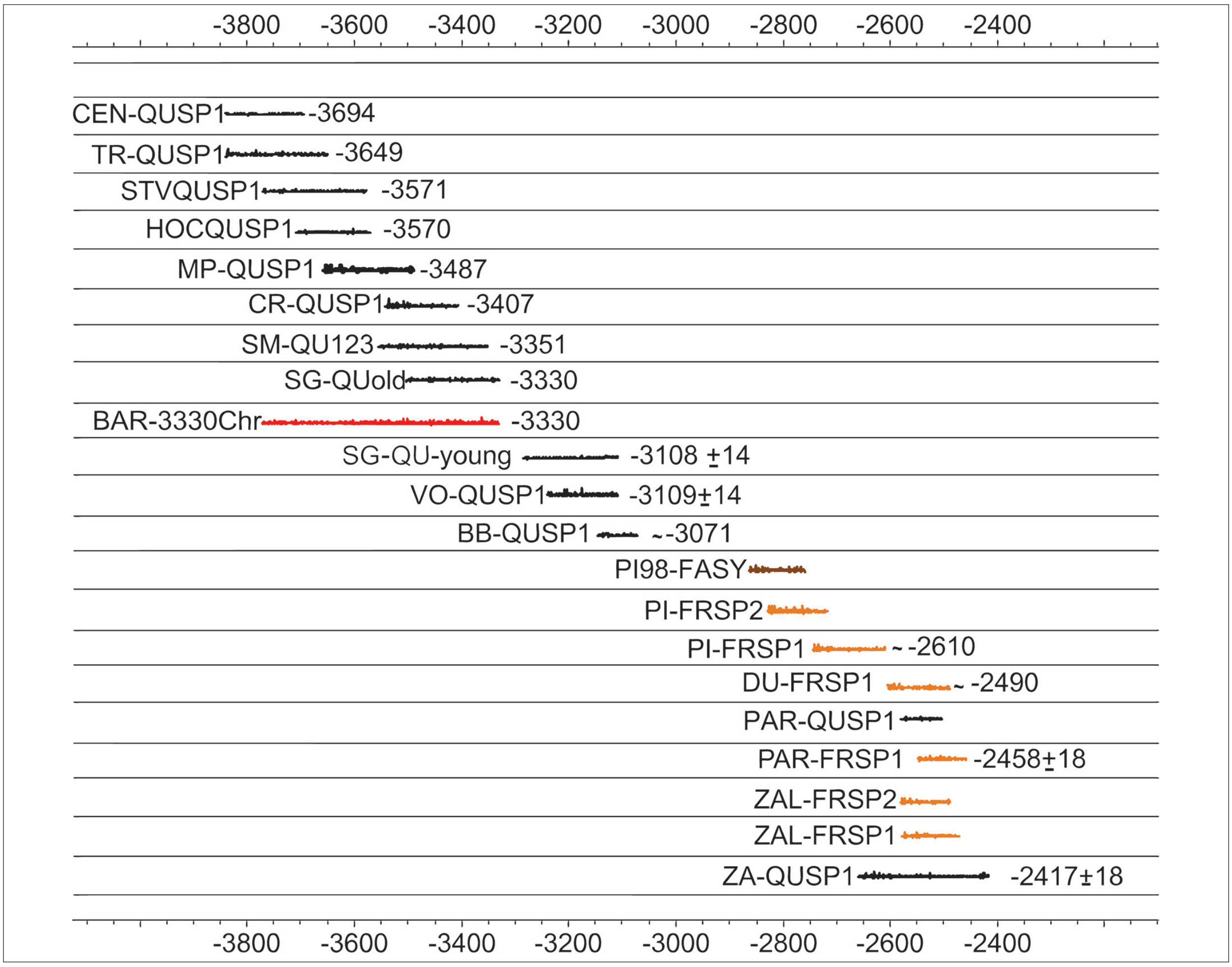
*Figure 5. Selection of wood in the pile-dwelling settlements: (a) proportions of wood species (genera) analysed between 1995-2021 and (b) variation in the proportions of oak (Quercus) and ash (Fraxinus) samples from the oldest to the youngest pile dwelling (for the meaning of the abbreviations and the time of the dwellings, see Table 1).*

*Preglednica 1. Koliščarske naselbine na Ljubljanskem barju: leta izkopavanj, število zbranih vzorcev lesa, odstotni delež vzorcev hrasta (Quercus) in jesena (Fraxinus) ter končni datumi kronologij posameznih kolišč, datiranih s pomočjo dendrokronologije (Dendro), radiokarbonskih analiz (14C) ali metode wiggle-matching (Wiggle).*
*Table 1. Pile-dwelling settlements in Ljubljansko barje, with years of excavation, number of wood samples collected, percentages of oak (Quercus) and ash (Fraxinus), and end dates of the tree-ring chronologies based on dendrochronology (Dendro), radiocarbon (14C) or radiocarbon wiggle-matching (Wiggle).*

| Št. / No. | Kolišče / Site | Koda / Code | Leta izkopavanj / Years of excavations | Število vzorcev / No. of Wood Samples | Quercus % / Quercus % | Fraxinus % / Fraxinus % | Zadnji datum pr.Kr. / End Date BC | Metoda / Dating Method |
|---|---|---|---|---|---|---|---|---|
| 1 | Resnikov prekop | RP | 2002 | 34 | 0 | 24 | ~4600 | 14C |
| 2 | Črnelnik | CEN | 2014 | 39 | 72 | 26 | 3694 | Dendro |
| 3 | Trebež | TR | 2017 | 83 | 25 | 47 | 3649 | Dendro |
| 4 | Strojanova voda | STV, SV | 2012 | 351 | 16 | 62 | 3578 | Dendro |
| 5 | Hočevarica | HOC, HO | 1995, 1998 | 361 | 37 | 51 | 3570 | Dendro |
| 6 | Maharski prekop | MP | 2005 | 234 | 35 | 30 | 3487 | Dendro |
| 7 | Črešnja pri Bistri | CR | 2003 | 124 | 49 | 20 | 3407 | Dendro |
| 8 | Spodnje mostišče | SM1+2 | 1996, 1997 | 690 | 59 | 22 | 3351 | Dendro |
| 9 | Stare gmajne | SG | 2002, 2004, 2006, 2007, 2021 | 984 | 36 | 44 | 3109±14 | Wiggle |
| 10 | Veliki Otavnik Ib | VO | 2006 | 30 | 57 | 20 | 3108±14 | Wiggle |
| 11 | Blatna Brezovica | BB | 2003 | 170 | 51 | 32 | ~3071 | 14C |
| 12 | Parte-Iščica | PI | 1997, 1998 | 1265 | 2 | 70 | ~2610 | 14C |
| 13 | Dušanovo (in Črn graben) | DU, CG | 2010, 2013, 2017 | 305 | 2 | 72 | ~2490 | 14C |
| 14 | Parte | PAR | 1996 | 242 | 33 | 62 | 2458±18 | Wiggle |
| 15 | Špica | SPC | 2009, 2010 | 2452 | 21 | 60 | ~2450 | |
| 16 | Založnica | ZAL, ZA | 1995, 1999, 2001, 2009 | 1465 | 30 | 55 | 2417±18 | Wiggle |

malo. Na vseh koliščih so uporabljali predvsem les listavcev, ki na Ljubljanskem barju in v okoliškem hribovju uspevajo tudi danes. Od iglavcev so občasno uporabili samo jelko in v redkih posebnih primerih tiso (na sliki 5a med ostalimi vrstami), ki smo ju tako kot bukev in brest našli samo na posameznih koliščih (Čufar & Velušček, 2012). Izbor lesnih vrst je bil praviloma odvisen od lege kolišča, če se je to nahajalo bliže jezeru oz. poplavni ravnici ali bliže kraškim pobočjem na jugu Ljubljanskega barja (Tolar et al., 2011). Na izbor je vplivala tudi razpoložljivost lesa, ki so ga, če je bilo mogoče, posekali v bližini kolišč. Največ dreves za pilote je imelo premer 7–10 cm (Čufar et al., 1999; Out et al., 2020). Če je bil premer dreves večji od 14 cm, so debla po dolgem razcepili na 4 ali več delov (slika 3b), tako da so pridobljene pilote lahko zabili v zemljo oz. jezersko dno. Čeprav številni viri poročajo, da so prebivalci kolišč zaradi velikih potreb po lesu gospodarili z gozdovi (npr. Bleicher & Staub, 2022), tega do sedaj za Ljubljansko barje še nismo mogli dokazati, kar kaže tudi raziskava, v katero je bila vključena naselbina Stare gmajne (Out et al., 2020).

