LES/WOOD

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LES/WOOD

UVODNIK / EDITORIAL

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Razvojno-raziskovalne aktivnosti v lesarstvu v znamenju digitalizacije in biogospodarstva

Pred vami je nova številka revije Les/Wood, ki je nastajala v drugem letu pandemije Covid-19, ki je krojila dogajanje tudi v gozdno-lesni verigi. Znanstveni prispevki v tej reviji prikazujejo nova spoznanja o tem, kako in kdaj nastaja kasni les pri navadni smreki, kaj se dogaja z zaščitenim lesom smreke na prostem po daljšem času izpostavitve in kaj se zgodi s kemičnimi in mehanskimi lastnostmi pri termični modifikaciji lesa hitro rastoče plantažne lesne vrste *Gmelina arborea*, ki jo uvajajo na degradiranih območjih v Gani, da bi omejili krčenje gozdov. Nadalje prispevki prikazujejo mehanizme nastanka nanodelcev srebra na ligninu, uporabnost lesnih ostankov invazivnih drevesnih vrst za proizvodnjo peletov in toplotno prevodnost različnih bio-izolacijskih materialov na osnovi lesnih ostankov. Vse naštete teme se dobro vklapljajo v razvojni koncept biogospodarstva, ki je posebej pomemben pri zagotavljanju dolgoročnejšega obvladovanja podnebnih sprememb in prehoda v nizkoogljično družbo. Biogospodarstvo kot pomembna razvojna usmeritev tako v gozdnem delu gozdno-lesne verige kot v predelavi lesa je bilo posebej izpostavljeno na Dnevih slovenskega lesarstva 2021 in na 9. Razvojnem dnevu gozdno-lesnega sektorja, kjer je bilo v letu 2021 identificiranih kar 59 aktualnih projektov, sofinanciranih iz javnih sredstev Republike Slovenije in/ali EU. To kaže na izjemno razvojno moč te verige, ki je ključna za njeno dolgoročnejšo uspešnost, poleg kratkoročnih odzivov podjetij na spremembe poslovnega okolja z uvajanjem novih tehnologij, poslovnih modelov in konceptov v svoja poslovanja.

Skupne teme razvojnih usmeritev bi poleg biogospodarstva lahko strnili še v eno ključno temo, to je digitalizacija oz. digitalna transformacija podjetij, panoge in družbe v celoti. Koncept digitalizacije z novimi tehnologijami in storitvami, ki so v času pandemije Covid-19 omogočale, da je družba bolj ali manj učinkovito delovala kljub (skoraj) popolnemu zaprtju javnega in gospodarskega okolja, še vedno predstavlja za podjetja (in državo) precejšen izziv. Največji izziv so digitalne kompetence managerjev, zaposlenih in vseh ljudi, ki so ključne za uvajanje novih digitalnih tehnologij in konceptov, ki so v sodobni družbi nujni za preživetje in nadaljnji razvoj. Razvoju le-teh je namenjen evropski projekt »All-View«, katerega cilj je digitalizacija in poenotenje izobraževalnih procesov na vseh nivojih izobraževanja na področju lesarstva, ki bodo vključevali digitalne vsebine za razvoj digitalnih kompetenc učečih se, in bodo temeljili na sodobni, pametni platformi. Ravno problem pomanjkanja digitalnih znanj in kompetenc v lesarstvu pa je bila skupna ugotovitev okrogle mize, ki je sledila predstavitvam projektov na prej omenjenem 9. Razvojnem dnevu gozdno-lesnega sektorja. Da pa je digitalizacija zelo prisotna tudi v lesarstvu, so na tem dogodku na primerih dobre prakse pokazali predstavniki več podjetij.

Glede na izkazano razvojno moč gozdno-lesne verige pri reviji Les/Wood tudi v prihodnje upamo na dotok zanimivih prispevkov. Hvala vsem, ki pomagate pri pripravi revije, še posebej (anonimnim) recenzentkam in recenzentom, ki ste nam s svojim ažurnim in kakovostnim delom priskočili na pomoč tudi pri pripravi te številke revije Les/Wood.

R&D activities in woodworking in the sign of digitalization and bioeconomy

In front of you is a new issue of the journal Les/Wood, which was created in the second year of the COVID-19 pandemic, which also shaped the entire forest wood chain. The scientific articles in this journal present new insights into how and when latewood is formed in spruce, what happens to protected spruce wood outdoors after a long exposure period, and what happens to the chemical and mechanical properties of thermally modified wood from the fast-growing plantation species *Gmelina arborea*, which is being introduced into degraded areas in Ghana to limit deforestation. In addition, this issue presents the mechanisms of the formation of silver nanoparticles on lignin, the usability of wood residues from invasive tree species for the production of pellets, and the thermal conductivity of various wood residue-based bio-insulation materials. All these topics fit well into the development of the concept of the bioeconomy, which is of particular importance for the long-term management of climate change and the transition to a low-carbon society. The bioeconomy as an important development direction in the forestry part of the forest-wood chain, as well as in wood processing, was particularly highlighted at the Days of the Slovenian Wood Sector 2021 and at the 9th Development Day of Forest-Wood Sector, where no less than 59 current projects supported by public funds of the Republic of Slovenia and/or the EU were identified. This shows the exceptional developmental strength of this chain, which is crucial for its long-term success, in addition to the short-term reactions of companies to changes in the business environment by introducing new technologies, business models and concepts into their businesses.

In addition to the bioeconomy, the common themes of the development orientations can be summarised in another key theme, namely digitalisation or the digital transformation of companies, industries and society as a whole. The concept of digitalisation with new technologies and services, which enabled society to operate reasonably efficiently despite the (almost) complete compartmentalisation of the public and economic environment during the COVID-19 pandemic, continues to pose significant challenges for both businesses and the state. The greatest challenges lie in the digital competencies of managers, employees, and the general population. These competencies are crucial for the adoption of new digital technologies and concepts in society, to aid both its further development and ultimate survival. The European project "AllView" is aimed at the development of these competencies, with the goal of digitalisation and unification of educational processes at all levels of education in the field of wood, which includes digital content for the development of digital competences of learners and is based on an up-to-date, smart platform. The lack of digital knowledge and competences in the wood sector was one of the main findings of the roundtable discussion which followed the presentations of projects at the previously mentioned 9th Development Day of the Forest-Wood Sector. However, with presentations of cases of good practice at this event, representatives of several companies showed that digitalisation is already present in the wood sector.

Due to the proven development strength of the forest-wood chain, an (increased) influx of interesting articles at Les/Wood journal can be expected in the future. Our thanks go to all those who contribute to the preparation of the journal, especially to the (anonymous) reviewers who help us with their up-to-date and high-quality work in the preparation of this issue of Les/Wood.

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NASTAJANJE IN STRUKTURA LESA IN FLOEMA PRI NAVADNI SMREKI

FORMATION AND STRUCTURE OF WOOD AND PHLOEM IN NORWAY SPRUCE

Jožica Gričar[1*], Katarina Čufar[2], Peter Prislan[1]

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Izvleček / Abstract

Izvleček: *Baze podatkov o nastajanju lesa in floema so pomembne za razumevanje vpliva podnebnih sprememb in izrednih vremenskih dogodkov na vrstno sestavo, vitalnost dreves, produkcijo ter kakovost lesa v slovenskih gozdovih. V tem članku predstavljamo najnovejše rezultate o debelinski rasti navadne smreke (*Picea abies *(L.) Karst.) z dveh rastišč v Sloveniji, na Panški reki (PAN – 400 m n. v.) in Menini planini (MEN – 1200 m n. v.) v letih 2009–2011. Poudarek je bil na sezonski dinamiki nastajanja ranega in kasnega lesa ter ranega in kasnega floema. Ugotovili smo, da rastiščne razmere v veliki meri vplivajo na sezonsko dinamiko nastajanja lesa in floema, kar se odraža v širini in strukturi prirastkov. Na višje ležečem rastišču MEN je bila rastna sezona približno mesec dni krajša (dolga slabe 4 mesece), posledično so bili letni prirastki ožji, in sicer v lesu za 39 % in v floemu za 15 %. Na MEN smo prehod iz ranega v kasni les v povprečju opazili le teden dni kasneje kot na PAN, medtem ko je prehod iz ranega v kasni floem nastopil v povprečju 20 dni kasneje. Informacije o vplivu rastiščnih razmer na debelinsko rast smreke in kakovost lesa so pomembne za vse deležnike v gozdno-lesni verigi, saj so lahko v pomoč pri sprejemanju ustreznih ukrepov upravljanja za prilagoditev spremenjenim razmeram.*

Ključne besede: *rani les, kasni les, rani floem, kasni floem, branika, kambij, navadna smreka-*Picea abies

Abstract: *Wood and phloem formation databases are important for understanding the effects of climate change and extreme weather events on species composition, tree vitality, wood production and wood quality in Slovenian forests. In this paper, we present the latest results on the radial growth of Norway spruce (*Picea abies *(L.) Karst.) at two sites in Slovenia, Panška reka (PAN – 400 m a. s .l.) and Menina planina (MEN – 1200 m a. s .l.) in 2009–2011. The focus was on the seasonal dynamics of early and latewood, and early and late phloem formation. We found that site conditions greatly affected the seasonal dynamics of wood and phloem formation, which was reflected in the width and structure of annual increments. At the higher elevation MEN site, the growing season was about a month shorter (about 4 months long), which resulted in 39% and 15% narrower wood and phloem increments, respectively. At MEN, the transition from early to latewood was observed on average only a week later than at PAN, while the transition from early to late phloem occurred on average 20 days later at MEN than at PAN. Information on the impact of site conditions on radial growth of spruce and wood quality is important for all stakeholders in the forest-wood value chain, as it can help to take appropriate management measures of adaptation to changing conditions.*

Keywords: *earlywood, latewood, early phloem, late phloem, growth ring, cambium, Norway spruce =* Picea abies

1 UVOD

1 INTRODUCTION

Podnebne spremembe in z njimi povezani izredni vremenski dogodki kot so suše, veter, žled, vročinski valovi in pozebe, vplivajo na vrstno sestavo, vitalnost dreves, produkcijo in kakovost lesa v slovenskih gozdovih (IPCC, 2014; Krajnc, et al., 2021). V Sloveniji je trenutno gospodarsko najpomembnejša drevesna vrsta navadna smreka (*Picea abies* (L.) Karst.), ki je v letu 2019 predstavljala 30,4 % delež v lesni zalogi. Vse pogostejši izredni dogodki v zadnjih letih so resno ogrozili njeno odpornost in s tem povečali dovzetnost oslabljenih dreves za napad podlubnikov ali drugih škodljivcev (de Groot & Ogris, 2019), kar se odraža v povečanem obsegu sanitarne sečnje v celotnem poseku v obdobju 2012–2017, ki znaša kar 62 % oziroma 9,4 mio m^3 (Zavod za gozdove Slovenije, 2020). Posledica intenzivnih sanitarnih sečenj so velike količine dostikrat poškodovanega in manj vrednega lesa smreke na domačih in svetovnih trgih. Razlog za veliko zasto-

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panost smreke v slovenskih gozdovih je nekdanja gozdarska praksa, ki je pospeševala pogozdovanja s smreko, tudi v obliki monokulturnih nasadov na zanjo nenaravnih rastiščih in v nižinah. Njen potencialni naravni delež je ocenjen na 8 % (Krajnc et al., 2020a). Pričakujemo, da se bodo intenzivne sanitarne sečnje nadaljevale tudi v prihodnje, obenem pa se bo nadaljevalo zmanjševanje deleža smreke v slovenski lesni zalogi.

Podnebne spremembe zaradi naraščajočih temperatur negativno vplivajo na rast dreves na rastiščih na nižjih nadmorskih višinah. Hkrati številne dendroklimatološke študije kažejo, da lahko naraščanje temperature pozitivno vpliva na debelinsko rast smreke na višje ležečih rastiščih (Levanič et al., 2009; Jevšenak et al., 2021). Podnebne spremembe bodo tako verjetno različno vplivale na rast in preživetje dreves v različnih okoljih, pri čemer moramo upoštevati še vpliv izrednih vremenskih dogodkov, ki se lahko pojavijo na globalni, regionalni ali lokalni ravni in praviloma negativno vplivajo na rast dreves.

Informacije o časovni dinamiki sezonske debelinske rasti dreves ter struktura lesa in floema lahko služijo kot kazalniki za odziv in prilagoditev dreves na dane rastiščne razmere in izjemne dogodke (Sass-Klaassen et al., 2016). Raziskave sezonske dinamike debelinske rasti dreves so časovno zamudne, saj zajemajo odvzem vaskularnih tkiv v kratkih časovnih intervalih tekom rastne dobe ter nadaljnjo pripravo vzorcev v laboratoriju za opazovanje tkiv pod mikroskopom. Posebno dragocena, a redka so večletna opazovanja, ki prikazujejo razlike v vzorcih rasti med leti na istem rastišču v odvisnosti od različnih (zunanjih) dejavnikov (Prislan et al., 2019). Zato so informacije o nastajanju lesa dokaj omejene, še redkejše pa so študije, ki vključujejo tudi nastajanje floema.

V Sloveniji imamo večletne podatke o sezonskem delovanju kambija ter nastajanju lesa in floema za navadno smreko in navadno bukev (*Fagus sylvatica* L.) na dveh rastiščih (npr. Prislan et al., 2013, 2019; Gričar et al., 2014, 2021) ter za puhasti hrast (*Quercus pubescens* Willd.), črni gaber (*Ostrya carpinifolia* Scop.), mali jesen (*Fraxinus ornus* L.) in črni bor (*Pinus nigra* Arn.) na Podgorskem krasu (Gričar et al., 2017, 2020). Za temeljite raziskave zvez med debelinsko rastjo in podnebjem pri različnih drevesnih vrstah na globalni ravni pa raziskovalci iz različnih laboratorijev po svetu intenzivno sodelujejo, izmenjujejo znanje, usklajujejo metodologijo raziskav in združujejo podatke v skupne baze (npr. Rossi et al., 2008; Cuny et al., 2015; Martinez del Castillo et al., 2018; Huang et al., 2020).

Širine letnih prirastkov (branik) v lesu so v tesni zvezi z njegovo strukturo, zato so informacije o vplivu rastiščnih razmer na gozdno produkcijo (debelinsko rast) in kakovost lesa pomembne za vse deležnike v gozdno-lesni verigi. Za drevesne vrste zmernega pasu je značilno periodično delovanje kambija, ki je povezano z okoljskimi dejavniki, z izmenjavami hladnih in toplih ali pa sušnih in deževnih obdobij (Lachaud et al., 1999). Rast in razvoj dreves se v normalnih razmerah začne spomladi in zaključi pozno poleti ali zgodaj jeseni. Na vzorce rasti vplivajo številni dejavniki, kot so rastiščne razmere, drevesna vrsta, starost, vitalnost in socialni položaj drevesa (Larson, 1994). S periodičnim delovanjem meristemskih tkiv in diferenciacijo celic variira tudi količina produktov fotosinteze, hormonov in drugih signalnih molekul v drevesu, ki vplivajo na strukturo lesa. Spomladi nastajajo traheide ranega lesa, ki imajo velike lumne in tanke celične stene. V drugi polovici rastne sezone nastajajo traheide kasnega lesa, ki imajo majhne lumne in debele celične stene (Čufar, 2006). Razmejevanje med ranim in kasnim lesom navadno temelji na razmerju med radialno dimenzijo lumna in debelino tangencialne celične stene (Denne, 1988). Omenjene razlike med ranim in kasnim lesom narekujejo njihovo vlogo v drevesu, pri čemer so traheide ranega lesa bolj učinkovite pri prevajanju vode, traheide kasnega lesa pa so pomembne za zagotavljanje mehanske trdnosti. Z vidika uporabe lesa so pomembne razlike v količini stenskega materiala, saj narekujejo gostoto lesa, pri čemer ima kasni les do trikrat višjo gostoto od ranega lesa. Širine branik ter deleži ranega in kasnega lesa zato bistveno vplivajo na gostoto lesa, ki je eden glavnih kazalnikov lastnosti in kakovosti lesa (Panshin & de Zeeuw, 1980; Čufar, 2006; Gorišek, 2009). Zelo malo je znanega o času prehoda iz ranega v kasni les oziroma o času nastanka ranega in kasnega lesa, a imajo te informacije zaradi zgoraj omenjenih razlogov velik aplikativni pomen.