Le manjši delež (približno 20 %) vzorcev hrasta in jesena je imel 45 ali več branik, kar je bil naš kriterij, da smo jih vključili v dendrokronološke raziskave. V prvi fazi raziskav smo se osredotočili predvsem na hrast, ki je najpogostejši in najpomembnejši les




*Slika 6. Časovni razponi glavnih kronologij hrasta (QUSP), jesena (FRSP) in bukve (PI98- FAG) ter končni datumi koliščarskih naselbin (polna imena v preglednici 1). Kronologije, ki se končajo z letom 3330 pr. Kr. ali prej, so datirane z dendrokronološko telekonekcijo in so vključene v kronologijo BAR-3330 (3840-3330 pr. Kr.). Kronologije SG-QU-young, VO-QUSP1 in ZAL-QUSP1 temeljijo na radiokarbonskem datiranju, podprtim z metodo wiggle matching. Za ostale kronologije so končni datumi manj natančni in so večinoma ocenjeni na podlagi enega samega radiokarbonskega datuma.*

*Figure 6. Time spans of the main chronologies of oak (QUSP), ash (FRSP), and beech (PI98- FAG) and end dates of pile dwellings (see Table 1 for complete names). Chronologies dated to 3330 BC or earlier are dated by dendrochronology (teleconnection) and are included in the chronology BAR-3330 (time span 3840-3330 BC). Chronologies SG-QU-young, VO-QUSP1, and ZAL-QUSP1 are dated by 14C and wiggle matching. For the others, end dates are estimated primarily on the basis of a single radiocarbon date.*

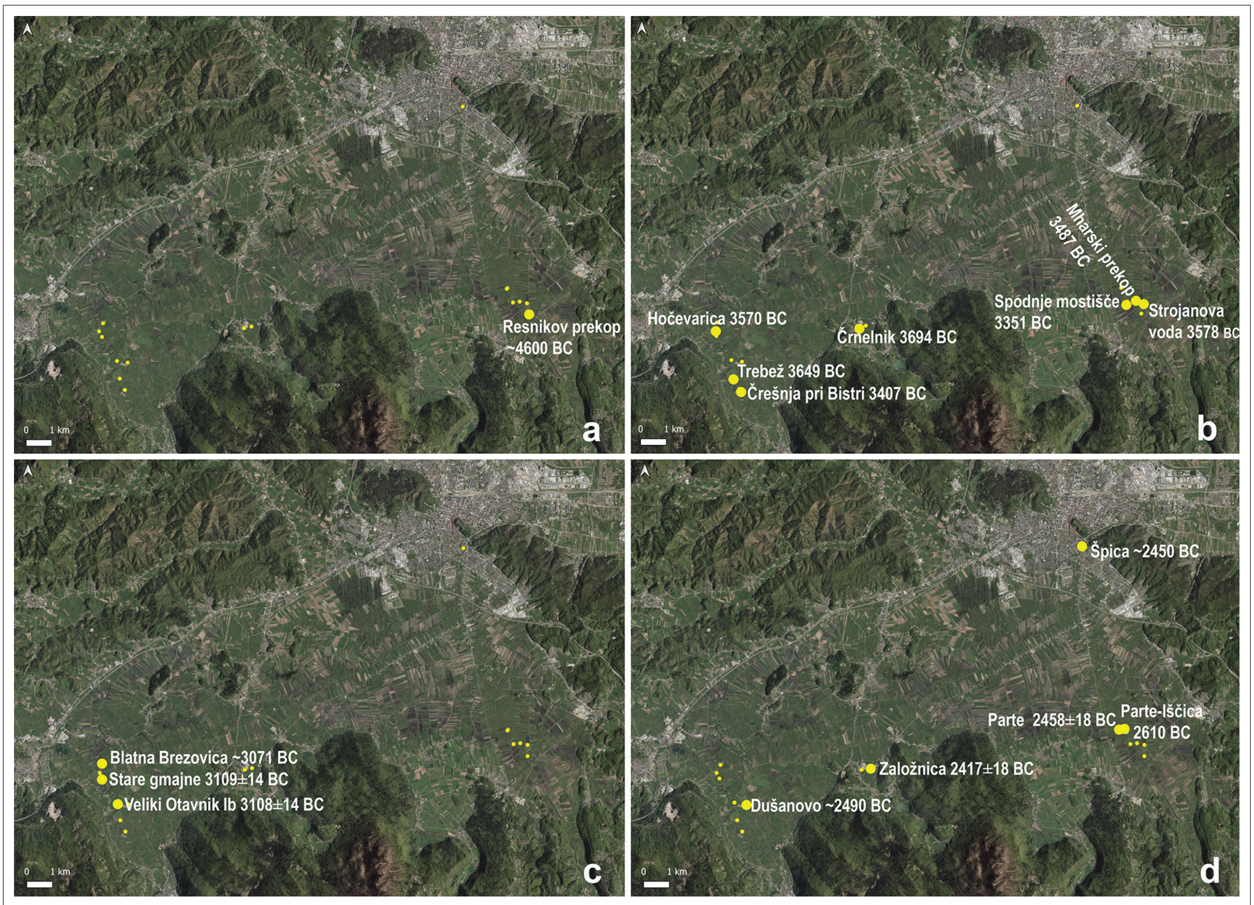
v evropski arheologiji in za katerega obstaja mreža dolgih referenčnih kronologij (npr., Baillie, 1995; Haneca et al., 2009; Tegel et al., 2022), ki v nekaterih primerih izkazujejo podobnost (telekonekcijo), tudi če izvirajo iz oddaljenih regij (npr., Čufar & Martinelli, 2004; Čufar et al., 2008a, 2014; Kolář et al., 2012; Wazny et al., 2014).

Za skupino kolišč iz sredine 4. tisočletja pr. Kr. (Strojanova voda, Hočevarica, Maharski prekop, Črešnja pri Bistri, Spodnje mostišče in starejši del najdišča Stare gmajne) smo sestavili hrastove kronologije, ki so se delno medsebojno prekrivale, tako da smo jih lahko združili v 442-let dolgo kronologijo BAR-3330 (slika 6). To kronologijo smo najprej datirali s pomočjo metode wiggle-matching z natačnostjo ± 10 let (zadnje leto 3332 ± 10 kal. pr. Kr.) (Čufar et al., 2010), v letih za tem, pa je sledilo dendrokronološko datiranje s pomočjo telekonekcije z nemško-švicarsko kronologijo, tako da smo BAR-3330 določili časovni razpon 3771-3330 pr. Kr. (Čufar et al., 2015). Kronologija je v času objave temeljila na 106 sinhroniziranih serijah širin branik lesa iz šestih kolišč, po tem smo raziskali še kolišči Črnelnik in Trebež ter kronologijo podaljšali na 511 let (razpon 3840 – 3330 pr. Kr.) (slika 6).

V tem prispevku (preglednica 1, sliki 6 in 7) za kolišča vedno navajamo zadnji datum kronologije, ki običajno sovpada z zaključkom poseka dreves in s tem gradbenih aktivnosti na kolišču, ki smo jih lahko ugotovili s pomočjo raziskanega lesa. Barje je bilo v različnih obdobjih različno intenzivno poseljeno. V grobem lahko obstoj kolišč delimo v štiri časovne skupine (slika 7).

Kronologije s končnimi datumi, mlajšimi od 3330 pr. Kr., so bile datirane samo z radiokarbonskim ujemanjem (wiggle-matching), njihovo dendrokronološko datiranje še ni bilo uspešno. Takšni sta kronologiji Stare gmajne (mlajši del) in Veliki Otavnik Ib–njuna skupna kronologija SG-VO pokriva obdobje 3285-3108 ± 14 kal. pr. Kr.

Za koliščarske naselbine iz 3. tisočletja pr. Kr. na Ljubljanskem barju smo sestavili predvsem jesenove (FRSP) kronologije (preglednica 1, slika 6), saj je bil hrast v tem času redko uporabljen. Edina daljša in dobro pokrita hrastova kronologija za obdobje 2659-2417 ± 18 kal. pr. Kr. je ZAL-QUSP1 iz Založnice.