V raziskavah debelinske rasti dreves je floemski prirastek dostikrat spregledan zaradi manjšega gospodarskega pomena tkiv skorje. Poleg tega so včasih domnevali, da na nastanek floema vplivajo predvsem notranji dejavniki (npr. Larson, 1994), novejše raziskave pa so pokazale, da je nastanek floema vsaj deloma podvržen rastiščnim razmeram, kar se odraža v njegovi strukturi, zato ga je smiselno vključiti v ekofiziološke in dendroekološke študije (Gričar et al., 2015, 2016, 2020). Nenazadnje je kambij bifacialen meristem, ki proizvaja celice na lesno in floemsko stran, zato je nujno, da je v raziskavah delovanja kambija vključen tudi floemski del (Gričar, 2017). Podobno kot v lesu tudi v nekolabiranem (prevodnem) floemu pri smreki lahko razločimo prirastne plasti (floemske branike), razmejene z letnicami. Znotraj floemskih branik razlikujemo rani in kasni floem, ki ju razmejuje bolj ali manj sklenjen tangencialni pas aksialnega parenhima (Gričar et al., 2005, 2006, 2014, 2015, 2016; Gričar, 2017). Sitaste celice ranega in kasnega floema se razlikujejo predvsem po radialni dimenziji lumnov. Osrednja naloga ranega floema je prevajanje asimilatov in drugih molekul iz listov po deblu proti koreninam do meristemskih in skladiščnih tkiv. Kasni floem, ki vsebuje aksialni parenhim, je pomemben za skladiščenje nestrukturnih ogljikovih hidratov (Jyske & Hölttä, 2015). Sitaste celice so žive in opravljajo svojo nalogo eno do dve leti, nato odmrejo in kolabirajo. Vsakoletni nastanek floemske branike je tako ključen za preživetje drevesa (Gričar, 2017).

Namen prispevka je predstaviti nekaj najnovejših rezultatov o debelinski rasti navadne smreke z dveh rastišč v Sloveniji, na Panški reki (PAN – 400 m n. v.) in Menini planini (MEN – 1200 m n. v.) v letih 2009–2011. Raziskali smo: (a) kambijevo celično produkcijo, z glavnimi fazami, ki vključujejo začetek, konec, trajanje in stopnjo produkcije celic lesa in floema; (b) prehod iz ranega v kasni les in iz ranega v kasni floem, ki je v splošnem slabo poznan ter (c) strukturo lesnih in floemskih prirastkov.

2 MATERIAL IN METODE

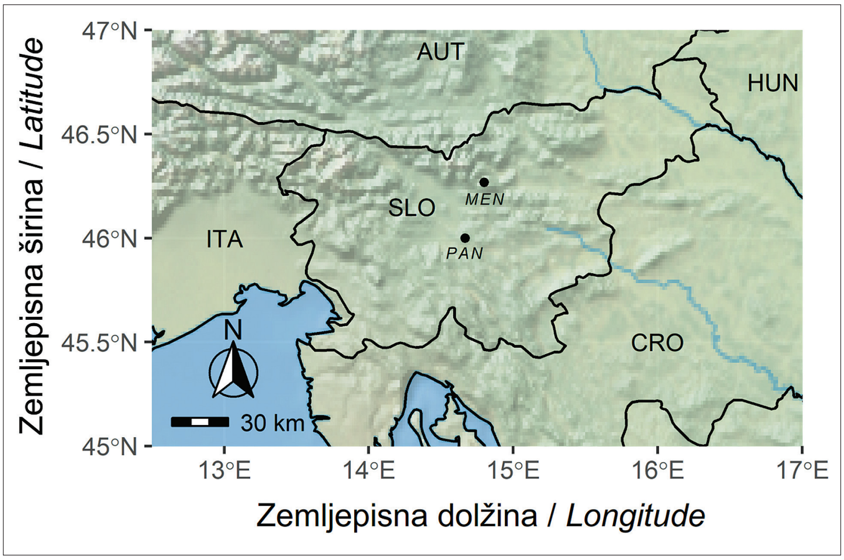
2 MATERIAL AND METHODS

2.1 RASKOVALNI PLOSKVI

2.1 STUDY SITES

Raziskava je bila opravljena na rastiščih Panška reka PAN in Menina planina MEN v Sloveniji, ki se razlikujeta v nadmorski višini (PAN – 400 m n.v. in MEN – 1200 m n.v.) (slika 1). Rastišče PAN se nahaja v bližini Ljubljane, kjer rastejo značilni podgorski bukovi gozdovi (*Hacquetio-fagetum typicum*) s prevladujočimi vrstami navadna bukev (*Fagus sylvatica* L.), gorski javor (*Acer pseudoplatanus* L.) in navadna smreka (*Picea abies* (L.) H. Karst.). Višje ležeče rastišče MEN se nahaja na Menini planini, predalpski planoti v Kamniško-Savinjskih Alpah, za katerega je značilen predalpski visokogorski gozd jelke in bukve (*Abieti fagetum prealpinum typicum*) in prevladujejo vrste navadna bukev, navadna smreka in bela jelka (*Abies alba* Mill.).

Vremenske podatke za rastišče PAN smo pridobili iz bližnje meteorološke postaje Agencije RS Slovenije za okolje (Ljubljana–Bežigrad 46°30´ N, 14°30´ E, 299 m n. v.). Na rastišču MEN smo za čas spremljanja debelinske rasti dreves namestili vremensko postajo Davis®, ki je v enournih intervalih beležila temperaturo in količino padavin. Namestili



Slika 1. Zemljevid Slovenije in lokaciji rastišč Panška reka (PAN – 400 m n. v.) in Menina planina (MEN – 1200 m n. v.) (sliko pripravil D. Arnič).

Figure 1. Map of Slovenia with locations of the selected sites Panška reka (PAN – 400 m a.s.l.) and Menina Planina (MEN – 1200 m a. s. l.) (figure prepared by D. Arnič).

smo jo na jasi, približno 2 m nad tlemi v bližini izbranih dreves. Letna količina padavin je bila na rastiščih primerljiva (PAN – 1384 mm in MEN – 1355 mm), razlike pa smo zabeležili v letni povprečni temperaturi (PAN 10,3 °C in MEN 7,4 °C).

2.2 IZBIRA DREVES, IZVEDBA VZORČENJA IN PRIPRAVA VZORCEV

2.2 SELECTION OF TREES, TISSUE SAMPLING, AND SAMPLE PREPARATION

Na obeh rastiščih smo izbrali šest dominantnih ali sodominantnih dreves smreke. Drevesa na PAN so bila stara 68 ± 8 let, s premerom na prsni višini 36 ± 5 cm in višino 30 ± 5 m. Na MEN so bila drevesa stara 102 ± 31 let, s premerom na prsni višini 34 ± 2 cm in višino 25 ± 1 m. V letih 2009, 2010 in 2011 smo med rastno sezono (t. j. od sredine marca do sredine oktobra) v tedenskih intervalih z uporabo orodja Trephor (Rossi et al., 2006) iz debel dreves jemali mikro izvrtke premera 1,8 mm in dolžine približno 20 mm. Vzorce smo odvzeli po obodu drevesa na višini debla od 1,1 do 1,7 m nad tlemi tako, da smo sledili obliki vijačnice. V izogib vpliva poškodb na odvzeta tkiva na sosednjih vzorcih smo zaporedna mesta vzorčenja izbrali z razdaljo od 5 do 10 cm. Vsak mikro izvrtek je vseboval vsaj dve najmlajši lesni braniki, kambij in ličje (floem). Postopke priprave trajnih preparatov prečnih prerezov mikro izvrtkov za opazovanje in izvedbo meritev pod mikroskopom smo podrobno opisali v Gričar et al. (2014). Rezine debele 10–12 µm za pregled pod svetlobnim mikroskopom smo narezali z rotacijskim mikrotomom in jih obarvali z vodno mešanico barvil safranin in astra modro ter jih vklopili v Euparal. Opazovanje in meritve nastajajočih lesnih (ksilemskih) in floemskih branik smo opravili s svetlobnim mikroskopom (svetlo polje in polarizirana svetloba) ter sistemom za analizo slike.

2.3 FENOLOGIJA KAMBIJEVE AKTIVNOSTI IN ANATOMIJA LESNIH IN FLOEMSKIH BRANIK

2.3 PHENOLOGY OF CAMBIAL ACTIVITY AND ANATOMY OF XYLEM AND PHLOEM INCREMENTS

Začetek, konec in trajanje kambijeve celične produkcije lesne in floemske branike smo določili, kot opisuje prispevek Gričar et al. (2014). Na vseh preparatih smo prešteli število celic v nastajajoči lesni in floemski braniki v vsaj treh radialnih nizih ter na podlagi podatkov za vsako drevo izračunali Gompertzovo funkcijo, ki opisuje sezonsko dinamiko nastajanja lesa in floema. Kambijevo produkcijo na lesni in floemski strani prikazujejo glavni mejniki: začetek, konec in trajanje nastajanja celic. Začetek kambijeve celične produkcije smo določili kot dan, ko smo opazili prve novo nastale lesne celice v fazi površinske rasti. Konec kambijeve celične produkcije smo določili kot dan, ko lesnih celic v fazi površinske rasti nismo več zasledili ob kambiju, opazili pa smo traheide v poznih fazah diferenciacije. Konec nastajanja lesa smo označili takrat, ko je bila lesna branika popolnoma oblikovana in je bil v vseh celicah proces diferenciacije zaključen. Obdobje maksimalne celične produkcije na lesni in floemski strani smo določili s pomočjo Gompertzove ali GAM funkcije (funkcije generaliziranih aditivnih modelov - Generalized additive models) (Gričar et al., 2021). Zabeležili smo datum prehoda iz ranega v kasni les in iz ranega v kasni floem in izračunali trajanje nastajanja ranega in kasnega ranega lesa ter ranega in kasnega floema.

Vzorce, ki so bili odvzeti konec rastne sezone in so imeli dokončno izoblikovane lesne in floemske branike, smo uporabili za izdelavo traheidogramov (za traheide v lesu) in floemogramov (za sitaste celice v floemu). To so grafi, ki prikazujejo variabilnost radialnih dimenzij celic in v primeru lesa tudi debelin celičnih sten znotraj branike. Meritve smo opravili v vsaj treh radialnih nizih. Povprečne vrednosti anatomskih spremenljivk za rani in kasni les ter rani in kasni floem smo izračunali ločeno za vsak niz celic. Ker je bilo število celic v radialnih nizih znotraj branik lesa ali floema različno, smo standardizirali velikost vzorca, da smo lahko opravili tudi primerjave med drevesi, območji in leti. Zato smo uporabili „relativni položaj“ vsake celice znotraj radialnega niza branike (Gričar et al., 2015). Za razlikovanje med treheidami ranega in kasnega lesa smo uporabili prvo interpretacijo, ki jo je predlagal Mork, po kateri imajo traheide kasnega lesa premer lumna, ki je manjši od dvakratne debeline celične stene (Denne, 1988). Datum prehoda iz ranega v kasni les smo izračunali s pomočjo predhodno izdelanih Gompertzovih oz. GAM funkcij (Gričar et al., 2021). Prehod iz ranega v kasni floem smo določili kot čas, ko smo na prečnih prerezih opazili prve celice aksialnega parenhima (Gričar & Čufar, 2008).

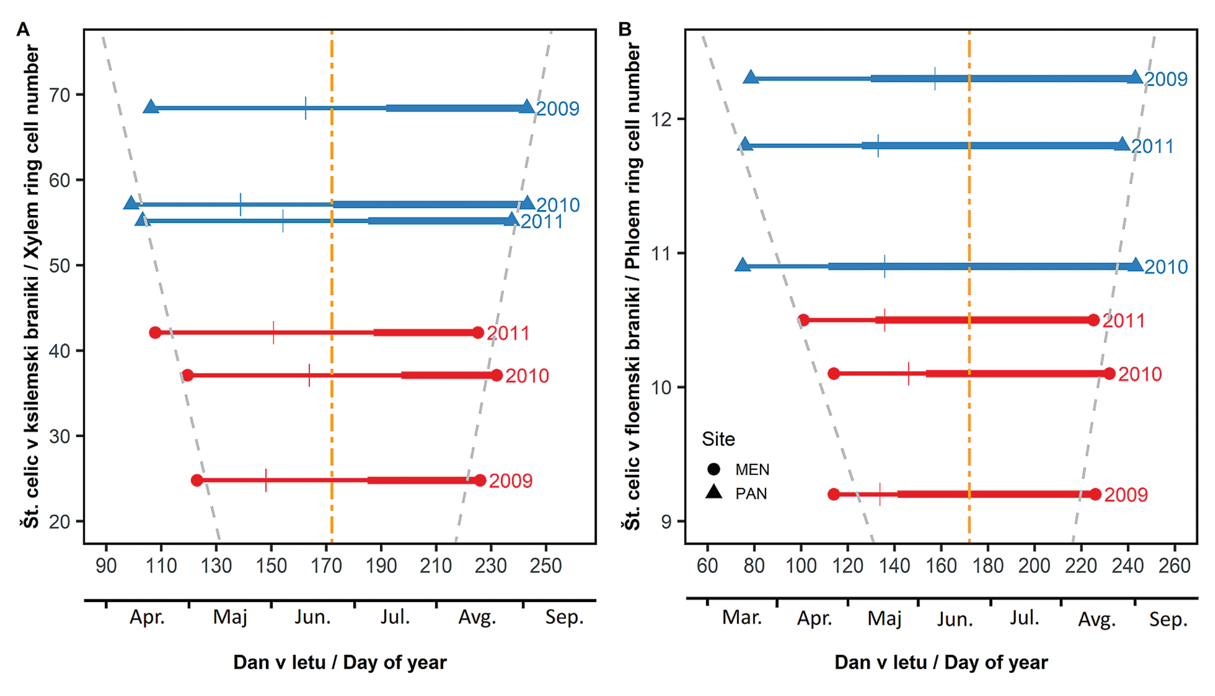
3 REZULTATI

3 RESULTS

3.1 KAMBIJEVA CELIČNA PRODUKCIJA IN DINAMIKA DEBELINSKE RASTI

3.1 CAMBIAL CELL PRODUCTION AND RADIAL GROWTH DYNAMICS

Fenologijo kambijeve celične produkcije smreke prikazujemo za leta 2009, 2010 in 2011 ločeno za posamezno rastišče PAN in MEN ter ločeno za ksilem (les) in floem (ličje) (slika 2). Prikazani rezultati predstavljajo letna povprečja 6 dreves na rastišču, pri čemer smo opazili tudi precejšnjo variabilnost



Slika 2. Zveza med fenologijo kambija (začetek, konec, trajanje) in številom celic v lesni in floemski braniki pri navadni smreki (Picea abies) *na rastiščih Panška reka (PAN) in Menina planina (MEN) v letih 2009, 2010 in 2011. Tanjši del horizontalnih linij označuje obdobje nastajanja ranega lesa oz. ranega floema, debelejši pa obdobje nastajanja kasnega lesa oz. kasnega floema. Vertikalne linije označujejo dan maksimalne celične produkcije. Rumena vertikalna črtkana črta označuje poletni solsticij. Sive črtkane črte predstavljajo zvezo med začetkom in koncem celične produkcije ter končnim številom celic v (a) lesni in (b) floemski braniki:*
število celic v lesni braniki = 1,39 · DOY *(začetek) + 200,11,* $r^2 = 0{,}62$, $P < 0{,}039$;
število celic v lesni braniki = 1,72 · DOY *(konec) - 355,65,* $r^2 = 0{,}71$, $P < 0{,}022$;
število celic v floemski braniki = *–0,051 · DOY (začetek) + 15,60,* $r^2 = 0{,}67$, $P < 0{,}029$;
število celic v floemski braniki = 0,107 · DOY *(konec) - 14,52,* $r^2 = 0{,}48$, $P < 0{,}076$.
(DOY = dan v letu).

Figure 2. Relationships between cambial phenology (onset, end, and duration) and the total number of xylem and phloem cells for Norway spruce (Picea abies) *Panška reka (PAN) in Menina planina (MEN) in 2009, 2010 and 2011. Different thickness of the horizontal lines represents periods of formation of early (thinner line) and late (thicker line) increment parts. Vertical bars indicate the dates of maximal cell production. Yellow vertical dot-dashed lines denote the summer solstice. Grey dashed lines show relationships between onset and cessation of cambial cell production and final (a) xylem and (b) phloem ring cell number:*
Xylem ring cell number = 1.39 · DOY (onset) + 200.11, $r^2 = 0.62$, $P < 0{,}039$;
Xylem ring cell number = 1.72 · DOY (end) – 355.65, $r^2 = 0.71$, $P < 0.022$;
Phloem ring cell number = –0.051 · DOY (onset) + 15.60, $r^2 = 0.67$, $P < 0.029$;
Phloem ring cell number = 0.107 · DOY (end) – 14.52, $r^2 = 0.48$, $P < 0.076$.
(DOY – day of the year).

med drevesi znotraj istega rastišča (Gričar et al., 2014, 2015). Ugotovili smo, da se je dinamika kambijeve celične produkcije med rastiščema na različnih nadmorskih višinah razlikovala. V splošnem je bilo trajanje kambijeve celične produkcije daljše na nižje ležečem rastišču PAN, in sicer zaradi zgodnejšega začetka in kasnejšega zaključka celičnih delitev, pri čemer je slednja faza bolj variirala med leti (slika 2). Razlike med rastiščema smo zabeležili tudi v dinamiki nastajanja lesnih in floemskih letnih prirastkov, kar se je odrazilo v njihovi širini in strukturi. Na istem rastišču so bile razlike med proučevanimi leti manj izrazite in statistično neznačilne.

Potrdili smo, da se je začetek in konec kambijeve celične produkcije lesnih in floemskih celic pri smreki začel sočasno, kar je značilnost iglavcev zmernega in hladnega pasu. Dinamika nastajanja lesne in floemske branike pa je različna (Gričar, 2017), zato v nadaljevanju podajamo rezultate ločeno za les in floem. V opazovanih letih smo na PAN začetek kambijeve celične produkcije zabeležili v prvi polovici aprila (dan 99–106), vrhunec produkcije lesnih celic med 19. 5. in 12. 6. in zaključek v drugi polovici avgusta (dan 238–243). Kasni les je začel nastajati po poletnem sončnem obratu (solsticiju), med 22. 6. in 11. 7. Kambij je proizvajal les 138,4 ± 14,3 dni (t. j. slabih 5 mesecev), od tega je 74–86 dni nastajal rani les ter 51–71 dni kasni les. V povprečju je tako rani les nastajal 23,8 % dlje časa. Lesna branika je bila popolnoma oblikovana v začetku oktobra (dan v letu 275,8 ± 13,3).

Na MEN smo vse fenološke faze, razen zaključka nastajanja lesa (t. j. zaključek kambijeve celične produkcije in diferenciacije zadnjih nastalih celic) zabeležili kasneje kot na PAN. Začetek kambijeve produkcije smo zabeležili med 18. 4. in 3. 5. (dan 108–123), maksimum med 19. 5. in 12. 6. in zaključek med 13. 8. in 20. 8. (dan 225–232). Kasni les je začel nastajati od 4. 7. do 16. 7. Kambij je proizvajal les 109,9 ± 16,3 dni (t. j. slabe 4 mesece), od tega je 62–79 dni nastajal rani les ter 35–41 dni kasni les. V povprečju je tako rani les nastajal 46.1 % dlje časa. Lesna branika je bila popolnoma oblikovana do konca septembra (dan v letu 264 ± 10,2).

Na floemski strani so se že pred začetkom kambijeve celične produkcije nediferencirane celice, ki so se nahajale na zunanjem robu kambija, začele oblikovati v sitaste celice ranega floema brez predhodnih delitev. Na PAN smo prve diferencirajoče celice floema zabeležili med 16. 3. in 20. 3. (dan 75–79), kar je 24–28 dni pred začetkom kambijeve celične produkcije. Kasni floem je začel nastajati med 22. 4. in 6. 5. (dan 112–130), preden je kambijeva produkcija floemskih celic dosegla maksimum med 13. 5. in 6. 6. (dan 133–157).

Na MEN so se vse faze nastajanja floema začele kasneje kot na PAN. Prve diferencirajoče celice floema smo zabeležili približno en mesec kasneje kot na PAN, med 11. 4. in 24. 4. (dan 101–114), kar je 6–9 dni pred začetkom kambijeve celične produkcije. Kasni floem je začel nastajati med 12. 5. in 2. 6. (dan 132–153), največjo produkcijo pa smo tudi na tem rastišču zabeležili v obdobju nastajanja kasnega lesa, in sicer med 14. 5. in 26. 5. (dan 134–146). Vsi omenjeni mejniki so na obeh rastiščih nastopili pred poletnim sončevim obratom.

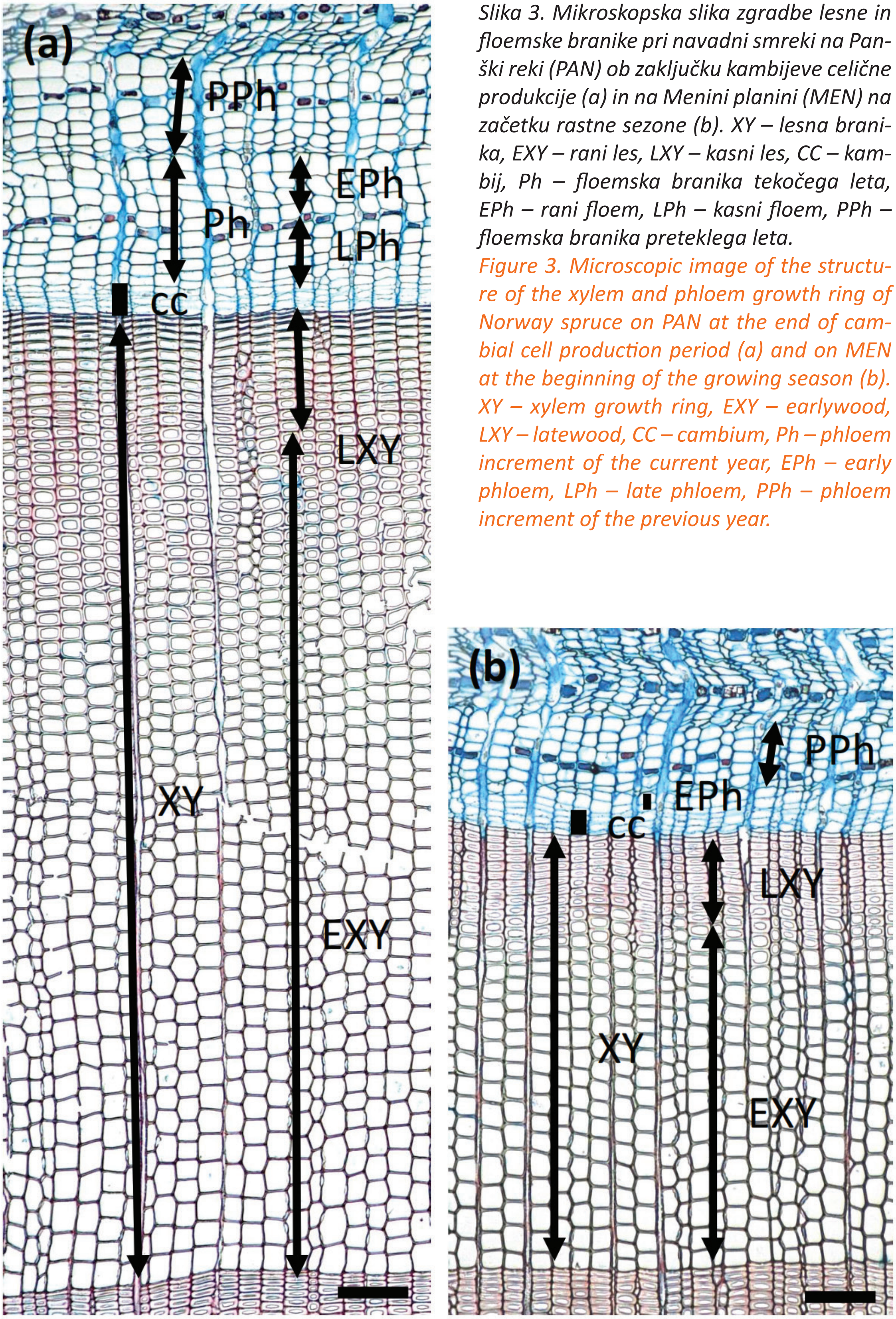
3.2 STRUKTURA LESNIH IN FLOEMSKIH PRIRASTKOV

3.2 STRUCTURE OF XYLEM AND PHLOEM INCREMENTS

Razlike med rastiščema v mejnikih in trajanju kambijeve produkcije so se odražale v širini in strukturi lesnih in floemskih prirastkov (slika 3, 4). Na PAN je bila povprečna lesna branika v obdobju 2009–2011 široka 60,2 ± 8,0 (srednja vrednost ± standardna napaka) slojev celic. Širina ranega lesa je znašala 30–31 celic, kasnega lesa pa 24–27 celic v radialnih nizih. Delež ranega lesa je bil v splošnem v vseh primerih nekoliko večji od kasnega lesa in je v povprečju v treh letih znašal 51,9 %. Gledano po posameznih letih pa smo v letu 2009 zabeležili največji delež kasnega lesa (54,6 %). Deleža ranega in kasnega lesa sta prikazana glede na število celic in ne glede na merjene širine prirastkov. Ker je radialna dimenzija traheid kasnega lesa približno 2–3-krat manjša od dimenzij traheid ranega lesa, bi bil delež kasnega lesa, preračunan glede na širino ranega in kasnega lesa v milimetrih, precej manjši.

Na MEN je bila povprečna lesna branika v lesu široka 37,0 ± 7,4 slojev celic. Širina ranega lesa je znašala 23–24 celic, kasnega lesa pa 9–18 slojev celic. Delež ranega lesa je bil na tem rastišču v vseh treh letih večji od kasnega lesa in je v povprečju znašal 64,6 %.

Na obeh rastiščih je bil floemski prirastek ožji v primerjavi z lesnim, in sicer na PAN za 81 %, na MEN pa za 73 % (slika 4, 5). V obdobju 2009–2011 je bila

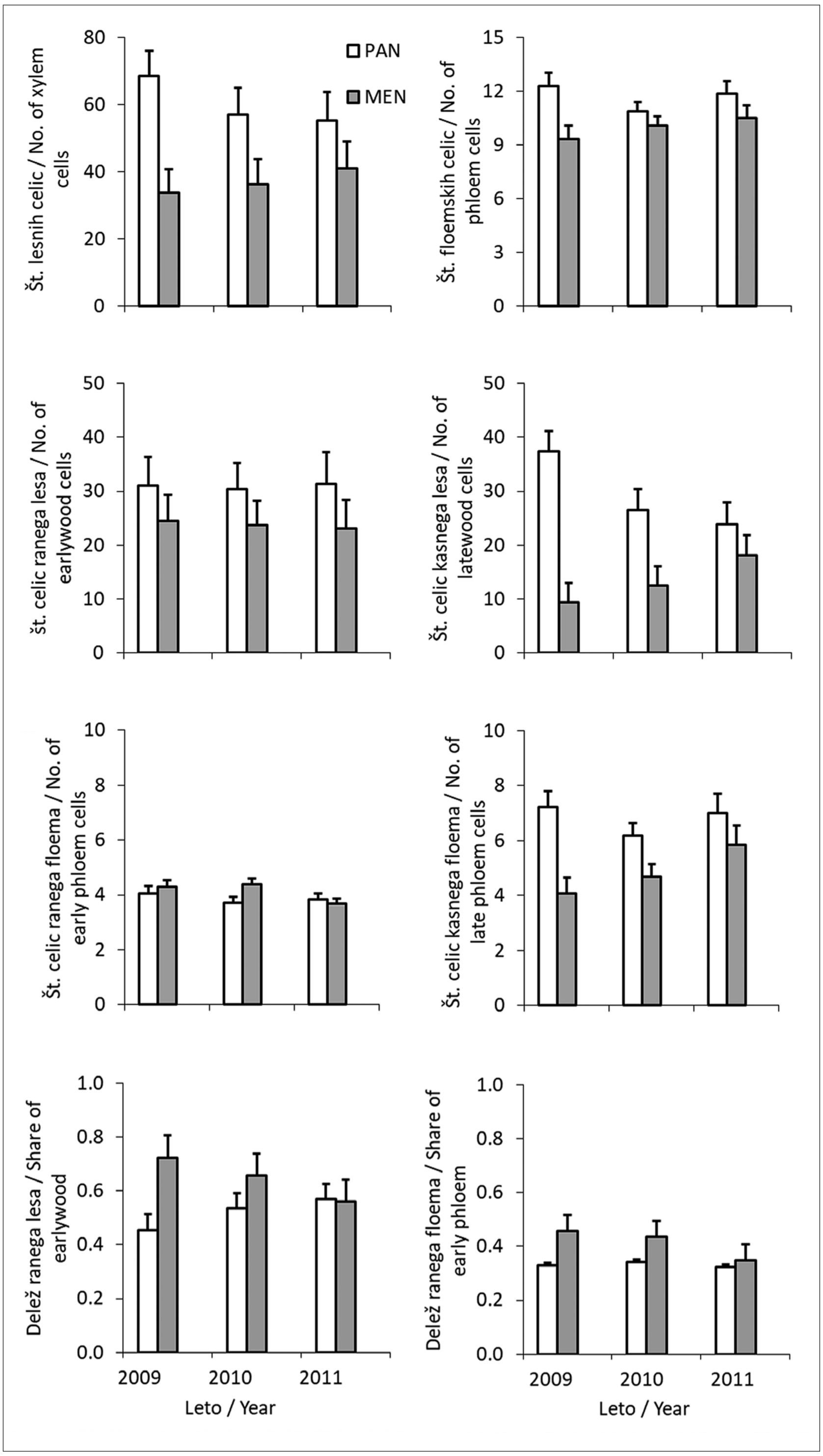


Slika 3. Mikroskopska slika zgradbe lesne in floemske branike pri navadni smreki na Panški reki (PAN) ob zaključku kambijeve celične produkcije (a) in na Menini planini (MEN) na začetku rastne sezone (b). XY – lesna branika, EXY – rani les, LXY – kasni les, CC – kambij, Ph – floemska branika tekočega leta, EPh – rani floem, LPh – kasni floem, PPh – floemska branika preteklega leta.