*Slika 7. Ljubljansko barje in mesta raziskanih koliščarskih naselbin: (a) iz 5. tisočletja, (b) iz sredine 4. tisočletja, (c) iz konca 4. tisočletja ter (d) iz 3. tisočletja pr. Kr. Zemljevidi: Nina Škrk, GURS (2021).*
*Figure 7. Map of Ljubljansko barje and the locations of the investigated pile dwellings: (a) from the 5th, (b) from the middle of the 4th, (c) from the end of the 4th, and (d) from the 3rd millennium BC. Maps: Nina Škrk, GURS (2021).*

Ob začetku raziskav je bilo datiranje jesenovih kronologij malo verjetno, danes pa bi sodobne radiokarbonske metode omogočile njihovo datiranje (Capano et al., 2020), vendar ta možnost še ni bila izkoriščena. Dendrokronološko datiranje jesenovih kronologij s hrastovimi referencami s severa Alp ni verjetno, obstaja pa možnost njihovega datiranja s pomočjo hrastovih kronologij iz istega območja (heterokonekcija). Jesenove kronologije so večinoma kratke (slika 6), njihovo variiranje širin branik pa odraža odziv na lokalne razmere in motnje, kot je obvejevanje dreves za prehrano živali. Datacije vseh jesenovih kronologij na Ljubljanskem barju bi zato morali izboljšati. To bi pripomoglo k boljšemu poznavanju poselitev kolišč Dušanovo, Parte in Založnica, ki označujejo konec bakrene dobe na Ljubljanskem barju (Velušček & Čufar, 2003; Velušček et al., 2011). Med mlajša kolišča uvrščamo tudi kolišče Špica, kjer smo pridobili kar 2452 vzorcev, a doslej nismo mogli sestaviti kakovostnih kronologij in podati prepričljivega datiranja lesa.

Prikazani končni datumi kronologij označujejo zadnji (datirani) posek na posameznem kolišču oz. najdišču (preglednica 1, sliki 6 in 7), sicer smo na podlagi datumov sečnje posameznih dreves na večini kolišč lahko določili eno ali več gradbenih faz. Na gradbene dejavnosti in popravila bivališč je bilo mogoče sklepati na podlagi velikega števila dreves, posekanih v istem letu ali v ozkem obdobju nekaj let. Nekatere koliščarske naselbine so bile poseljene več desetletij, medtem ko so druge delovale le 20 let ali manj (npr. Velušček, 2005).

Raziskana koliščarska naselja se (z izjemo najdišča Špica) nahajajo na južnem delu Ljubljanskega barja in tvorijo dve skupini, ki sta med seboj oddaljeni 10-15 km (slika 7). Skoraj v vseh preučevanih obdobjih smo lahko zaznali sočasne gradbene dejavnosti na več koliščih, na primer Strojanova voda–Hočevarica, Spodnje mostišče–Stare gmajne (starejši del) in Parte – Založnica–Dušanovo. V naselbinah, kjer je bila sočasnost dokazana z dendrokronologijo, to potrjujejo tudi skoraj identične arheološke najdbe, kot je na primer keramika, orodje in orožje.

V obdobju, 4600 do 2400 pr. Kr., ko so se na Ljubljanskem barju pojavljale koliščarske naselbine, je bila poselitev večkrat prekinjena. Prekinitve smo zabeležili med približno 4600 in 3700 pr. Kr., med 3300 in 3150 pr. Kr., ter po letu 3100 pr. Kr. Razlogov za to še ne znamo v celoti pojasniti, verjetno so bile prekinitve poselitve posledica gospodarskih in podnebnih dejavnikov (Velušček, 2004b).

Dendrokronološke ugotovitve se dopolnjujejo z ugotovitvami o materialni kulturi, kjer je bilo na podlagi keramike mogoče razmejiti več kulturnih horizontov oziroma poselitvenih obdobij.

## 4 ZAKLJUČKI
## 4 CONCLUSIONS

Arheološka izkopavanja in raziskave lesa iz koliščarskih naselbin so omogočila datiranje kolišč. Na leto natančno smo lahko ugotovili gradbene dejavnosti (posek lesa) in ocenili trajanje naselitev. Umestitev v čas je omogočila vpogled o domnevnem stanju okolja in gozdnih virov. Izbor in raba lesa nam povesta veliko tudi o življenju, razmišljanju in tehničnem znanju koliščarjev in njihovi rabi okolja (Tolar et al., 2011; Out et al., 2020). Izbira, obdelava in raba lesa so pokazali, da so imeli koliščarji bogato znanje o lesnih vrstah in lastnostih lesa. Znali so na najboljši možni način izbrati, obdelati in uporabiti les iz neposredne okolice naselij ali iz bolj oddaljenih gozdov (Tolar et al., 2011). V drugi polovici 4. tisočletja pr. Kr. so si koliščarji znali izdelati voz, katerega ohranjeni leseni ostanki z najdišča Stare gmajne predstavljajo najstarejše znano kolo z osjo na svetu, ki je mojstrovina prazgodovinske obrti (Velušček et al., 2009). Poleg znanja o lesu so za to potrebovali tudi znanje o materialih, saj so si iz razpoložljivih virov (kamen, les, roženina, baker) morali sami izdelati orodje in ga vzdrževati.

Raziskave lesa so bile le manjši, a bistveni del multidisciplinarnih raziskav, ki so omogočile rekonstruirati celostno sliko človekovega življenja, okolja in povezav z drugimi najdišči. V zadnjih 27 letih raziskav smo preučevali najdišča iz različnih obdobij in z različnimi kulturnimi ozadji. Analize peloda in makrobotaničnih ostankov so pokazale, da so bili najzgodnejši prebivalci kolišč lovci, ribiči in nabiralci, tudi poljedelci in kmetje, ki so vzrejali domače živali (npr. Andrič et al. 2008; Tolar et al. 2011; Toškan et al. 2020).

Obdobje koliščarskih naselbin je povezano z začetki lokalne metalurgije, ki so jo naše raziskave postavile v prvo polovico 4. tisočletja pr. Kr., uporabo arzenovega bakra v 4. tisočletje, antimonovega bakra v začetek 3. tisočletja pr. Kr. (Trampuž-Orel

& Heath, 2008). Raziskovanje pasjega koprolita je omogočilo tudi vpogled v vlogo človekovega prvega živalskega spremljevalca v skupnosti (Tolar et al., 2021; Velušček et al., 2020).

Za prikaz oziroma razumevanje prazgodovinskega vsakdanjega življenja na Ljubljanskem barju je bila pomembna tudi rekonstrukcija tlorisa hiš v koliščarski naselbini Parte-Iščica, ki je potrdila, da je na koliščih stalo več manjših hiš s pretežno usmeritvijo od JZ proti JV (Velušček et al., 2000).

Tematika raziskav arheološkega lesa je objavljena tudi v video posnetkih, filmih in intervjujih, dostopnih na spletu. Predstavljeno je, kako je arheološki les občutljiv, če ga izpostavimo zraku (Drying characteristics, 2014a, b), širša tematika najstarejšega kolesa (Skrivnost barjanskega kolesa, 2015) in druga vprašanja v zvezi z lesom koliščarskih naselbin (Podobe znanja 2016, 2021; Vrhunci slovenske znanosti, 2020).

## 5 POVZETEK
## 5 SUMMARY


Ljubljansko barje is the only area with preserved archaeological remains of prehistoric pile-dwellings that has been discovered in Slovenia. In 2011, two groups of pile dwellings from the surroundings of the village of Ig were inscribed to the list of “Prehistoric Pile Dwellings around the Alps” on the UNESCO World Heritage List (Figure 1).

We present some results of wood research from the excavations held between 1995 and 2021 and coordinated by the Institute of Archaeology, Scientific Research Centre of the Slovenian Academy of Science and Arts (IA ZRC SAZU). In this campaign particular attention was paid to the collection of archaeological wood and the introduction of dendrochronology, which had to be newly developed in this area (Figures 2, 3, 4). The interdisciplinary approach also included the development of palynology, archaeobotany and archaeozoology, and collaboration with numerous partners from other fields such as archaeometry, textile engineering, etc. (e.g., Velušček 2004a, 2009).

We performed 29 field studies on 16 sites of prehistoric pile dwellings (Table 1). At each of the sites we collected samples of all woody elements, and then conducted wood analysis. The most common species were ash (*Fraxinus excelsior*), oak (*Quercus robur* and *Quercus petraea*), alder (*Alnus glutinosa*), maple (*Acer* sp.), willow (*Salix* sp.), poplar (*Populus* sp.), hazel (*Corylus avellana*), hornbeam (*Carpinus betulus*), beech (*Fagus sylvatica*), silver fir (*Abies alba*), and elm (*Ulmus* sp.) (Figure 5a, b).