Figure 3. Microscopic image of the structure of the xylem and phloem growth ring of Norway spruce on PAN at the end of cambial cell production period (a) and on MEN at the beginning of the growing season (b). XY – xylem growth ring, EXY – earlywood, LXY – latewood, CC – cambium, Ph – phloem increment of the current year, EPh – early phloem, LPh – late phloem, PPh – phloem increment of the previous year.

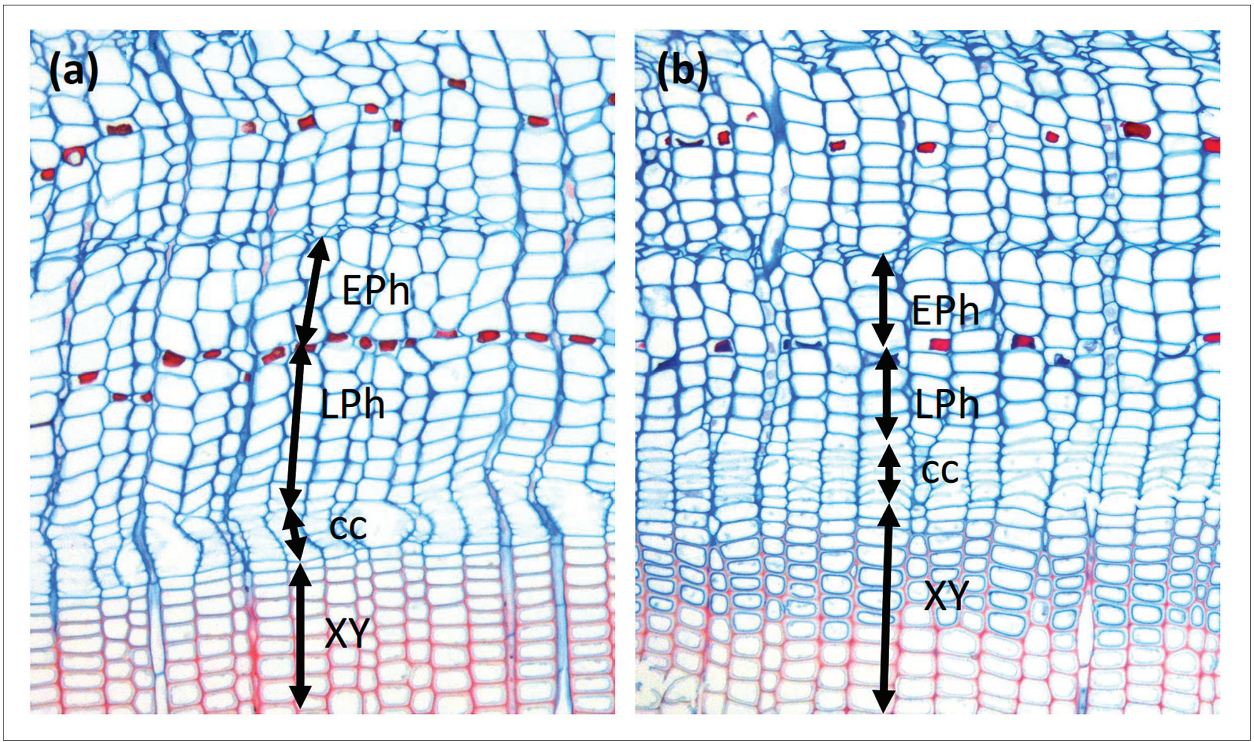
floemska branika na PAN v povprečju široka 11,7 ± 0,7 slojev celic, pri čemer je bil rani floem širok 3,9 ± 0,2 slojev celic, kasni floem pa 6,8 ± 0,6 slojev celic. Pri smreki na PAN je bil delež celic ranega floema v vseh primerih manjši od kasnega floema in je znašal 33,3 %.

Na MEN je bila floemska branika v proučevanih letih v povprečju široka 10,0 ± 0,7. Širina ranega floema je znašala okoli 4,1 ± 0,3 slojev celic, širina kasnega floema pa je znašala 4,9 ± 0,6 slojev. Na tem rastišču je znašal delež celic ranega floema v povprečju 45,7 %.



Slika 4. Širina in struktura lesnih in floemskih branik pri navadni smreki na Panški reki (PAN) in Menini planini (MEN) v obdobju 2009–2011. Delež ranega lesa in ranega floema je preračunan glede na število celic.

Figure 4. Width and structure of xylem and phloem growth rings in Norway spruce on Panška reka (PAN) and Menina planina (MEN) in the period 2009–2011. The proportion of earlywood and early phloem is calculated based on the number of cells.



Slika 5. Mikroskopska slika zgradbe floemske branike pri navadni smreki na PAN(a) in MEN (b). XY – les, CC – kambij, EPh – rani floem, LPh – kasni floem.

Figure 5. Microscopic image of the structure of the phloem growth ring of Norway spruce on PAN (a) and MEN (b). XY – xylem, CC – cambium, EPh – early phloem, LPh – late phloem.

4 RAZPRAVA

4 DISCUSSION

Naše raziskave kažejo, da rastiščne razmere v veliki meri vplivajo na sezonsko dinamiko nastajanja lesa in floema, kar se odraža v širini in strukturi prirastkov. Na višje ležečem rastišču MEN je bila rastna sezona približno mesec dni krajša, posledično so bili letni prirastki ožji, in sicer v lesu 39 % in v floemu 15 % kot na PAN. Poleg trajanja kambijeve celične produktivnosti je bila tudi dinamika debelinske rasti na proučevanih rastiščih različna. Za nastanek lesne branike sta bila na obeh rastiščih ključna meseca maj in junij, za nastanek floemske branike pa predvsem maj.

Stopnja celičnih delitev je bila na lesni strani na MEN večja kot na PAN (Gričar et al., 2014), kar pomeni, da je kambij pri smreki na višje ležečem rastišču v krajšem času proizvedel večje število lesnih celic. Stopnja celičnih delitev poleg trajanja kambijeve celične produkcije vpliva na končno širino branike (Skene, 1972). Rezultati so skladni z ugotovitvami drugih študij o prilagoditvi drevesih vrst danim okoljskim razmeram, kar kaže na njihovo veliko fleksibilnost in plastičnost (Gregory & Wilson, 1968; Alpert & Simms, 2002; Rossi et al., 2007). Gregory in Wilson (1968) sta ugotovila, da se je bela smreka (*Picea glauca*) na Aljaski s hitrejšimi delitvami kambijevih celic prilagodila na krajšo rastno sezono. Posledično so bile širine lesnih branik primerljive s širinami branik bele smreke, ki je rasla v Novi Angliji, kjer so bile razmere za rast bolj ugodne.

Prehod iz ranega v kasni les smo na PAN zabeležili le kakšen teden prej kot na MEN, in sicer v obdobju od konca junija do prve polovice julija. Čeprav so bili lesni prirastki širši na PAN, pa je bil delež celic ranega lesa večji na MEN, kar je v nasprotju s predhodnimi poročanji o pozitivni zvezi med širino branike in deležem ranega lesa (Dinwoodie, 1981). Na obeh rastiščih je bilo obdobje nastajanja ranega lesa daljše od nastajanja kasnega lesa, njuni deleži pa so se na rastiščih razlikovali, kar lahko pripišemo kombinaciji vpliva trajanja in stopnje kambijeve celične produkcije na širino prirastka, ki se tekom rastne sezone spreminja (Gričar et al., 2021). Na strukturo lesne branike in značilnosti celic ranega in kasnega lesa (t. j. velikost lumnov in debelina celične stene) pa vpliva še proces diferenciacije traheid (Cuny et al., 2014).

Kot že omenjeno, razlike v morfoloških značilnostih traheid ranega lesa v smislu dimenzij in debelin celičnih sten vplivajo na gostoto lesa. Srednja gostota absolutno suhega lesa smreke znaša 430 kg/m^3, z razponom od 300 do 640 kg/m^3 (Grosser & Teetz, 1985). To variiranje gostote pripisujemo različnim deležem kasnega lesa, ki pri iglavcih s širino branike praviloma pada, posledično pada tudi gostota (Dinwoodie, 1981). Zveze med širino branike ter deležem in strukturo kasnega lesa so kompleksne, saj številni notranji (genetika, hormoni) in zunanji (abiotski in biotski) dejavniki različno vplivajo na sezonsko dinamiko kambijeve celične produkcije in celično diferenciacijo (Larson, 1994; Fonti et al., 2013). Zaradi tega bo za boljše razumevanje zvez v prihodnje potrebno opraviti še več tovrstnih analiz pri različnih iglavcih iz različnih okolij. Ker je gostota v tesni zvezi z mehanskimi lastnostmi lesa (trdnost in trdota) in ima velik vpliv tudi na druge lastnosti in kakovost lesa, imajo takšne študije velik aplikativni pomen (Krajnc et al., 2020b).

Obdobje najintenzivnejše celične produkcije je nastopilo na floemski strani približno en mesec prej kot na strani lesa. Na strani lesa je bilo to obdobje vedno v času nastajanja ranega lesa, pri floemu pa je bilo to povezano s širino letnega prirastka. Pri branikah, *ožjih od 10 slojev celic*, je bilo obdobje najintenzivnejše rasti v času nastajanja ranega floema, v prirastkih, širših od 10 slojev celic, pa v času nastajanja kasnega floema. Na PAN je maksimum floemske rasti tako vedno zabeležen v obdobju nastajanja kasnega floema, pri smreki na MEN pa različno; v letih 2009 in 2010 v obdobju nastajanja ranega floema in v letu 2011 v obdobju nastajanja kasnega floema.

Na floemski strani sta bila trajanje in stopnja celične produkcije večja na PAN kot na MEN, kar se je odražalo v širših prirastkih. Čeprav se je kambijeva celična produkcija pri smreki začela in končala na lesni in floemski strani istočasno, se je dinamika nastanka obeh prevodnih tkiv razlikovala. Na obeh rastiščih so bili lesni prirastki širši od floemskih, kar je skladno s predhodnimi raziskavami (Gričar et al., 2009), da je v normalnih razmerah kambijeva celična produkcija na lesni strani intenzivnejša kot na floemski strani. V stresnih razmerah se lahko razmerje obrne in je floemski prirastek lahko širši od lesnega (Gričar et al., 2009); priraščanje lesa lahko lokalno celo izostane (pojav manjkajočih branik na deblu drevesa) (Novak et al., 2016). Floemska branika mora nastati vsako leto, kar je ključno za preživetje drevesa, saj sitaste celice po 1–2 letih delovanja odmrejo in kolabirajo, zato so za vzdrževanje prevodnega sistema v floemu potrebne nove celice (Esau, 1939).

Poleg različne dinamike celične produkcije, ki vpliva na širino prirastkov, smo ugotovili tudi različen vzorec nastajanja lesa in floema. Nastanek lesne branike se je začel s kambijevo produkcijo lesnih celic, pri floemu pa z diferenciacijo celic, ki so nastale z delitvami v kambiju v predhodni sezoni (slika 3b). Pred začetkom kambijeve celične produkcije so se torej nediferencirane celice, ki so se nahajale na zunanjem robu kambija, začele oblikovati v sitaste celice ranega floema brez predhodnih delitev, kar je v skladu z opažanji, ki sta jih objavila Alfieri in Evert (1973). Te celice, nastale v preteklem letu, so sestavljale inicialne celice ranega floema. Obdobje najintenzivnejše celične produkcije je nastopilo na floemski strani približno en mesec prej kot na strani lesa. Širina in struktura ranega floema je bila pri smreki na obeh rastiščih primerljiva in manj variabilna v primerjavi s kasnim floemom. Rani floem je bil sestavljen iz 3–4 slojev sitastih celic z velikimi lumni. Prehod iz ranega v kasni floem je označeval bolj ali manj sklenjen pas aksialnega parenhima. Širina kasnega floema je bila večja na PAN. Sitaste celice kasnega floema so imele manjše lumne, prisoten je bil aksialni parenhim. Različna struktura ranega in kasnega floema podpira predhodne ugotovitve, da je njuna vloga v drevesu različna (Gričar et al., 2015). Razlike lahko pripišemo veliki plastičnosti debelinske rasti smreke na proučevanih rastiščih, kjer so razmere za njeno rast ugodne.

5 SKLEPI

5 CONCLUSIONS

Spremljanje sezonske dinamike nastanka lesa in podrobne lesno-anatomske analize je zelo primerno za oceno odziva in prilagoditve debelinske rasti dreves na okoljske razmere. Naše dolgoletne raziskave potrjujejo, da informacije o nastanku in strukturi floema pomembno dopolnjujejo znanja na tem področju. V drevesu sta osnovni funkciji lesa in floema povsem različni, vendar sta obe tkivi ključni za rast, razvoj in preživetje drevesa. Ker sta les in floem povezana preko trakov (Spicer, 2014), nekateri avtorji predlagajo, da bi se ju obravnavalo kot enoten vaskularni sistem (Pfautsch et al., 2015). Navkljub številnim raziskavam o debelinski rasti dreves pa so podatki o času prehoda iz ranega v kasni les in še posebej iz ranega v kasni floem zelo redki, čeprav predstavljajo pomemben mejnik v razumevanju vplivov variabilnosti fenologije kambija in celične diferenciacije na strukturo in lastnosti lesa. Nova spoznanja, prikazana v pričujoči študiji, so pomembna z vidika gostote lesa, ki v veliki meri vpliva na kakovost lesa, kar je zanimivo za različne deležnike gozdno-lesne verige.

6 POVZETEK
6 SUMMARY

Norway spruce (*Picea abies* (L.) Karst.) is currently the most economically important tree species in Slovenia, accounting for 30.4% of the wood stock in 2019. Increasingly frequent extreme events, mostly related to climate change, have seriously threatened its resilience and survival in recent years, especially on sites at lower altitudes. Information on the temporal dynamics of wood and phloem formation can serve as indicators of tree response and adaptation to site conditions and extreme events.

In Slovenia, we have established long-term data on seasonal activity of the cambium and wood and phloem formation at two sites, Panška reka (PAN – 400 m a.s.l.) and Menina planina (MEN – 1200 m a.s.l.) for Norway spruce (Gričar et al., 2014, 2015, 2021) and European beech (*Fagus sylvatica* L.) (e.g. Prislan et al., 2013, 2019). These studies were conducted according to an internationally harmonized methodology and the results were included in international databases, which made it possible to answer important research questions on tree growth at a global scale (e.g. Rossi et al., 2008; Cuny et al., 2015; Martinez del Castillo et al., 2018; Huang et al., 2020). Despite numerous studies on wood formation, data on the timing of the transition from early to latewood and especially from early to late phloem are very scarce.

The purpose of this article is to present some of the latest results on the growth of spruce from the two sites during three years in Slovenia. The study included analyses of: (a) production of cambium cells, with the beginning, end, duration and rate of production of wood and phloem cells; (b) the transition from early to latewood and from early to late phloem, which is generally poorly known; and (c) structure of wood and phloem formed in each year.

On two typical forest sites, PAN (400 m a.s.l.) and MEN (1200 m a.s.l.), six dominant or co-dominant Norway spruce trees were selected for sampling. In the years 2009, 2010 and 2011, microcores of 1.8 mm in diameter were taken with the Trephor tool from the tree stems at weekly intervals during the growing season (i.e., mid-March to mid-October). The microcores were used to cut cross-sections of tissues prepared for observation and measurements under the microscope according to the established methodology proposed by Gričar et al. (2014). The beginning, end, and duration of cambium cell production of wood and phloem annual increments were determined as described by Gričar et al. (2014). On all cross sections, we counted the number of cells in the forming wood and phloem along at least three radial rows and calculated the Gompertz function based on the data for each tree to describe the seasonal dynamics of wood and phloem formation. The period of maximum cell production of wood and phloem was determined using the Gompertz or GAM function (Gričar et al., 2021). We recorded the date of transition from early to latewood and from early to late phloem, and calculated the duration of formation of early and latewood and early and late phloem. Samples collected at the end of the growing season with fully formed current growth rings in wood and phloem were used to generate tracheidograms (for tracheids in wood) and phloemograms (for sieve cells in phloem). Mork's rule was used to distinguish between early and latewood tracheids (Denne, 1928). The timing of the transition from early to latewood was calculated using previously constructed Gompertz or GAM functions (Gričar et al., 2021). The transition from early to late phloem was defined as the time when the first cells of the axial parenchyma were observed on the cross sections (Gričar & Čufar, 2008).

In the study years 2009, 2010 and 2011, the beginning of cambium cell production on the PAN was recorded in the first half of April (DOY = day

of the year 99-106), the maximum wood cell production between 19 May and 12 June, and the end in the second half of August (DOY 238-243). Latewood formation began after the summer solstice, between 22 June and 11 July. The cambium produced wood for 138.4 ± 14.3 days, of which the duration of earlywood formation was 74-86 days and latewood 51-71 days. The annual rings in the wood were fully formed at the beginning of October. At MEN, all phenological phases except the completion of wood formation (i.e., the completion of cambium cell production and differentiation of the last formed cells) were recorded later than at PAN. The onset of cambium production was recorded between 18 April and 3 May (DOY 108-123), maximum production between 19 May and 12 June, and termination between 13 and 20 August (DOY 225-232). Latewood production began between 4 and 16 July. The cambium produced wood for 109.9 ± 16.3 days, of which earlywood production lasted 62-79 days and latewood 35-41 days.

On the phloem side, undifferentiated cells located at the outer edge of the cambium began to differentiate into sieve cells of the early phloem without prior division and before the onset of cambium cell production. The first phloem differentiating cells were recorded at PAN between 16 and 20 March (DOY 75-79), 24-28 days before the onset of cambium cell production. Late phloem began to form between 22 April and 6 May (DOY 112-130), before cambium phloem cell production peaked between 13 May and 6 June (DOY 133-157). At MEN, all phases of phloem formation began later than at PAN. The first differentiating phloem cells were recorded about a month later than at PAN, between 11 and 24 April (DOY 101-114), 6-9 days before the onset of cambium cell production. Late phloem began to form between 12 May and 2 June (DOY 132-153), and maximum production was recorded at this site during the period of late phloem formation, between 14 and 26 May (DOY 134-146). Maximum cell production and the transition from early to late phloem occurred at both sites before the summer solstice.

May and June were critical months for wood and phloem increment formation. Differences between sites in milestones and duration of cambium production were reflected in the width and structure of wood and phloem annual rings (Figure 3, 4). At PAN, the average wood increment in 2009-2011 was 60.2 ± 8.0 cell layers wide. Earlywood width was 30-31 cells and latewood width was 24-27 cells per radial row. At MEN, the mean wood increment was 37.0 ± 7.4 cell layers wide. The width of the earlywood was 23-24 cells, and that of the latewood was 9-18 cell layers. In both sites, the phloem increment was narrower than the wood increment (Figs. 4, 5). In 2009-2011, the phloem increment at PAN was on average 10.7 ± 0.7 cell layers wide, with the early phloem consisting of 3.9 ± 0.2 and the late phloem consisting of 6.8 ± 0.6 cells.

The presented study brings new information on the onset and duration of early and latewood formation, as well as early and late phloem, and in this way complements previous studies on wood formation at the same sites. It also places the formation of early and latewood and phloem in the broader framework of cambium production, differentiation and wood and phloem quality.

Furthermore, the cell structure and ratios of early and latewood, which significantly influence wood density, are presented. Density is one of the main indicators of wood properties; therefore the results are important for wood quality assessment, which is interesting for stakeholders in the forest-wood chain. Importantly, the study also provides new insights into the early and late phloem, which has been particularly poorly studied.

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ANALIZA RAZKROJENEGA SMREKOVEGA LESA, ZAŠČITENEGA Z BIOCIDNIM PROIZVODOM CCB, PO 14 LETIH IZPOSTAVITVE NA PROSTEM

ANALYSIS OF DECAYED NORWAY SPRUCE WOOD IMPREGNATED WITH CCB AFTER 14 YEARS OF OUTDOOR EXPOSURE

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Izvleček / Abstract

Izvleček: *Les na prostem je izpostavljen delovanju abiotskih in biotskih dejavnikov. Če hočemo njihovo delovanje upočasniti, moramo les zaščititi. V preteklosti je bil biocidni proizvod na osnovi bakrovih, kromovih in borovih spojin (CCB) ena najpomembnejših rešitev za zaščito lesa v ostrih pogojih izpostavitve. Kljub temu, da se CCB v EU praktično ne uporablja več, lahko služi kot referenca za vrednotenje novih biocidnih proizvodov. Na terenskem polju Oddelka za lesarstvo Biotehniške fakultete že 14 let poteka poskus v realnih pogojih, kjer so impregnirani vzorci izpostavljeni vremenskim vplivom v skladu z dvoslojnim testom. Pri zaščitenem lesu pogosto opažamo, da les propade hitreje kot je pričakovano. V okviru tega prispevka želimo na podlagi analize razkrojenega s CCB impregniranega lesa s terenskega polja Oddelka za lesarstvo ugotoviti, zakaj je prišlo do prezgodnjega razkroja. Rezultati kažejo, da ustrezno življenjsko dobo zagotavljata ustrezna retencija in penetracija aktivnih učinkovin v les.*

Ključne besede: *les, zaščita lesa, CCB, impregnacija, razkroj, lesne glive*

Abstract: *Wood in outdoor applications is exposed to abiotic and biotic factors. If we want to slow down the decay, the wood must be protected. In the past, biocidal products based on copper, chromium, and boron compounds (CCB) were one of the most important solutions for wood protection under extreme conditions. Although CCB is in practice no longer used in the EU, it can serve as a reference for the evaluation of new biocidal products. At the field test site of the Department of Wood Science and Technology, Biotechnical Faculty, an experiment has been carried out under real conditions for 14 years, in which impregnated samples are exposed to the weather according to a double-layer test. In the case of treated wood, we often find that the wood decays faster than expected. In this work we want to determine what contributes to decay based on the analysis of decayed impregnated wood from the field test site. The results show that sufficient retention and penetration of the active substances into the wood ensures the planned service life.*

Keywords: *wood, wood protection, CCB, impregnation, decay, wood inhabiting fungi*

1 INTRODUCTION

1 UVOD

Les na prostem je izpostavljen delovanju biotskih in abiotskih dejavnikov. V našem podnebnem pasu glive sodijo med najpomembnejše vzroke za propadanje lesa na prostem. Na lesu iglavcev se najpogosteje pojavita glivi tramovka (*Gloeophyllum* sp.) in bela hišna goba (*Fibroporia* sp.) (Schmidt, 2006). Če neodporen les ni zaščiten, se razkroj v našem podnebnem pasu pojavi že po prvem ali najkasneje drugem letu izpostavitve (Humar et al., 2019a). V naravi so razkrojni procesi zaželeni, ko les uporabljamo v gospodarske namene, želimo njegov razkroj upočasniti. V preteklosti smo v ta namen praviloma uporabljali le biocidne proizvode (Preston, 2000). Za zaščito lesa v bolj izpostavljenih pogojih se v EU in ZDA najpogosteje uporabljajo biocidni proizvodi na osnovi bakrovih spojin (Freeman & Mcintyre, 2008).

Bakrovi pripravki ostajajo ena izmed najpomembnejših sestavin biocidnih proizvodov za zaščito lesa tudi po implementaciji evropske zakonodaje s področja biocidov (EC, 2000). Glavni razlogi za uporabo bakrovih spojin so dobra učinkovitost, nizka toksičnost za neciljne organizme, ugodno razmerje med kakovostjo in ceno in veliko povpraševanje po poceni impreg-

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niranem lesu (Humar, 2002). Poleg tega njihovo uporabnost povečuje dejstvo, da so v EU poleg kreozotnega olja edino bakrovi zaščitni pripravki primerni za zaščito lesa v četrtem razredu uporabe (les v stiku z zemljo) (Humar et al., 2018). Bakrove učinkovine se za zaščito lesa ne uporabljajo samostojno, ker se iz lesa izpirajo (Humar et al., 2007) in nimajo insekticidnih lastnosti (Mbitnkeu Fetnga Tchebe et al., 2020). V preteklosti so bakrovim pripravkom dodajali kromove spojine za izboljšanje vezave v les, tako da je še danes v uporabi relativno veliko lesa, impregniranega s pripravki na osnovi bakrovih in kromovih spojin (Eaton & Hale, 1993; Richardson, 1993; Freeman & Mcintyre, 2008). Danes biocidnih proizvodov na osnovi bakrovih in kromovih spojin v EU skoraj ne uporabljamo več. Glavni razlog je šestvalentni krom, ki je škodljiv za okolje in živa bitja (Humar et al., 2006). Kljub temu, da so bakrovi pripravki na trgu že več desetletij, mehanizem vezave teh pripravkov v les še ni v celoti pojasnjen. Poleg tega ne vemo, zakaj s temi pripravki impregniran les občasno propade hitreje kot smo načrtovali (Ribera et al., 2017). Ali so temu vzrok na bakrove pripravke tolerantne glive, ali so tolerantne glive okužile les po tem, ko se je iz njega izprala velika večina aktivnih učinkovin? Toleranca gliv razkrojevalk na baker je povezana z izločanjem oksalne kisline, ki jo izločajo glive razkrojevalke (Takao, 1965). Oksalna kislina ima močno afiniteto na tvorbo kompleksov z bakrovimi spojinami. Bakrov oksalat je v vodi zelo slabo topen in zato za glive praktično nestrupen. Med glivami razkrojevalkami se toleranca najpogosteje pojavlja pri sivi hišni gobi (*Serpula lacrymans*), beli hišni gobi (*Fibroporia vaillantii*) in drugih glivah tega rodu (Steenkjær Hastrup et al., 2005; Liew & Schilling, 2012; Karunasekera et al., 2017).

Namen tega prispevka je raziskati lastnosti razkrojenega lesa in izpiranje bakrovih spojin iz lesa v tretjem razredu uporabe. Kljub temu da pripravkov na osnovi bakra in kroma (CCB in CCA) v Sloveniji skoraj ne uporabljamo več, so ti podatki zelo pomembni za načrtovanje življenjske dobe lesa na prostem in za razvoj novih biocidnih proizvodov na osnovi bakra.

2 MATERIALI IN METODE

2 MATERIALS AND METHODS

2.1 MATERIALI IN IZPOSTAVITEV VZORCEV

2.1 MATERIALS AND EXPOSURE

Smrekove (*Picea abies*) vzorce dimenzij 2,5 cm × 5,0 cm × 50 cm smo pred impregnacijo tri tedne uravnovešali pri 20 °C in 65-odstotni relativni zračni vlažnosti (RH). Vzorci so bili polradialni, branike so z vzdolžno površino tvorile kot 45° ± 15°. Za impregnacijo smo uporabili biocidni proizvod CCB (Silvanol GBP, Silvaprodukt), na osnovi bakrovega(II) sulfata, kalijevega dikromata(VI) in borove kisline (Richardson, 1993). Koncentracija aktivnih učinkovin v pripravku je ustrezala zahtevam za rabo v tretjem razredu uporabe (CEN, 2013). Predpisan suh navzem učinkovin biocidnega proizvoda CCB za uporabo v tretjem razredu uporabe je 4 kg/m^3 (Willeitner, 2001). Vzorce smo impregnirali v skladu s postopkom polnih celic. Postopek je sestavljen iz treh stopenj: 1 h pri tlaku -0,02 MPa, 2 h pri tlaku 1 MPa in 2 h namakanja pri normalnem tlaku. Po impregnaciji smo vzorcem gravimetrično določili mokri navzem in jih štiri tedne počasi sušili ter s tem omogočili redukcijo kroma iz Cr(VI) v Cr(III). Za primerjavo smo uporabili neimpregnirane smrekove vzorce (kontrola).

Vzorce smo izpostavili vremenskim vplivom 7. 4. 2006 na terenskem polju Oddelka za lesarstvo v Rožni dolini v Ljubljani na pretežno senčni in zatišni legi (310 m n.m.). Izpostavljeni so bili v tretjem razredu izpostavitve (nepokrito na prostem nad tlemi, pogosto močenje) (CEN, 2013). Za določanje odpornosti lesa smo v raziskavi uporabili dvoslojni test (ang. double layer test) (Rapp & Augusta, 2004; CEN, 2015). Pet enako obdelanih vzorcev smo zložili v spodnjo in pet v zgornjo vrsto. Vzorci v zgornji vrsti so bili za polovico vzorca zamaknjeni. Na ta način smo ustvarili vodno past, kjer je zastajala voda. S tem smo pospešili glivni razkroj (slika 1).

Ocenjevanje vzorcev je potekalo vsako leto med petnajstim majem in petnajstim junijem. Vsak vzorec smo si natančno ogledali in ocenili stopnjo razkroja po standardu SIST EN 252 (CEN, 2015) (preglednica 1). Po 14 letih izpostavitve je propadel prvi zaščiten vzorec, ki smo ga podrobneje raziskali, da bi določili vzrok za razkroj. Vzorec smo prežagali na 12 mestih in z optičnim čitalcem preslikali preseke.

Slika 1. Dvoslojni test

Figure 1. Double-layer test

Preglednica 1. Ocene razkroja vzorcev (CEN, 2015).

Table 1. Decay ratings of samples (CEN, 2015).

Ocena / **Rating**	**Razvrstitev /** **Classification**	**Opis preizkušanca /** **Definition of condition**
0	Ni znakov razkroja	Na preizkušancu ni zaznavnih sprememb.
1	Neznaten razkroj	Na vzorcu so vidni znaki razkroja, vendar razkroj ni intenziven in je zelo prostorsko omejen: - Spremembe, ki se pokažejo predvsem kot sprememba barve ali zelo površinski razkroj, mehčanje lesa je najpogostejši kazalec, razkroj sega do 1 mm v globino.
2	Zmeren razkroj	Jasne spremembe v zmernem obsegu: - Spremembe, ki se kažejo kot mehčanje lesa 1 mm do 3 mm globoko na 1 cm^2 ali večjem delu vzorca.
3	Močen razkroj	Velike spremembe: - Izrazit razkroj lesa 3 mm do 5 mm globoko na velikem delu površine (večje od 20 cm^2), ali mehčanje lesa globlje kot 10 mm na površini, večji od 1 cm^2.
4	Propad	Preizkušanec je močno razkrojen: - Ob padcu z višine 0,5 m se zlomi.

2.2 ELEMENTNA ANALIZA
2.2 ELEMENTAL ANALYSIS

Lesene vzorce smo z dletom in krožnim žagalnim strojem razdelili v tri sloje in odstranili čela (2,5 cm od roba). Posebej smo ločili čela, ki jih je mogoče lažje impregnirati, ter zgornji, srednji in spodnji sloj. V nadaljevanju smo vzorce posameznih plasti zmleli (velikost sita = 1 mm) z rezalnim mlinom (Retsch SM 2000, Haan, Nemčija). Iz zmletega lesa smo s stiskalnico (Chemplex, Palm City, FL, Združene države Amerike) izdelali vsaj pet tablet. Te tablete smo uporabili za elementno analizo, ki smo jo izvedli z rentgenskim fluorescenčnim spektrometrom (TwinX, Oxford instruments, Velika Britanija) in določili delež Cu. Vse meritve smo izvedli s PIN detektorjem (U = 26 kV, I = 115 µA, t = 300 s).