Samples of oak, ash, and beech that contained more than 45 tree-rings were selected for dendrochronological analyses (Čufar & Velušček, 2012; Čufar et al., 2013; Velušček & Čufar, 2014). For each sample and site, the tree-ring series were cross-dated and assembled into floating chronologies (Figure 4, 6). For their dating, we collected representative samples for radiocarbon and wiggle-matching analyses (e.g. Čufar et al., 2010), and to teleconnect them with the existing references of the remote sites.

Oak (*Quercus* sp.) chronologies from the 4th millennium BC could be assembled to form a 442-year long chronology BAR-3330; its time span of 3771-3330 BC was defined with dendrochronological teleconnection (Čufar et al., 2015). The chronology was later extended and is currently 511 years long (time span 3840 – 3330 BC). Other chronologies with end dates younger than 3330 BC were dated using radiocarbon wiggle matching. These are Stare gmajne young and Veliki Otavnik Ib – their combined chronology SG-VO spans the period 3285-3108 ± 14 cal BC.

In the pile dwellings of the 3rd millennium BC in Ljubljansko barje, ash chronologies predominated. Their dating is currently mainly based on one radiocarbon date with lower accuracy and their dendrochronological dating is not very likely. While there exists a possibility to precisely date them by using modern radiocarbon methods supported by dendrochronology, this has not yet been fully attempted. The only longer and well replicated oak chronology of the 3rd millennium BC is the one from Založnica, ZAL-QUSP1, which spans the period 2659-2417 ± 18 cal BC.

The sites of Založnica with Parte and Dušanovo mark the end of the Copper Age at Ljubljansko barje (Velušček & Čufar, 2003; Velušček et al., 2011).

The presented end dates of the chronologies denote the last (dated) felling activity on the sites (Table 1, Figures 6, 7). Based on the felling dates of trees, we were also able to determine construction phases at the dwellings. Building activities or

repairs could be inferred from a large number of trees felled in the same year or within a narrow period of a few years. Some pile-dwelling settlements were occupied for several decades or even longer, while others existed only 20 years or less (Velušček, 2005).

The pile-dwellings from the Ljubljansko barje that were studied using dendrochronology existed in a time frame between ca. 4600 and 2400 BC. During this long period, the occupation of the Ljubljansko barje basin also showed several interruptions, the exact reasons for which remain unknown.

The dendrochronological findings complement each other with the findings on material culture, which helped to propose several cultural horizons delineated on the basis of the pottery.

The introduction of dendrochronology helped to define the time of existence of these cultures, which was a significant achievement for Slovenian prehistoric archaeology. It is now possible to compare absolute dates with all kinds of archaeological finds.

Dendrochronology also helped to obtain reliable dates for specific finds, like the oldest preserved wooden wheel with axle in the world, from the Stare gmajne site, aged around 5,150 years (Velušček et al., 2009).

The use of dendrochronology in the study of pile dwellings in Slovenia provides us with a number of new opportunities to think about various topics, such as prehistoric woodland management, which has been studied by analysing the age and diameter of roundwood from the pile-dwelling site of Stare gmajne (Out et al., 2020). Among other things, this work points to new possible future directions in the study of Ljubljansko barje as an archive of natural and anthropogenic history and human-environment interactions.

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# DR. JURE ŽIGON JE PREJEL JESENKOVO NAGRADO BIOTEHNIŠKE FAKULTETE UL ZA NAJBOLJŠEGA DIPLOMANTA DOKTORSKEGA ŠTUDIJA 3. STOPNJE 2021

# DR. JURE ŽIGON RECEIVED THE JESENKO AWARD OF THE BIOTECHNICAL FACULTY UL FOR THE BEST PHD GRADUATE IN 2021

Sebastian Dahle[1*], Marko Petrič[1]

*Slika 1. Dekanja Biotehniške fakultete prof. dr. Nataša Poklar Ulrih je nagrajencu dr. Juretu Žigonu podelila Jesenkovo nagrado za najboljšega diplomanta doktorskega študija.*

*Figure 1. The Dean of the Biotechnical Faculty, Prof. Dr. Nataša Poklar Ulrih, presented the Jesenko Award for the best PhD graduate to Dr. Jure Žigon.*

## *Izvleček / Abstract*

**Izvleček:** Jesenkove nagrade Biotehniške fakultete UL so najvišja priznanja za pedagoške, raziskovalne in strokovne dosežke ter za gospodarski in splošni družbeni napredek na področju biotehniških ved v Sloveniji. Biotehniška fakulteta jih podeljuje že vse od leta 1973, med njimi tudi Jesenkovo nagrado za najboljšega diplomanta doktorskega študija. Za leto 2021 je to nagrado na slovesnosti v marcu 2022 prejel asist. dr. Jure Žigon, ki je na Oddelku za lesarstvo BF zaposlen kot samostojni strokovni delavec.

**Ključne besede:** Jesenkova nagrada, najboljši diplomant doktorskega študija, Biotehniška fakulteta, Univerza v Ljubljani

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Dahle, S., & Petrič, M.: Dr. Jure Žigon received the Jesenko Award of the Biotechnical Faculty UL for the best PhD graduate in 2021

**Abstract:** The Jesenko Awards of the Biotechnical Faculty, University of Ljubljana, are the highest awards for pedagogical, research and professional achievements and for economic and general social progress in the biotechnical field in Slovenia. The Biotechnical Faculty has been giving these awards since 1973, including the Jesenko Award for the best doctoral studies graduate. For 2021 the recipient of this award was Assist. Dr. Jure Žigon, who received it at a celebration in March 2022. Dr. Žigon is employed at the Department of Wood Science and Technology at the Biotechnical Faculty as an independent professional associate.

**Keywords:** Jesenko Award, the best graduate of doctoral studies, Biotechnical faculty, University of Ljubljana

Jesenkove nagrade predstavljajo najvišja priznanja Biotehniške fakultete za pedagoške, raziskovalne in strokovne dosežke ter za gospodarski in splošno družbeni napredek na področju biotehniških ved v Sloveniji. Jesenkovo nagrado, ustanovljeno leta 1973, podeli Biotehniška fakulteta vsako leto do trem posameznikom, in sicer za življenjsko delo, za najboljšega diplomanta doktorskega študija ter za najboljšega diplomanta magistrskega študija. Podelitev nagrade za najboljšega diplomanta doktorskega študija v letu 2021 letošnjemu prejemniku dr. Juretu Žigonu je prikazana na sliki 1.

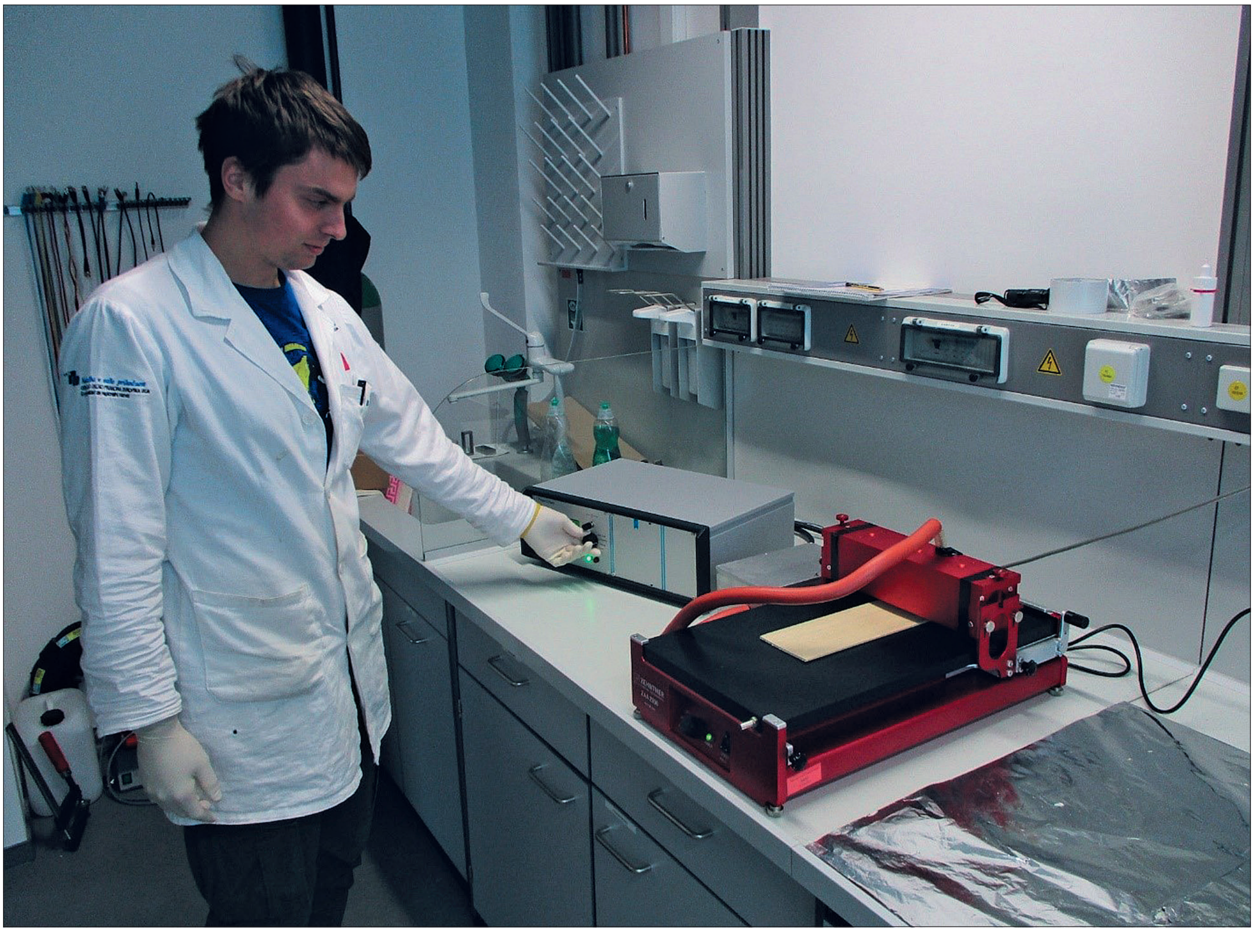
Prejemnik se je rodil v Ljubljani, kjer je obiskoval osnovno šolo in nato Srednjo lesarsko šolo, preden je začel s študijem lesarstva na Biotehniški fakulteti Univerze v Ljubljani. Le-tega je končal leta 2012 (BSc.) in 2014 (MSc.).

Osnovno področje znanstvenega delovanja dr. Jureta Žigona je lesarstvo, s svojimi raziskavami pa deluje tudi na drugih povezanih področjih, tako znotraj Univerze v Ljubljani in tudi izven nje. Njegove objave vključujejo raziskave premazov za les, njihove oprijemnosti, vremenske vplive na obdelane površine, lepljenje lesa in drugih materialov, modifikacijo lesa z organskimi in anorganskimi pripravki, utekočinjanje lesa, odpornost proti glivam, lesne mikrostrukture, 3D tisk in celo oprijem bakterij na lignocelulozne podlage.

Nagrajenec je doktoriral z izjemno bibliografijo, ki vsebuje 31 izvirnih znanstvenih objav, 1 pregledni znanstveni članek, 1 patentno prijavo ter vrsto drugih objavljenih del in znanstvenih uspehov. Od teh predstavlja 13 objav neposredni del njegove doktorske disertacije. Na področju lesarstva je kljub svoji mladosti že mednarodno priznan, tako s svojimi objavami, kot tudi zaradi gostovanj na drugih institucijah v okviru mednarodnih znanstvenih izmenjav. Raziskovalno delo je izven dela na matični instituciji opravljal na šestih znanstvenih inštitucijah v štirih evropskih državah (sliki 2 in 3).

Tema doktorata Jureta Žigona je bila osredotočena na tehnologijo, ki smo jo na Oddelku za

*Slika 2. Jure Žigon med udeleženci mednarodne šole inovacij Sprungbrett, 2016.*
*Figure 2. Jure Žigon among the participants of the international Innovation School Sprungbrett, 2016.*



*Slika 3. Jure Žigon med kratkim znanstvenim obiskom (COST action FP1407) na Tehniški univerzi Clausthal v Nemčiji, leta 2017.*
*Figure 3. Jure Žigon during a short-term scientific mission (COST action FP1407) at Clausthal University of Technology, Germany, 2017.*

lesarstvo uvedli nedavno, in sicer na izkoriščanje netermičnih plazemskih razelektritev. Skupaj z mentorjem doc. dr. Sebastianom Dahlejem je dr. Žigon razvil in izdelal plazemsko napravo z novim principom delovanja za obdelavo lesa, tako da naprava uporablja lesene obdelovance kot elektrode s plavajočim potencialom. To novo vrsto plazme z dielektrično barierno razelektritvijo s plavajočo elektrodo je uspešno uporabil za modifikacijo površin lesa, npr. za večjo hidrofilnost in združljivost s premazi na vodni osnovi. Čeprav te spremembe niso trajne, je bilo ugotovljeno, da trajajo dovolj dolgo in so zato uporabne za različne aplikacije, vključno z obdelavo lesa z notranjimi in zunanjimi premazi ter izboljšano trdnostjo lepilnega spoja.

Poleg znanstvenoraziskovalnega dela se dr. Žigon aktivno ukvarja z vsakodnevnimi dejavnostmi na Oddelku za lesarstvo. Njegove odgovornosti segajo od upravljanja analitske opreme, npr. vrstičnega elektronskega mikroskopa, do dejavnosti testiranja za komercialne partnerje in najrazličnejših tehničnih opravil v laboratorijih in v delavnici Oddelka, vključno s praktičnimi mizarskimi spretnostmi obdelave lesa in z načrtovanjem in izdelavo najrazličnejših lesenih izdelkov v okviru raziskovalnih projektov ter pedagoškega dela. Poleg tega dr. Žigon sodeluje v dejavnostih odnosov z javnostjo, vključno z dogodki za dijake (slika 4), pri poučevanju eksperimentalnih predmetov in pri nadzoru diplomskega dela študentov.

Z vso svojo angažiranostjo, motivacijo in znanstveno odličnostjo se je dr. Žigon izkazal kot pomemben del Katedre za lepljenje, lesne kompozite, obdelavo površin in konstruiranje (slika 5) ter se je

*Slika 4. Dr. Žigon dijakom predstavlja laboratorij za obdelavo površin lesa.*
*Figure 4. Dr. Žigon presents the wood surface treatment laboratory to pupils.*

*Slika 5. Skupina laboratorija za obdelavo površin, od leve: doc. dr. Pavlič, doc. dr. Dahle, dr. Žigon, prof. dr. Petrič.*
*Figure 5. Work group of the surface treatment laboratory, from left: Assist. Prof. Dr. Pavlič, Assist. Prof. Dr. Dahle, Dr. Žigon, Prof. Dr. Petrič.*

*Slika 6. Dekanja prof. dr. Nataša Poklar Ulrih s tremi letošnimi nagrajenci Jesenkovih nagrad: dr. Jure Žigon (levo), Anja Kos, prejemnica Jesenkove nagrade za najboljšo diplomantko magistrskega študija (desno ob dekanji) in prof. dr. Jurij Diaci, prejemnik Jesenkove nagrade za življenjsko delo (desno)*
*Figure 6. The dean, Prof. Dr. Nataša Poklar Ulrih, with this year's three recipients of the Jesenko Awards: Dr. Jure Žigon (left), Anja Kos, recipient of the Jesenko Award for the best graduate of MSc studies (on the right side of the dean) and Prof. Dr. Jurij Diaci, recipient of the lifetime achievement Jesenko Award.*

med Jesenkove nagrajence gotovo uvrstil zasluženo (slika 6). Vsi na Oddelku za lesarstvo smo ponosni, da imamo v svoji sredini takega sodelavca kot je dr. Žigon in veseli, da lahko z njim delimo čast Jesenkove nagrade (slika 7).

The Jesenko Awards represent the Biotechnical Faculty's highest recognitions for pedagogical, research and professional achievements, as well as for economic and general social progress in the field of biotechnical sciences in Slovenia. Established in 1973, the Biotechnical Faculty honours up to three individuals a year with the Jesenko Award for Lifetime Achievement, for the Best Doctoral Graduate, and for the Best Master's Degree. The presentation of the award for the best doctorate to this year's recipient, Dr. Jure Žigon, is shown in Figure 1.

Dr. Žigon was born in Ljubljana, where he attended primary school, followed by the wood-working high school, before he began his studies in Wood Science and Technology at the Biotechnical Faculty of the University of Ljubljana, which he finished in 2012 (B.Sc.) and 2014 (M.Sc.).

The primary area of Dr. Žigon's investigations is the field of wood science and technology, although he is active in a variety of other scientific fields across the University of Ljubljana and beyond. His published research includes work on wood coatings, adhesion and weathering, bonding of wood and other materials, organic and inorganic surface modification of wood, liquefaction of wood, resistance against fungi, wood microstructures, 3D printing, and even adhesion of bacteria on various lignocellulosic substrates.