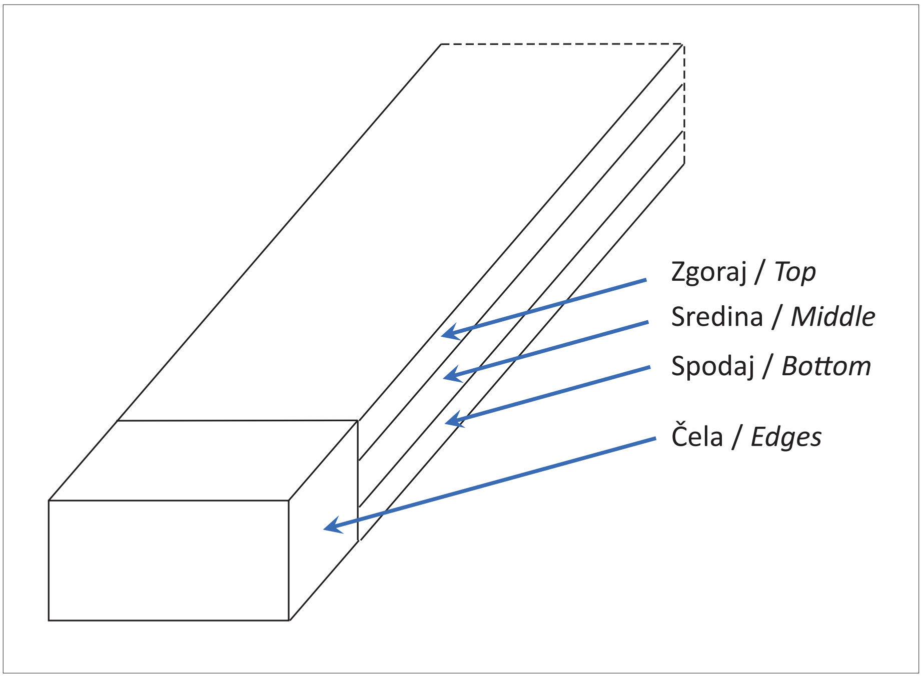
2.3 MIKROSKOPSKA ANALIZA
2.3 MICROSCOPIC ANALYSIS

Na razkrojenem impregniranem vzorcu smo opravili tudi morfološko in mikroskopsko analizo. Za morfološko analizo smo uporabili laserski konfokalni vrstični mikroskop (Olympus, Lext OLS 5000). Ta tehnika ne zahteva posebne priprave, zato je še posebej primerna za preiskave vlažnega in trhlega lesa (Humar et al., 2019b; Žigon et al., 2020). Analizo smo izvedli na zgornji strani vzorca, na najmanj razkrojenem delu. Del vzorcev je bil analiziran tudi s klasično svetlobno mikroskopijo. Vzorce smo obrezali in jih vklopili v parafin. Pripravo rezin smo izvedli z rotacijskim mikrotomom (Leica, RM2245). Rezine so bile obarvane z barviloma safranin in astra-modro. Rezine so bile vklopljene v Euparal (Prislan et al., 2008).

3 REZULTATI IN RAZPRAVA
3 RESULTS AND DISCUSSION

Gostota lesa je osnovni indikator, ki posredno nakazuje na nekatere ključne lastnosti lesa. Povprečna gostota (r_{12}) vremenskim vplivom izpostavljenih impregniranih in neimpregniranih vzorcev se med seboj značilno ne razlikuje (slika 3). Gostota lesa, impregniranega s CCB, je bila 488 kg/m^3, gostota kontrolnih vzorcev 490 kg/m^3. Te vrednosti so povsem v skladu z literaturnimi podatki za smrekovino (Wagenfuhr, 2007), kar nakazuje, da je bil za raziskavo uporabljen reprezentativen material.

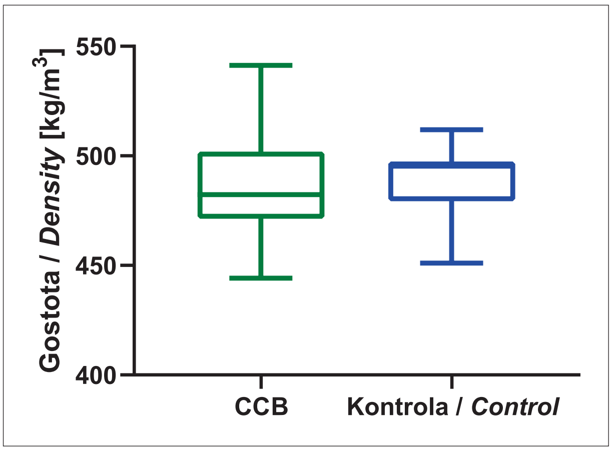
Prve znake razkroja na nezaščiteni smrekovini smo opazili že po prvem letu izpostavitve. Šibek razkroj se je pojavil na dveh od desetih izpostavljenih vzorcev. V nadaljevanju je razkroj počasi napredoval. Prvi kontrolni vzorec je propadel po štirih letih, polovica vzorcev je propadla po šestih, vsi kontrolni vzorci so propadli po osmih letih izpostavitve (slika 4A). To obdobje je relativno dolgo. Neimpregnirani smrekovi vzorci na terenskem polju v Ljubljani



Slika 2. Prikaz vzorčenja z XRF analizo

Figure 2. Sketch of the sampling for XRF analysis

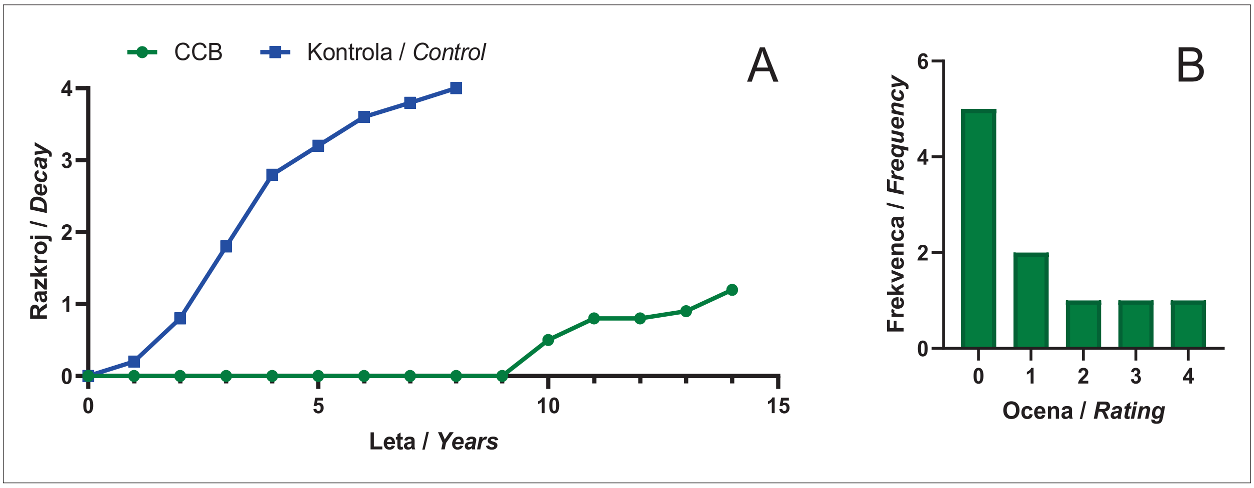
navadno propadejo hitreje in sicer po 4 do 6 letih izpostavitve (Humar et al., 2019a; Humar et al., 2020). Impregnacija s CCB je bistveno upočasnila razkroj. Prvi znaki razkroja so se na smrekovini, impregnirani s CCB, pojavili po 10 letih testiranja. Razkroj je v povprečju potem počasi napredoval s povprečne ocene 0,3 po desetih letih na 1,1 po 14 letih izpostavitve (slika 4A). Kot je razvidno iz porazdelitve ocen, je po 14 letih delovanja biotskih in abiotskih dejavnikov potek razkroja relativno nehomogen. Vzorci izpostavljeni v spodnji vrsti, so ostali nerazkrojeni, medtem ko smo intenziven razkroj opazili predvsem na vzorcih v zgornjem sloju (slika 4B).



Slika 3. Gostota zračno suhega impregniranega (CCB) in neimpregniranega (Kontrola) smrekovega lesa
Figure 3. Density of air dry impregnated (CCB) and non-impregnated (Control) wood

Dvoslojni test je zasnovan tako, da med vzorci zastaja voda, zato smo pričakovali, da se bo razkroj najprej razvil med obema slojema vzorcev. V nasprotju s pričakovanji je analiza preseka impregniranega vzorca pokazala, da se je razkroj impregniranih vzorcev pričel z zgornje strani (slika 5). Očitno je, da so se na zgornji strani pojavile razpoke. V razpokah je zastajala voda in s tem so se vzpostavili ugodni pogoji za razvoj gliv (slika 8). Hrapavost izpostavljene površine (Sa) znaša kar 500 µm, hrapavost referenčne, vremenskim vplivom neizpostavljene impregnirane smrekovine znaša le 20 µm.

Na podlagi barve razkrojenega lesa sklepamo, da so površino lesa razkrojile glive rjave trohnobe. Po 14 letih razkroja je bila površina močno razpokana, kar verjetno vodi v spiralo propada, v razpokah zastaja voda, zato so pogoji za razkroj ugodnejši skozi daljše časovno obdobje kot pri nerazpokanih vzorcih. Profil površine na najmanj razkrojenem delu vzorca je razviden na spodnji sliki (slika 6). Poleg tega se je na površini smrekovine, impregnirane s CCB, razvil intenziven biofilm (slika 7). Biofilm, pritrjen na podlago, je skupek mikroorganizmov in njihovih zunajceličnih izločkov, ki

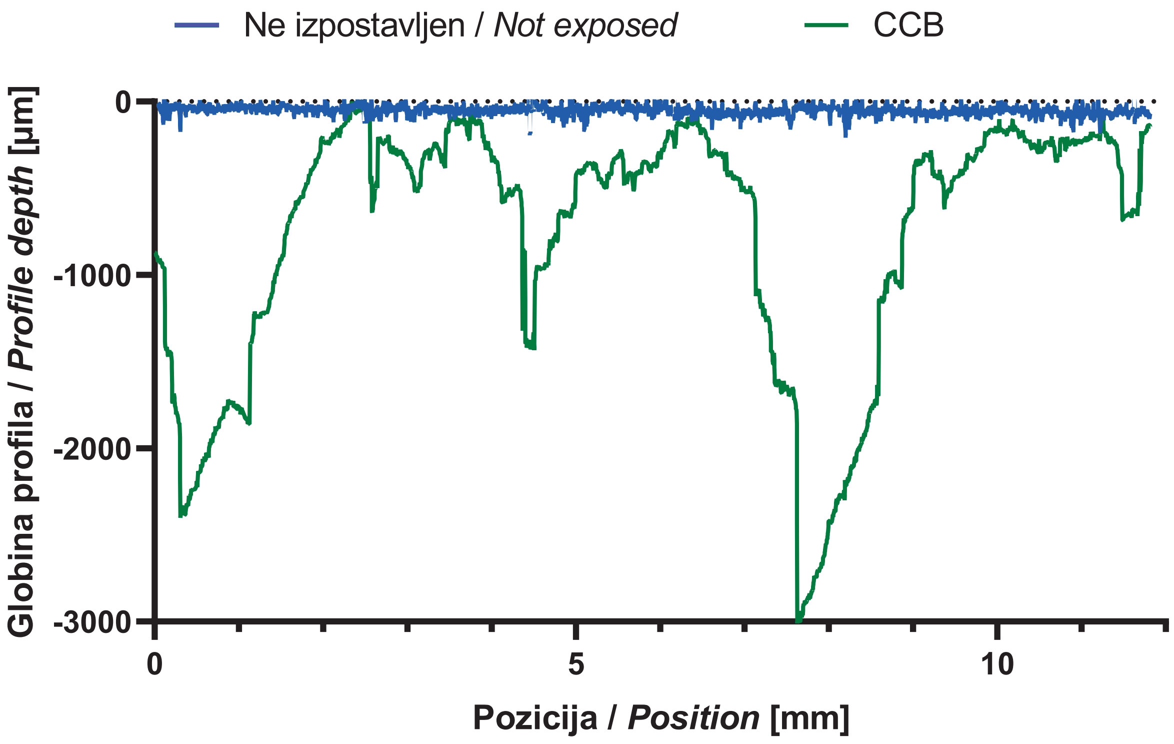


Slika 4. (A) Potek razkroja na impregniranih (CCB) in neimpregniranih (Kontrola) vzorcih smrekovega lesa; (B) Distribucija ocen razkroja vzorcev, impregniranih z biocidnim proizvodom CCB, po 14 letih izpostavitve na prostem
Figure 4. (A) Decay development on impregnated (CCB) and non-impregnated (Control) Norway spruce wood samples; (B) Distribution of decay ratings of wood impregnated with CCB preservative solution after 14 years of outdoor exposure

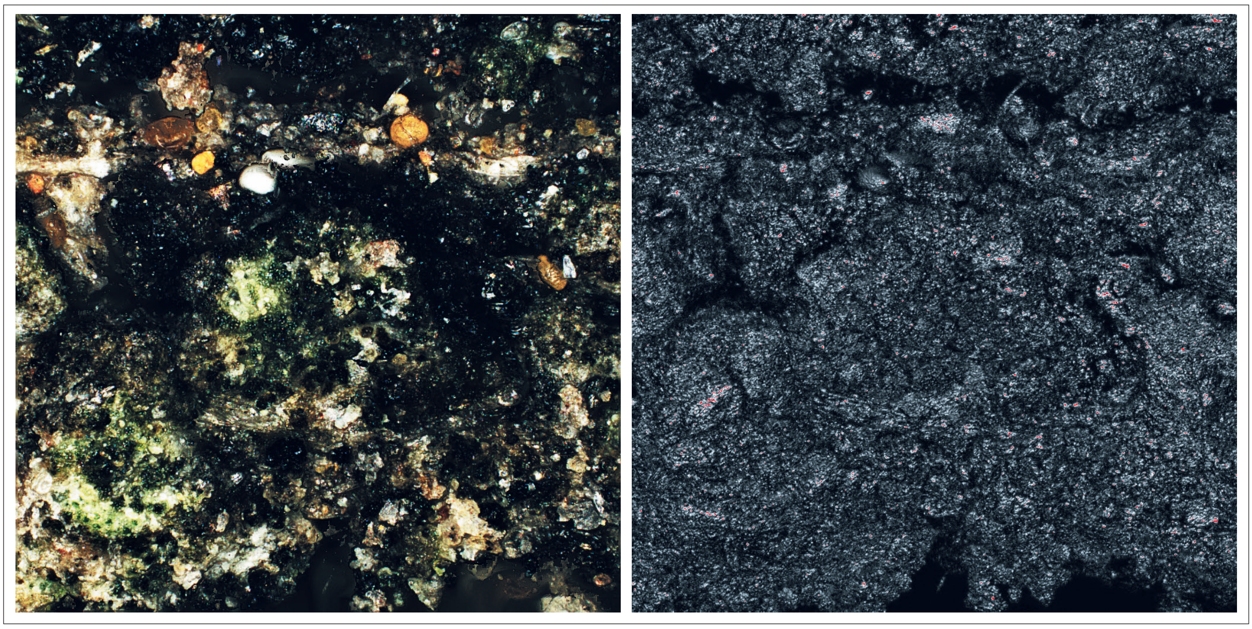
delujejo kot povezovalni člen. Na podlagi CLSM analize sklepamo, da so se na površini razvile glive modrivke, plesni in alge. Ta pojav je značilen za les, izpostavljen na prostem. Najpogostejša gliva (črna kvasovka) na površini lesa je *Aureobasidium pullulans* (Sailer et al., 2010). Biofilmi na prostem ne nastajajo le na lesu, temveč tudi na drugih materialih, kot so beton ali polimerni materiali (Miao et al., 2019) in služijo kot substrat za kolonizacijo mikroorganizmov, ki tvorijo biofilme. Različne mikrobne združbe med mikroplastiko in obdajajočo vodo so bile dobro dokumentirane, kljub temu pa ni dovolj znanja o kolonizaciji plastičnih in neplastičnih substratov, kljub dejstvu, da se mikrobne združbe običajno pojavljajo na naravnih trdnih površinah. Biofilm pripomore k zadrževanju vode in ustvarjanju ugodnih pogojev za glivni razkroj. Biofilm na lesu, impregniranem s CCB, je tako debel, da pod biofilmom lesa sploh ni opaziti (slika 7).

Slika 5. Presek vzorca, impregniranega z biocidnim proizvodom CCB po 14 letih izpostavitve na prostem
Figure 5. Several cross-sections of wood impregnated with CCB preservative solution after 14 years of outdoor exposure

Analiza lesa s svetlobno mikroskopijo je pokazala, da je na lesu opaziti razkroj, ki je značilen za glive mehke trohnobe (slika 7). Za glive mehke trohnobe je značilno, da z razkrojem celuloze in hemiceluloz ustvarijo vrzeli v S2 sloju sekundarne celične stene, v nadaljnjih stopnjah razkroja ta sloj povsem razgradijo (Reinprecht, 2016). Značilen pojav mehke trohnobe je viden na vzorcu impregniranem s CCB. Kljub temu, da je mehka trohnoba značilna za okolja z zelo visoko vlažnostjo



Slika 6. Profili površine vzorca, impregniranega z biocidnim proizvodom CCB po 14 letih izpostavitve na prostem
Figure 6. Profile of the CCB-treated wood after 14 years of outdoor exposure

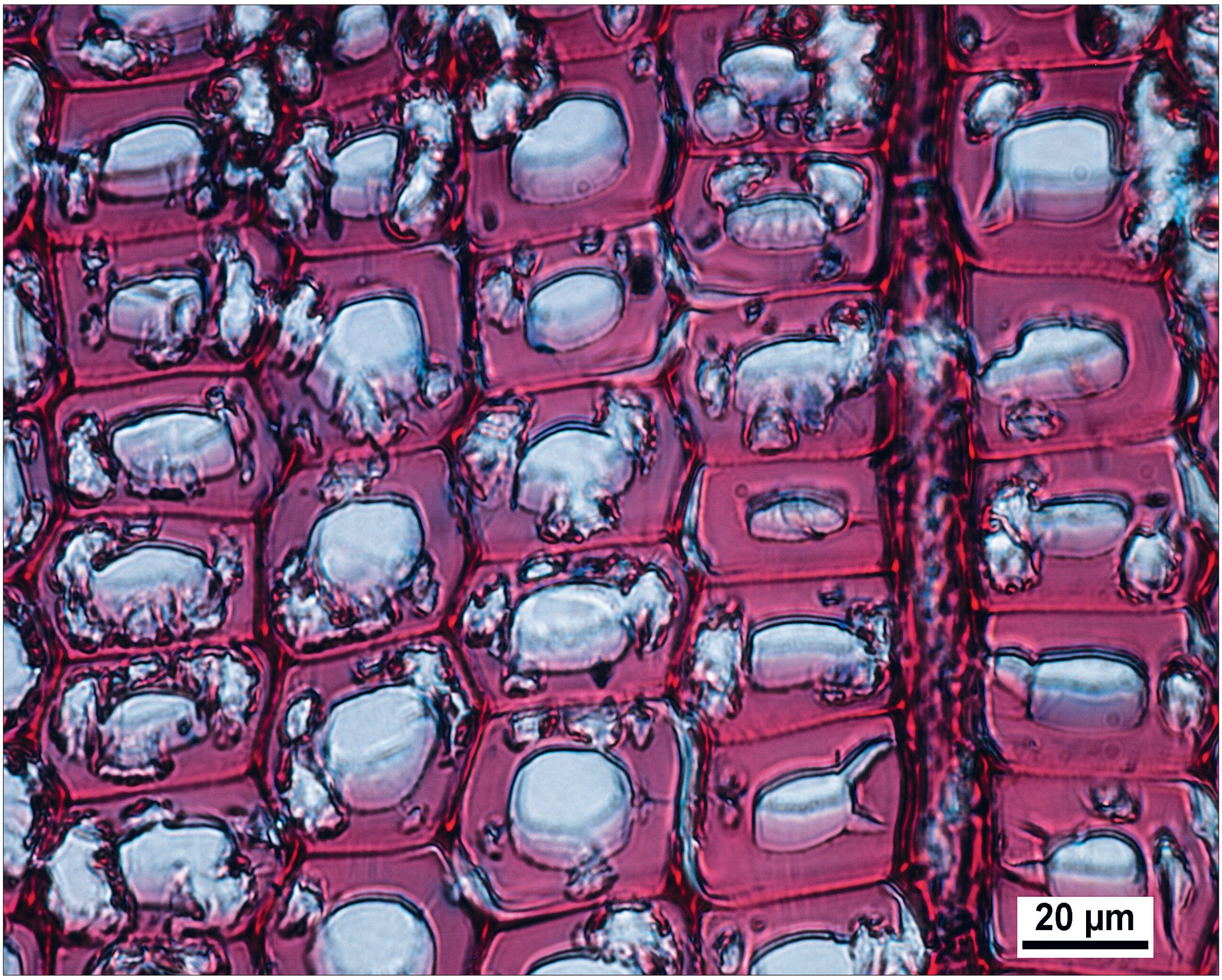


Slika 7. Biofilm na površini s CCB impregniranega vzorca po 14 letih izpostavitve na prostem. Na levi (A) je barvna slika, na desni (B) sliki je razvidna morfologija. (540 µm × 540 µm)
Figure 7. Biofilm on the surface of CCB-treated wood after 14 years of outdoor exposure. On the left (A) there is a colour image, and on the right (B) the morphology is resolved. (540 µm × 540 µm)

in vsebnostjo dušika, kot na primer les v vodnem okolju, se je ta trohnoba pojavila tudi na našem vzorcu, izpostavljenem v tretjem razredu uporabe. Po vsej verjetnosti so k temu pripomogle razpoke in biofilm, kar omogoča zastajanje vode. Tako se lahko tudi na lesu v tretjem razredu uporabe vzpostavijo ugodni pogoji za razkroj. Prisotni biocidi preprečujejo razvoj večine lesnih gliv. Na impregniranem lesu se lahko pojavijo na baker tolerantne glive. V tej skupini so tudi glive mehke trohnobe. K uspešnosti gliv mehke trohnobe pogosto pripomore dejstvo, da pogosto živijo v simbiozi z bakterijami, ki jim pomagajo razstrupiti impregniran les (Clausen, 1996).

Eden od ključnih vzrokov, ki preprečuje razvoj gliv na lesu, je prisotnost biocidnih učinkovin. Suhi navzem aktivnih učinkovin v impregniranih vzorcih je razviden iz slike 8A. Za tretji razred uporabe je priporočeno, da mora les vsebovati vsaj 4 kg aktivnih učinkovin na kubični meter (Willeitner, 2001). Pri izpostavljenih vzorcih smo v povprečju to vrednost dosegli. Povprečni suhi navzem znaša 4,2 kg/m^3, vendar je med vzorci opaziti velike razlike. Suhi navzem niha med 1,8 kg/m^3 in 7,0 kg/m^3 (slika 9A). Razloge za te razlike lahko pripišemo slabi impregnabilnosti smrekovega lesa (CEN, 2016). Slaba impregnabilnost smrekovega lesa je med drugim povezana z aspiracijo pikenj v procesu ojedritve. Najnižji navzem smo zabeležili pri vzorcu 6, ki je po 14 letih propadel. Ta vzorec je tudi predmet analize, opisane v tem članku. Poleg nizkega suhega navzema je k hitremu razkroju pripomoglo še dejstvo, da se je vzorec nahajal v zgornjem sloju dvoslojnega testa, ki je bil bolj izpostavljen razkroju kot spodnji vzorci. Menimo, da navzem ni edini dejavnik, saj med navzemom aktivnih učinkovin in razkrojem nismo odkrili povezave.

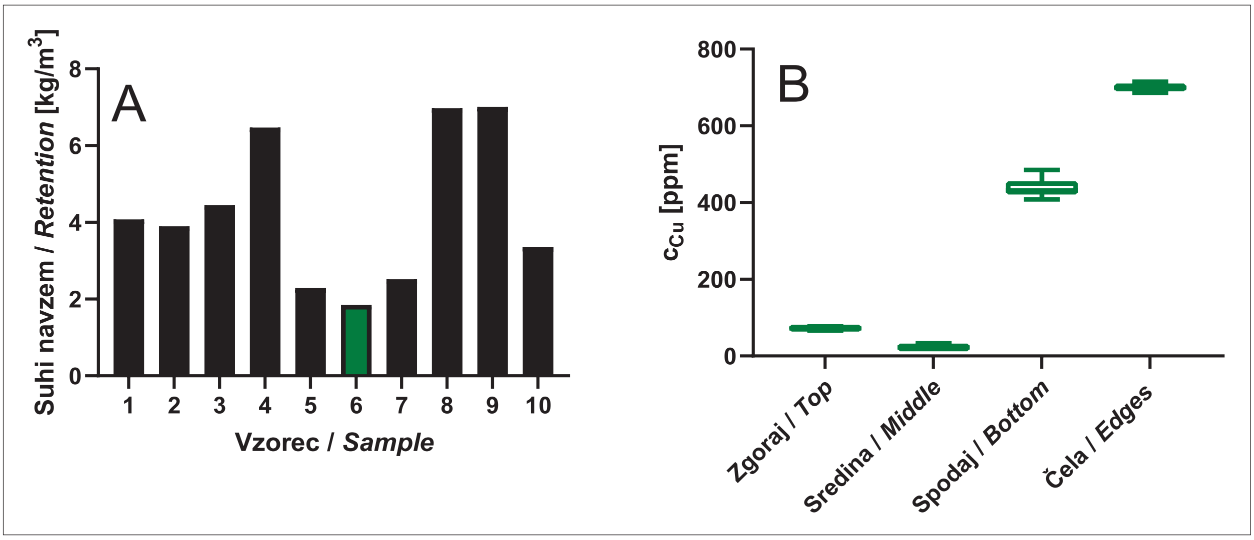
V nadaljevanju opisujemo natančnejšo analizo porazdelitve aktivnih učinkovin po vzorcu. Glede na to, da sta koncentraciji bakrovih in kromovih učinkovin v impregniranem lesu prisotni v enakem razmerju, v nadaljevanju opisujemo le prisotnost bakra. Baker je v biocidnem proizvodu prisoten kot ključna aktivna učinkovina, medtem, ko kromove spojine nimajo biocidnih lastnosti. Kromove spojine v prvi vrsti omogočajo vezavo bakrovih spojin v les (Humar et al., 2004a). Rentgenska fluorescenčna spektrometrija (XRF) je pokazala, da je najvišji navzem bakra mogoče opaziti v okolici čel (c_{Cu} = 700 ppm). Biocidni proizvodi v les bistveno bolje prodirajo v aksialni smeri kot v tangencialni in ra-



*Slika 8. Prečni prerez s CCB impregniranega smrekovega vzorca (*Picea abies*) po 14 letih izpostavitve na prostem*

*Figure 8. Cross-section of CCB-treated Norway spruce wood (*Picea abies*) after 14 years of outdoor exposure*

dialni, zato je ta rezultat pričakovan. Sredica vzorca je bila zelo slabo impregnirana, na kar nakazuje že nizek suhi navzem, zato je nizka koncentracija bakrovih učinkovin v sredini razumljiva (c_{Cu} = 23 ppm). Zanimiva je velika razlika med koncentracijo aktivnih učinkovin v zgornjem (c_{Cu} = 72 ppm) in spodnjem sloju vzorca (c_{Cu} = 438 ppm). Ocenjujemo, da je koncentracija bakra v čelih in spodnjem sloju primerljiva s koncentracijo te aktivne učinkovine v izhodišču. Po drugi strani je koncentracija bakra na zgornji površini skoraj šestkrat nižja od koncentracije bakra na spodnji strani. Menimo, da je k temu pripomoglo izpiranje zaradi kombinacije abiotskih (padavine, UV …) in biotskih dejavnikov (bakterije, glive). Bakterije in glive izločajo cel spekter organskih kislin (oksalna, metanojska, etanojska, hidroksi dikarboksilna butanojska …) (Takao, 1965). V kislem okolju so aktivne učinkovine zaradi nastanka topnih kompleksov bolj topne in se izpirajo iz lesa (Humar et al., 2004b), kar prispeva k počasnemu razstrupljanju lesa in v nadaljevanju pripelje do razkroja. Ta proces se uporablja tudi za bioremediacijo odsluženega lesa za pridobivanje kovin z učinkovitimi biometalurškimi procesi iz jalovine, odpadne elektronike ipd. (Ilyas & Lee, 2015).



Slika 9. (A) Suhi navzem vzorcev, izpostavljenih na terenskem polju. Vzorec šest je bil uporabljen v tej študiji. (B) Koncentracija bakra v posameznih slojih vzorca 6, po 14 letih izpostavitve.

Figure 9. (A) Retention of the wood specimens exposed in the field test site. Sample no 6 was used in the current study. (B) Copper concentration in sample 6 after 14 years of exposure.

4 ZAKLJUČKI
4 CONCLUSIONS

Na nezaščitenem smrekovem lesu se je kmalu po izpostavitvi zunanjim biotskim in abiotskim dejavnikom pojavil razkroj. Prisotnost biocidnih učinkovin (CCB) v impregniranem lesu je uspešno upočasnila razkroj. Kljub vsemu je prvi vzorec, zaščiten z biocidnim proizvodom CCB propadel po 14 letih. Vzorec je propadel zaradi delovanja gliv rjave in mehke trohnobe. Razkroj na dvoslojnem testu je hitrejši pri vzorcih, ki so bili v dvoslojnem testu na zgornji strani, izpostavljeni vremenskim vplivom, zato sam dvoslojni test ni imel izrazitega vpliva na dinamiko razkroja.

Rezultati te raziskave nedvoumno kažejo, da je neodporen les smrekovine treba zaščititi, da mu zagotovimo ustrezno življenjsko dobo. Zaščita mora biti izvedena kvalitetno. Les mora biti prepojen po celotnem preseku, navzem mora ustrezati zahtevam proizvajalca. V nasprotnem primeru tudi zaščiten les lahko hitro propade.

5 POVZETEK
5 SUMMARY

Wood in outdoor applications is exposed to abiotic and biotic factors. Fungi are the most important reason for the failure of wooden constructions. If we want to slow down the decay, there are three possibilities: Using naturally durable wood, wood modification and wood preservation. Since most wood species in Europe do not have durable wood and modification is quite expensive and not suitable for in ground exposures, impregnation with biocides remains the most commonly used alternative. In the past, biocidal products based on copper, chromium, and boron compounds (CCB) were one of the most important solutions for wood protection under extreme conditions. Today, these products have almost entirely been taken off the market in the EU. In order to assess the effectiveness of copper-based wood preservatives, a comprehensive field test site has been set up at the Department of Wood Science and Technology, Biotechnical Faculty. For 14 years, field tests have been carried out under real conditions, in which impregnated samples are exposed to the weather in a double-layer test, with the samples assessed annually for decay. With treated wood, we often find that the wood decays faster than expected. In this work we wanted to determine what contributes to the decay based on an analysis of decayed impregnated wood from the field test site. The decayed sample was analysed by light and laser scanning confocal microscopy. Furthermore, the presence of copper in decayed wood was determined by X-ray fluorescence spectroscopy. The results show that the untreated control wood samples were completely degraded after eight years of exposure. In contrast, CCB-treated wood performed significantly better. The first signs of decay on CCB-treated wood were observed after 10 years of exposure. Decay is associated with surface cracks, biofilm formation and insufficient retention and penetration. Sufficient retention and penetration of the active substances into the wood ensures the planned service life.