Dr. Žigon finished his doctorate with an exceptional bibliography, presenting 31 original scientific publications, one review article, one patent appli-

*Slika 7. Dr. Jure Žigon, prejemnik Jesenkove nagrade za najboljšega diplomanta doktorskega študija 3. stopnje 2021.*
*Figure 7. Dr. Jure Žigon, recipient of the Jesenko Award for the best doctoral studies graduate in 2021.*

cation and a number of other published works and scientific successes. Out of these, 13 publications are a direct part of the doctoral dissertation. In spite of his youth, he is already internationally recognised in the field of wood science and technology, both through his published works as well as through international scientific exchanges, including work performed at six scientific institutions in four European countries (Figs. 2 & 3).

Dr. Žigon's doctorate focused on a technology that was only recently introduced at the Department of Wood Science and Technology – the utilisation of non-thermal plasma discharges. Together with his mentor, Assist. Prof. Dr. Sebastian Dahle, Dr. Žigon developed and built a plasma device with novel operating principle for wood treatments, utilising the wooden work pieces like electrodes at a floating potential. This new floating-electrode dielectric barrier discharge plasma successfully modified wood surfaces for increased hydrophilicity and compatibility with water-based coatings. Although these modifications were not permanent, they were found to last for durations long enough to be useful for various applications, including wood finishing with indoor and outdoor coatings as well as improved adhesive bonding.

In addition to scientific research, Dr. Žigon is actively engaged in the daily activities at the Department of Wood Science and Technology. His responsibilities range from operating analytical equipment like the department's Scanning Electron Microscope, to testing activities for commercial partners and technical activities in the department's laboratories and workshop, including practical carpenter skills and the construction and building of various wooden products in the frame of research projects and for pedagogical purposes. Moreover, Dr. Žigon participates in public relations activities, including events for pupils (Fig. 4), in teaching experimental courses and in support of students' diploma work.

Through all his engagement, motivation and scientific excellence, Dr. Žigon has proven an important part of the department's Chair of Adhesives, Wood Composites, Surface Treatment and Construction (Fig. 5) and a worthy recipient among the Jesenko awardees (Fig. 6). Together with the department, we are glad to have such a colleague as Dr. Žigon in our midst, and happy for and with him to have received this well-deserved honour (Fig. 7).

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# Srečanje mikroskopistov Slovenije – nagrada za najboljši poster dr. Angeli Balzano

# Meeting of Microscopists of Slovenia – Best Poster Award to Dr. Angela Balzano

Maks Merela

V mesecu maju 2022 se je v Ankaranu v organizaciji Slovenskega društva za mikroskopijo (SDM) odvilo 4. slovensko posvetovanje mikroskopistov. Cilji posveta so povezovanje mlajših in že uveljavljenih raziskovalcev in drugih uporabnikov mikroskopskih tehnik s področij znanosti o življenju in o materialih ter industrije, predstavitve novejših dosežkov s področja mikroskopije, povezovanje laboratorijev, raziskovalnih skupin in predstavnikov industrije ter njihovih mikroskopskih metodologij na vseh nivojih ločljivosti. Namen predstavitev je tudi pregled nad dostopnimi mikroskopskimi tehnikami v Sloveniji, z namenom izboljšanja izkoriščenosti opreme ter izmenjave znanja in izkušenj med različnimi vejami mikroskopije. Srečanja se je udeležila tudi raziskovalna skupina Biotehniške fakultete–Oddelka za lesarstvo. Z vodilno avtorico dr. Angelo Balzano je ekipa prejela priznanje za najboljši poster. Dr. Balzano je predstavila nov protokol priprave vzorcev mokrega arheološkega lesa za analizo z vrstičnim elektronskim mikroskopom "Scanning Electron Microscopy protocol for the characterization of waterlogged archaeological wood". Plakat je dostopen na povezavi: https://znc.si/dogodki/ostali-dogodki/4-slovensko-posvetovanje-mikroskopistov.

Iskrene čestitke za odlično pripravljen in predstavljen poster!

The 4th Slovenian meeting of microscopists, organized by the Slovenian Society of Microscopy (SDM), was held in Ankaran in May. The aim of the meeting was to bring together young scientists and established researchers as well as other users of microscopy techniques in the fields of life sciences, material sciences and industry, presenting the latest advances in microscopy, connecting laboratories, research groups and industry representatives and their microscopy methods at all resolution levels. The meeting also aimed to provide an overview of the microscopy techniques available in Slovenia in order to improve the use of the equipment and to share knowledge and experience among the different fields of microscopy. The meeting was also attended by the research group of the Biotechnical Faculty–Department of Wood Science and Technology. The team received the award for the best poster presented by the leading author, Dr. Angela Balzano. The poster, titled "Scanning Electron Microscopy protocol for the characterization of waterlogged archaeological wood", presented a new protocol for preparing waterlogged archaeological wood for scanning electron microscopy. The poster is available at: https://znc.si/dogodki/ostali-dogodki/4-slovensko-posvetovanje-mikroskopistov.

Congratulations to Dr. Angela Balzano for an excellently prepared and presented poster!

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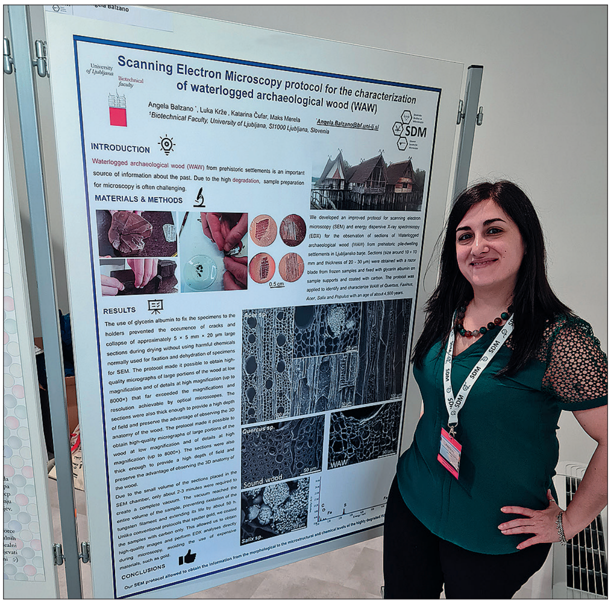



*Slika 1. Dr. Angela Balzano z nagrajenim plakatom na srečanju 4. slovensko posvetovanje mikroskopistov, Ankaran, 12. in 13. maj 2022.*

*Figure 1. Dr. Angela Balzano with the award-winning poster at the 4th SLOVENIAN MEETING OF MICROSCOPISTS, Ankaran, May 12-13, 2022.*

# Katedra za management in ekonomiko lesnih podjetij (Oddelek za lesarstvo Biotehniške fakultete) je prejela prestižno priznanje mednarodne asociacije WoodEMA

# The Chair of Management and Economics of Wood Companies (Department of Wood Science and Technology, Biotechnical Faculty) receives the prestigious award of the international association WoodEMA

Leon Oblak

Na 15. mednarodni znanstveni konferenci WoodEMA, ki je potekala od 7. do 10. junija v Trnavi na Slovaškem, je aktualni predsednik združenja prof. dr. Roman Dudik iz Prage Katedri za management in ekonomiko lesnih podjetij (Oddelek za lesarstvo Biotehniške fakultete) podelil prestižno priznanje mednarodne asociacije WoodEMA.

Združenje za ekonomiko in management v predelavi lesa in pohištvu WoodEMA je mednarodna, nepolitična, neprofitna in odprta znanstvena asociacija. Cilj združenja je promocija znanosti in rezultatov znanstvenega in strokovnega dela svojih članov, medsebojno znanstveno sodelovanje ter podpora znanosti in strokovnemu razvoju na področju dela asociacije. Združenje WoodEMA je bilo ustanovljeno leta 2007, danes pa ima člane, ki delujejo na širokem spektru ekonomskih in managerskih področij v gozdarstvu, predelavi lesa in proizvodnji pohištva. Združenje ima skoraj 100 aktivnih članov na treh celinah, od ZDA do Indije in Malezije ter v Evropi od Španije do Rusije ter od Finske do Severne Makedonije.

Vsako leto združenje WoodEMA na svoji skupščini podeli “štipendijo Roy Damary” za mlajše raziskovalce, v skladu s svojim statutom pa lahko podeli tudi posebno priznanje asociacije uglednim članom ali instituciji, ki so pomembno prispevali k znanstvenemu in strokovnemu razvoju združenja. Letos je priznanje prejela Katedra za management in ekonomiko lesnih podjetij, kar je prvi primer v zgodovini asociacije, da je nagrado prejela katera od institucij.

V obrazložitvi je bilo zapisanih veliko razlogov za podelitev tega priznanja. Vsi člani Katedre za management in ekonomiko lesnih podjetij so tesno vpeti v delo asociacije in nosilci številnih funkcij. Prof. dr. Leon Oblak je bil prvi predsednik asociacije, kasneje član upravnega, trenutno pa je član nadzornega odbora. Doc. dr. Jože Kropivšek je bil eden od ustanoviteljev združenja in dolgoletni član upravnega odbora. Doc. dr. Matej Jošt je trenutno član upravnega odbora, naslednje leto pa bo prevzel mesto predsednika asociacije. Anton Zupančič, ki je bil do leta 2018 član katedre, pa sedaj skrbi za urejanje spletne strani asociacije, ki je bila v celoti razvita v tej katedri. Katedra je tudi dvakrat zelo uspešno organizirala vsakoletno znanstveno konferenco, in sicer leta 2011 v Kozini in leta 2021 v Kopru, ki je zaradi covid epidemije potekala v hibridnem načinu in je bila po dveletnem obdobju pandemije prva konferenca, izvedena v živo na Univerzi v Ljubljani.

Zaradi vseh zgoraj navedenih in številnih drugih razlogov, zaradi prizadevanj in predanega dela članov katedre v korist združenja, je mednarodno Združenje za ekonomiko in management v predelavi lesa in pohištvu Katedri za management in ekonomiko lesnih podjetij podelilo priznanje asociacije WoodEMA za pomemben prispevek k razvoju združenja.


At the 15th international scientific conference WoodEMA, held in Trnava (Slovakia) from June 7 to 10, 2022, the current president of the association, Prof. Dr. Roman Dudik from Prague, Chair of Management and Economics of Wood Companies (Department of Wood Science and Technology, Biotechnical Faculty), received the prestigious recognition of the international association WoodEMA.

WoodEMA, the International Association for Economics and Management in Wood Processing and Furniture Manufacturing, is an international, non-political, non-profit and open scientific association. The aim of the association is to promote science and the results of the scientific and professional work of its members, mutual scientific cooperation and support of science and professional development in the association's field of work. WoodEMA was founded in 2007 and today has members working in a wide range of economic and management fields in forestry, wood processing and furniture manufacturing. The association has nearly 100 active members on three continents, from the United States to India and Malaysia, and in Europe from Spain to Russia and from Finland to Northern Macedonia.

Each year at its meeting WoodEMA awards a "Roy Damary Scholarship" to young scientists and, in accordance with its statutes, may give special recognitions to prominent members or institutions that have made significant contributions to the scientific and professional development of the association. This year, the Chair of Management and Economics of Wood Companies received the award, marking the first time in the association's history that an institution has received this honour.

Numerous reasons were cited in the rationale for giving the award. All members of the Chair of Management and Economics of Wood Companies are closely involved in the work of the association and hold many positions. Prof. Dr. Leon Oblak was the first president of the association, later a member of the Executive Board, and is currently a member of the Supervisory Board. Doc. Dr. Jože Kropivšek was one of the founders of the association and a long-time member of the Executive Board. Doc. Dr. Matej Jošt is currently a member of the Executive Board, and he will assume the position of president of the association next year. Anton Zupančič, who was a member of the department until 2018, is now taking care of editing the association's website, which was entirely developed in this department. The Chair of Management and Economics of Wood Companies has also organised the annual scientific conference twice, both times very successfully, in 2011 in Kozina and in 2021 in Koper, with the latter held in a hybrid mode due to the COVID epidemic, and after the two-year pandemic the first conference was held live and in person at the University of Ljubljana.

For all these and many other reasons, due to the efforts and dedicated work of the members of the Chair of Management and Economics of Wood Companies, the International Association for Economics and Management in Wood Processing and Furniture Industry has recognised the Chair of Management and Economics of Wood Enterprises for its important contributions to the development of the association itself.

## Gozdno-lesna veriga in podnebne spremembe: prehod v krožno biogospodarstvo

### Nov raziskovalni program, ki povezuje tri raziskovalne organizacije.

Davor Kržišnik in Jožica Gričar

Cilj novega raziskovalnega programa je identifikacija trenutnih izzivov znotraj gozdno-lesne verige vrednosti (od stoječih dreves do končnih izdelkov) in iskanje optimalnih rešitev na podlagi znanja in razpoložljivih virov treh raziskovalnih organizacij: Gozdarskega inštituta Slovenije, Univerze v Ljubljani, Biotehniške fakultete in Zavoda za gradbeništvo Slovenije. Raziskovalci teh treh organizacij prvič oblikujemo raziskovalno skupino z namenom zagotavljanja medsektorskega (gozdarstvo, lesarstvo, gradnja z lesom) in inovativnega pristopa za doseganje zastavljenih ciljev ter povezovanje skupine z industrijo.

Rezultati raziskovalnega programa se bodo kot inovativne rešitve prenesli tudi v prakso. S podporo trajnostnemu gospodarjenju, celovitemu varovanju gozdnih virov in biotske raznovrstnosti ter z uvajanjem okolju prijaznih, družbeno sprejemljivih in ekonomsko upravičenih izdelkov in delovnih procesov na osnovi lesa se bo povečal prispevek gozdno-lesne verige k nacionalnemu gospodarstvu.

Na področju 4.01 (gozdarstvo, lesarstvo in papirništvo) v okviru vede biotehnika je s 1. januarjem 2022 začel delovati raziskovalni program P4-0430 Gozdno-lesna veriga in podnebne spremembe: prehod v krožno biogospodarstvo. Javna agencija za raziskovalno dejavnost Republike Slovenije (ARRS) bo raziskovalni program P4-0430 financirala za obdobje šestih let v letnem obsegu 4250 raziskovalnih ur cenovne kategorije A letno.

Program je organiziran v pet delovnih sklopov (DS): DS1: sodobno gojenje gozdov za višjo kakovost gozdnih proizvodov, DS2: lastnosti lesa in izboljšani materiali na osnovi lesa, DS3: učinkovita raba lesa v gradbeništvu, DS4: gozdno-lesni sektor v biogospodarstvu ter DS5: projektno vodenje in diseminacija. Teme, ki jih naslavljamo v novem programu, so slabo raziskane, vendar imajo velik aplikativni potencial. Zastavljeni cilji zahtevajo sodelovanje več institucij v smislu zagotovitve potrebnih kompetenc in raziskovalnih zmogljivosti. Vsak delovni sklop sestavljajo raziskovalci vseh treh raziskovalnih organizacij, da bi zagotovili izmenjavo znanja in raziskovalnih idej med raziskovalci z različnim ozadjem. Takšno ustvarjalno okolje je osnova za raziskovanje širokih interdisciplinarnih raziskovalnih vprašanj in uporabo novih tehnik v gozdno-lesni vrednostni verigi. Interdisciplinarni pristop ter nacionalno in mednarodno sodelovanje bo eden temeljnih kamnov novega raziskovalnega programa.

# Klub alumnov lesarstva v letu 2022

## Alumni Club Wood Science and Technology in 2022

Katarina Čufar, Boštjan Lesar

Klub alumnov lesarstva, ki deluje kot sekcija v okviru kluba alumnov Univerze v Ljubljani Biotehniške fakultete in Društva lesarjev Slovenije, nadaljuje aktivnosti, ki jih je zavrla pandemija COVID, ko se alumni niso mogli zbirati in srečevati.

Na Oddelku za lesarstvo smo v maju 2022 organizirali akcijo seznanjanja študentk in študentov s klubom alumnov. Posvetili smo se spodbujanju vpisa v klub, kamor smo posebej povabili študentke in študente zaključnih 3. letnikov prvostopenjskega univerzitetnega študija lesarstva in visokošolskega študija Lesarsko inženirstvo, ki bodo polnopravni člani kluba lahko postali po diplomi. Še enkrat smo nagovorili študentke in študente 1. in 2. letnika magistrskega študija lesarstva, v kolikor se še niso vpisali v klub po diplomi prve stopnje.

Za zaključek akcije smo jih 12. 5. 2022 povabili tudi na skupno malico, ki smo jo priredili sredi delovnega četrtka v lepem vremenu pred Oddelkom za lesarstvo ob podpori tutork in tutorjev, mentoric in mentorjev letnikov in ostalih zaposlenih na oddelku. Malico je sponzoriralo Društvo lesarjev Slovenije, predsednik Tomaž Kušar in tajnik Jure Žigon pa sta bila skupaj z avtorjema tega prispevka med glavnimi organizatorji dogodka, ki ima veliko podporo vodstva in zaposlenih na Oddelku za lesarstvo, ki so kot vedno z veseljem priskočili na pomoč. Interes študentk in študentov je bil velik.

V juniju so Oddelek za lesarstvo obiskale alumne in alumni, ki so študij zaključili pred 40 leti. To je generacija, ki se je na univerzitetni študij lesarstva vpisala leta 1977 / 1978 in ga večinoma zaključila 1982. Organizator Janko Kebler je zbral imena in naslove 27 sošolcev in sošolk, od tega se jih je 19 udeležilo srečanja. V petek 10.6.2022 so se najprej zbrali na Oddelku za lesarstvo. Za mnoge je bil to prvi obisk fakultete po diplomi, ob čemer je treba poudariti, da so študij zaključili še v prostorih na Večni poti 2, v stavbi Gozdarskega inštituta Slovenije. Na Oddelku za lesarstvo jih je sprejel prodekan Marko Petrič, predstavitev Oddelka je pripravila Katarina Čufar, laboratorije pa so jim razkazali Marko Petrič, Miran Merhar, Eli Keržič, Luka Krže in Angela Balzano ob podpori Urške Kovačič in ostalih zaposlenih. »Alumni 40« so bili nad obiskom navdušeni. Obisk fakultete po tako dolgem času se jim je zdel zanimiv in nepozaben. Hvaležni so bili za gostoljubje zaposlenih in se veselili, koliko novega in zanimivega se dogaja na fakulteti, ki uživa velik mednarodni ugled. »Alumni 40« so družabni del srečanja nadaljevali pri Čadu, mi pa smo veseli, da so uradni del lahko izpeljali skupaj z nami.

Delo kluba alumnov Univerze v Ljubljani, kamor spada naša sekcija, je posebej vidno preko delovanja platforme https://alumniul.online, ki ima že več kot 11.000 članic in članov. Klub ponuja številne

ugodnosti. Alumni lesarstva na platformi še nismo množično prijavljeni, na srečo pa smo povezani preko Društva lesarjev Slovenije in preko Facebook skupine Alumni klub Oddelka za lesarstvo.

The Alumni Club of the Department of Wood Science and Technology, which functions as a section of the Alumni Club of the University of Ljubljana, Biotechnical Faculty, and the Association of the

Slovenian Wood Technologists, continues the activities that were put on hold by the COVID pandemic, when alumni were unable to get together and meet in person.

In May 2022, the Department of Wood Science and Technology organised a campaign to introduce the Alumni Club to students. The campaign was concluded with a joint brunch on May 12, 2022, in beautiful weather in front of the department. The event was supported by tutors, students, teachers, and the entire staff of the department. The brunch was sponsored by the Association of the Slovenian Wood Technologists.

On June 10, 2022, a group of alumni who graduated 40 years ago visited the Department of Wood Science and Technology. This is the generation that started studying wood science and technology in 1977/78 and mainly graduated in 1982. For many of them, it was their first visit to the department since graduation. The "Alumni 40" were welcomed by Vice-Dean Marko Petrič. They visited the laboratories, which were presented by the staff of the faculty. The "Alumni 40" were enthusiastic and found the visit interesting and memorable. We are glad that they continued the tradition of celebrating graduation anniversaries within the Alumni Club. •

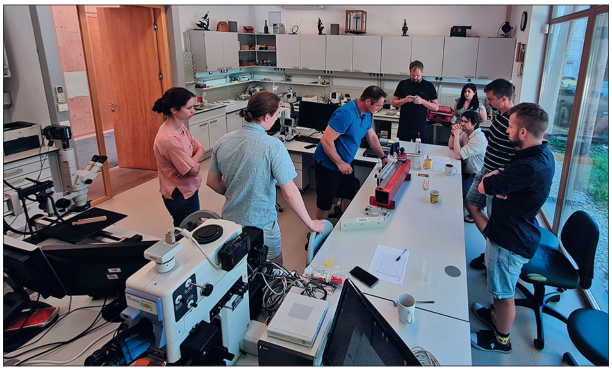
## Mednarodna delavnica »Manj znane lesne vrste v dendrokronologiji in kulturni dediščini«

## International workshop »Less known wood species in dendrochronology and cultural heritage«

Maks Merela, Angela Balzano

Od 14. do 16. junija 2022 je bila na Oddelku za lesarstvo Biotehniške fakultete Univerze v Ljubljani organizirana mednarodna delavnica v okviru projekta Interdisciplinary, collaborative learning and teaching for resilient wood resources and innovations in a digital world (WooD+; 2020-1-CZ-01-KA203-078483). Delavnica je pokrivala tematiko dendrokronologije manj znanih lesnih vrst na primeru dreves in elementov kulturne dediščine, tematiko različnih načinov vzorčenja in priprave vzorcev, meritve in analize, različne pristope dendrokronologije ter moderne izzive v dendrokronologiji.

Udeležence z Univerze Mendel Brno in Univerze za naravne vire in vede o življenju na Dunaju (BOKU) smo povabili k delavnici s pestrim programom, ki ga je v uvodu predstavil izr. prof. dr. Maks Merela. Program je vseboval predavanja, delo na terenu, praktične vaje in družabne dogodke. Oddelek za lesarstvo je udeležencem predstavil prof. dr. Milan Šernek, razvoj dendrokronologije v Sloveniji pa je v uvodnem predavanju predstavila prof.



dr. Katarina Čufar. Doktorska študentka Nina Škrk in dr. Angela Balzano sta s kratkimi predstavitvami predstavili bazo podatkov in spletno platformo "SloClim", ki vsebuje dnevne podatke o padavinah in temperaturah v visoki ločljivosti na mreži 1 x 1 km za celotno Slovenijo in je pomemben za dendrokronologijo in proučevanje podnebnih sprememb ter izzive na področju dendrokronologije pri sredozemskih in tropskih vrstah.

Terenske dejavnosti v okviru delavnice so vključevale odvzem vzorcev za dendrokronološke preiskave tako iz živih dreves v gozdnem sestoju, kot tudi iz objektov kulturne dediščine (lesene zgradbe). Prikazani so bili različni načini priprave vzorcev ter različna merilna in analitična orodja, ki se uporabljajo v dendrokronologiji. Prikazana je bila tudi priprava vzorcev arheološkega lesa in izzivi, povezani z datiranjem (C14 in metode wiggle matching). Poleg dejavnosti delavnice so bili za boljše povezovanje udeležencev organizirani družabni dogodki, ki so vključevali tudi »brain storming«, razprave na prostem in ogled Ljubljane z rečno ladjo, kjer nam je na pomoč priskočil naš alumen Anže Logar in firma Lakercraft. Intenzivna delavnica z delom v manjših skupinah se je izkazala za zelo uspešno tudi za izmenjavo izkušenj in predstavlja dobro osnovo za nadaljnje sodelovanje med vsemi udeleženci.

As part of the project "Interdisciplinary, collaborative learning and teaching for resilient wood resources and innovations in a digital world" (WooD+; 2020-1-CZ01-KA203-078483), the workshop "LKWS in dendrochronology & cultural heritage" was held at the Department of Wood Science and Technology, Biotechnical Faculty, University of Ljubljana from June 14 to 16, 2022.

The workshop covered topics related to the dendrochronology of Less Known Wood Species (LKWS) and wood from cultural heritage. The program included sampling, new methods and techniques, measurements and data analysis, different tools and current challenges in dendrochronology.

The participants from Mendel University Brno and the University of Natural Resources and Life Sciences, Vienna (BOKU) were welcomed to join the workshop with a program presented by Assoc. Prof. Maks Merela. The program included lectures, field sampling, practical exercises and social events. Prof. Dr. Milan Šernek presented the department, while Prof. Dr. Katarina Čufar gave an introductory lecture on the development of dendrochronology in Slovenia. PhD student Nina Škrk and Dr. Angela Balzano gave short presentations on "SloClim", a high-resolution daily gridded precipitation and temperature dataset for Slovenia useful for combining dendrochronology and climatic conditions, and the challenges of applying dendrochronology to Mediterranean and tropical species.

Field activities included coring for dendrochronological purposes from both living trees in a forest stand and cultural heritage objects (wooden buildings). Various sample preparation methods were shown, and the measurement and analysis tools used in dendrochronology were demonstrated. Sample preparation of archaeological wood and the challenges associated with dating (including C14 and wiggle matching methods) were also demonstrated. In addition to the workshop activities, social events such as outdoor brainstorming discussions and a Ljubljana city tour by boat supported by our alumni Anže Logar and Lakercraft were organised to better connect participants. The intensive small group workshop proved to be very successful with regard to sharing experiences, and we hope for further fruitful collaboration among all participants.