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CHEMICAL AND MECHANICAL CHARACTERIZATION OF THERMALLY MODIFIED *GMELINA ARBOREA* WOOD

KEMIJSKA IN MEHANSKA KARAKTERIZACIJA TERMIČNO MODIFICIRANEGA LESA VRSTE *GMELINA ARBOREA*

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Abstract / Izvleček

Abstract: Gmelina arborea *(Roxb. ex. Sm.) wood samples were thermally modified at 180 °C, 200 °C and 220 °C for 3 h, by employing a process similar to ThermoWood®. The resulting effects on the basic chemical composition and mechanical properties were determined. The results were analyzed statistically with ANOVA, and Least Square Deviation was used to compare means. Generally, after the thermal modification (TM) process, the cellulose, hemicelluloses and extractives content decreased significantly. By contrast, lignin proportions increased significantly. Untreated wood and samples modified at 180 °C indicated comparable modulus of elasticity (MOE), modulus of rupture (MOR), degree of integrity (I), fine fraction (F) and resistance to impact milling (RIM). Noteworthy reductions however occurred at 200 °C and 220 °C. Significant increases in Brinell hardness (BH) took place at 180 °C, recording a high decrease at 220 °C.* Gmelina arborea *could be modified suitably at 180 °C for structural and other purposes. To take advantage of other improved properties, modification at 200 °C could be employed for non-structural uses.*

Keywords: *High-Energy Multiple Impact (HEMI), Resistance to Impact Milling (RIM), Thermal modification, Static Bending*

Izvleček: *Vzorce lesa vrste* Gmelina arborea *(Roxb. ex. Sm.) smo 3 ure termično modificirali pri 180* °C, 200 °C in 220 °C *po postopku, ki je soroden ThermoWood® procesu. V nadaljevanju smo ocenili vpliv modifikacije na kemijsko sestavo in mehanske lastnosti. Rezultate smo testirali z analizo variance ANOVA, za primerjavo povprečij pa smo uporabili metodo najmanjših kvadratov. Na splošno se je delež celuloze, hemiceluloz in ekstraktivnih snovi v termično modificiranem lesu znatno zmanjšal. Nasprotno se je delež lignina v modificiranem lesu statistično značilno povečal. Neobdelani les, modificiran pri 180 °C, je imel primerljivo upogibno trdnost (MOR) in modul elastičnosti (MOE), stopnjo integritete (I), delež fine frakcije (F) in odpornost proti udarnemu mletju (RIM). Pri modifikaciji pri temperaturah 200* °C in 220 °C *je prišlo do znatnega poslabšanja omenjenih lastnosti. Trdota po Brinellu (BH) se je znatno povečala pri 180* °C, pri 220 °C *pa se je* močno zmanjšala. *Les vrste* G. arborea *bi bilo za konstrukcijske namene primerno modificirati pri 180 °C. Da bi izkoristili druge izboljšane lastnosti, bi za les za nekonstrukcijske namene lahko uporabili modifikacijo pri 200 °C.*

Ključne besede: *večkratni visokoenergijski udarci (HEMI), odpornost proti udarnemu mletju (RIM), termična modifikacija, statični upogib*

1 INTRODUCTION

1 UVOD

Wood is a very important environmentally friendly, renewable and accessible material, which is widely preferred around the globe. However, the sustainable supply of tropical timber on the world market is increasingly threatened (Boonstra, 2008). For instance, deforestation in Ghana has assumed alarming proportions, leading to massive reductions in the nation's primary forest cover (FAO, 2006). In its 2016 report, the Forestry Commission (FC) of Ghana indicated that about 80% of the country's forest resources under state management had been lost to illegal logging activities facilitated mainly by farming, the chainsaw operations which supply most of the local lumber demand, and mining (FC, 2016a;

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Hansen & Treue, 2008). Asiedu (2019) also recorded a 60% increase in loss of primary forest cover in 2018 compared to 2017 in Ghana, with clearing for cocoa farming being a leading cause of deforestation, while mining remains the biggest threat.

To reduce over-exploitation of primary forests and ensure sustainable supply of timber on the local and export markets, Ghana has embarked on the establishment of plantations, covering about 190,500 ha as of 2015. The FC further plans to cultivate 100,000 ha of plantations in the years 2016 – 2040. Among the many species to be cultivated is *Gmelina arborea,* which is an exotic species (FC, 2016b), indigenous to the Indo-Burma region of Southeast Asia (Nwoboshi, 1994). Over 5,000 ha of *Gmelina arborea* plantations have been established in Ghana, 2,000 ha of which was cultivated by the Subri Industrial Plantation Limited (SIPL), near Sekondi-Takoradi in the Western Region (Nwoboshi, 1994). *Gmelina arborea* has oven-dry density ranges of 390 – 599 kg/m^3 and good machining properties (Mitchual et al., 2018). It is rated as naturally non-durable and thus requires preservative treatment to prolong its service life (Owoyemi et al., 2011). Kasmudjo (1990) indicated that suitable uses of *Gmelina arborea* wood are fuelwood, pulp and paper, plywood, furniture, construction and matches. The researcher further suggested harvesting cycles of five years for fuel wood, 15 years for plywood and boards, 20 years for construction and eight years for uses such as boxes, toothpicks and matches.

To complement the efforts of the FC there is the need to research the properties of these plantation species, in order to establish their suitability for furniture and other such applications. When thermally modified, *Gmelina arborea* could be used for applications exposed to the weather and humidity variations above ground, including cladding, garden furniture, windows and saunas, but could also be used for interior applications such as stairs, decorative panels, flooring, kitchen furniture, etc. (Sandberg & Kutnar, 2016). The current study aimed at redirecting the attention of wood industry stakeholders to plantation species like *Gmelina arborea* and encouraging its use.

Thermal modification (TM) of wood is one important method for improving wood properties, including dimensional stability, natural durability and hygroscopicity. Wood retains its environmentally friendly nature after this process. TM of wood is less costly compared to wood preservation using biocidal treatment (Esteves et al*.,* 2014). However, TM may cause a reduction in mechanical strength. The extent of the effect of TM on the properties of wood depends on species and process conditions (Esteves & Pereira, 2009). Differences in wood species' response to TM treatments are due to variations in chemical, anatomical and physical properties (Ranin, 2004). Výbohová et al. (2018) analysed the effect of TM intensity at the temperatures of 160 °C, 180 °C and 200 °C under atmospheric pressure in the presence of air for durations of 3, 6, 9, and 12 h on ash wood. The researchers reported that the extractives content initially increased up to 200 °C and 3 h, which decreased with extended treatment duration. Hemicellulose monosaccharide (D-xylose) degraded under all treatment conditions, which resulted in a decrease in holocellulose content. Lignin content also increased at temperatures of 180 °C and 200 °C. Bekhta and Niemz (2003) indicated that the MOR of spruce (*Picea abies* (L.) Karst.) decreased by 44% to 50% as the modification temperature was raised from 100 °C to 200 °C, while temperature had no effect on MOE. Korkut et al. (2008) reported that the surface hardness, MOE and MOR in the radial direction of Scots pine *(Pinus sylvestris* L.) decreased by 27%, 32% and 33%, respectively at 180 °C for 10 h.

To reduce the focus on traditional primary timber species in order to curb deforestation in Ghana, it is necessary to promote lesser used species (LUS) like *Gmelina arborea* on both local and export markets. Successful adoption of *Gmelina arborea* for utilization will depend on its properties. This study therefore aimed to investigate the impact of the TM process applied to enhance the properties of *Gmelina arborea* wood from Ghana with regard to its basic chemical and mechanical properties.

2 MATERIALS AND METHODS

2 MATERIAL IN METODE

2.1 MATERIALS

2.1 MATERIAL

2.1.1 Source of Material and Preparation

2.1.1 Izvor materiala in priprava

Four *Gmelina arborea* trees with diameter at breast height (DBH, 1.3 m above ground) of 35 –

55 cm were obtained from a 40-year old plantation of the Centre for Scientific and Industrial Research – Forestry Research Institute of Ghana's (CSIR – FORIG) research plot at Abofour, Offinso District in the Ashanti Region of Ghana. Abofour is located within the moist Semi-Deciduous Forest Zone with average annual rainfall of 1,400 mm. It lies 7°8"0' N and 1°45"0' W and it is about 60 km from the Ashanti regional capital, Kumasi.

Trees were bucked into 2.5 m length bolts with a chainsaw, which were further processed using a portable Wood-Mizer sawmill (LT 30) into boards of 25 mm thickness at the CSIR – FORIG wood workshop at Fumesua, Kumasi. The boards were air-dried for at least 12 months until a moisture content (MC) below 20% was achieved. Boards were randomly selected from within 15 cm radius of the pith to assure the use of heartwood. These boards were further processed into test slats of dimensions 20 x 50 x 650 mm^3 (radial (r) × tangential (t) × longitudinal (l)) for the TM process. The slats were sorted according to weight and those in the range of 300 to 400 g were used for the study. This pre-sorting was necessary for the homogenization of the lot and to minimize the effect of the initial density on the results.

2.2 METHODS

2.2 METODE

2.2.1 Thermal Modification

2.2.1 Termična modifikacija

The open system TM similar to the ThermoWood® process (Mayes & Oksanen, 2002) was employed, using a 65 L capacity laboratory scale reactor. Firstly, the temperature in the vessel was raised at 12 °C/h to 100 °C and then 4 °C/h to 130 °C to allow high temperature drying of slats to nearly 0% MC. Secondly, the temperature was again increased at 12 °C/h until reaching the peak temperatures of 180, 200 and 220 °C. Each peak temperature was held for 3 h. Finally, the temperature was decreased at 20 °C/h until reaching 65 °C, at which the vessel was opened and the slats removed. Eventually, the thermally modified wood samples at the three peak temperatures resulted in three treatments with the untreated wood as control. The wood samples were assessed for selected chemical and mechanical properties.

2.2.2 Mass loss

2.2.2 Izguba mase

The mass loss of each slat after the thermal modification process was calculated as described by Metsä-Kortelainen et al. (2006). Each air-dried slat was weighed before and after the thermal modification process. Moisture content was then determined for the slats before and after the thermal modification process, in order to determine the mass loss.

Mass loss% (ML%), was thus calculated as in Equation 1.

$$\mathrm{ML} = \frac{(m1 - m2) \times 100}{m1} \quad [\%] \qquad (1)$$

Where m_1 is the dry mass of the sample before the thermal modification, in g; m_2 is the dry mass of the sample after the thermal modification, in g.

Dry mass (g), mdry, was calculated as follows (Equation 2).

$$\mathrm{mdry} = \frac{100 \times mu}{u + 100} \quad [\mathrm{g}] \qquad (2)$$

where m_u is the mass of the sample at moisture content u%, in g.

2.2.3 Chemical Analysis

2.2.3 Kemijska analiza

Slats were randomly selected per treatment for the analysis. Two samples of the dimensions 20 × 20 × 20 mm^3 (r × t × l) were cut from both ends of each slat to make up six parts. The parts of the slats were reduced manually and ground in a cutting mill (Retsch, model SM 2000, Haan, Germany) with a sieve of 1 mm diameter, resulting in a compound sample per treatment. The powder was screened in a vibratory sieve shaker (Retsch, model KS 1000, Haan, Germany), operating at 200 rotations min^{-1} for four minutes. The chemical analysis was done on the fraction remaining on the 40-mesh sieve. All chemical analyses were carried out in triplicate for each TM treatment.

The extractives content was determined in accordance with the hot water solubility method, described in the standard T 207 cm-99 (TAPPI, 1999b). The insoluble lignin was determined using 72% sulfuric acid, following the method used by Sluiter et al. (2008). Holocellulose was prepared using a solution of sodium chlorite and acetic acid according to the method of Wise et al. (1946). The filtered fraction from the holocellulose analysis was used in the α-cellulose analysis, which was carried out with a solution of sodium hydroxide (17.5%), according to the standard T-203 cm-99 method (TAPPI, 1999a). The amount of hemicelluloses was calculated by difference [holocellulose - α-cellulose].

2.2.4 Static Bending

2.2.4 Statični upogib

A three-point bending test based on the DIN 52186 (1978) test norm was performed on a universal testing machine (Zwick Roell Z010, Zwick, Ulm, Germany) using the central loading method and 30 samples per treatment, measuring 10 × 10 × 180 mm^3 (r × t × l). For each TM treatment, three slats were used with each providing 10 samples. All the samples were conditioned at 20 °C and 65% relative humidity for seven days until constant weight. This ensured that the moisture content and temperature, which affect the strength of wood, were maintained to enhance comparability of the results. The loading heads moved at constant speeds of 3 mm/min for untreated wood, 3 mm/min, 2 mm/min and 1 mm/min for samples thermally modified at 180 °C, 200 °C and 220 °C, respectively. Loading head speed was varied for each modification to be able to cause the failure of the samples within 90 ± 30 s. The test samples were supported at the ends by rotating supports with the diameter of 15 mm. The distance between the supports was 160 mm. At the point of failure the modulus of elasticity (MOE) and modulus of rupture (MOR) generated by the universal testing machine and displayed on a monitor were recorded. MOE and MOR were determined according to Equations 3 and 4, respectively:

$$MOE = \frac{l3}{4 * b * h^3} * \frac{\Delta F}{\Delta f} \quad [\text{N/mm}^2] \qquad (3)$$

Where ΔF is the force difference in N (Newton) in the elastic deformation range of the specimen; Δf is the deflection, corresponding to the force difference ΔF, in the middle of the specimen in mm; *l* is distance between the supports (span) in mm; b is width of the sample in mm; and h is height of the sample in mm.

$$MOR = \frac{1.5 * P * l}{b * h^2} \quad [\text{N/mm}^2] \qquad (4)$$

Where P is the breaking (maximum) load in N; l and h are the span and height of the samples, respectively.

2.2.5 High Energy Multiple Impact (HEMI-test)

2.2.5 Visokoenergijski večkratni udar (HEMI-test)

The HEMI-test was carried out according to the procedure presented by Brischke (2017). For each treatment, the test was replicated five times. Each replicate was made up of 20 oven-dry samples of 10 × 10 × 10 mm^3 (r x t x l). In all, a total of 100 samples from four slats were assessed for each TM treatment. Each of the 20 samples was placed into a bowl of 140 mm inner diameter of a heavy impact ball mill (Herzog HSM 100, Osnabrück, Germany), together with one steel ball of 35 mm diameter, three steel balls of 12 mm diameter, and three steel balls of 6 mm diameter. The bowl was shaken for 60 s with a rotary frequency of 23.3 s^{-1} and a stroke of 12 mm. The fragments of the 20 samples were fractionated on a slit sieve with a slit width of 1 mm using an orbital shaker at an amplitude of 25 mm and a rotary frequency of 200 min^{-1} for 1 min. The following values were calculated using Equation 5, Equation 6, and Equation 7:

$$I = \left({m_{20}}/{m_{all}} \right) \times 100 \quad [\%] \qquad (5)$$

Where the degree of integrity (I) is the ratio of the mass of the 20 biggest fragments (m_{20}) to the mass of all fractions (m_{all}) after the crushing process.

$$F = \left({m_{<1mm}}/{m_{all}} \right) \times 100 \quad [\%] \qquad (6)$$

Where the fine fraction (F) is the ratio of the mass that is sieved and has a diameter of less than 1 mm ($m_{<1mm}$) to the mass of all fractions (m_{all}).

$$RIM = \frac{(I - 3 * F + 300)}{4} \ [\%] \qquad (7)$$

Where RIM is the resistance to impact milling and represents the value of the measure for the structural integrity of the material.

2.2.6 Brinell hardness

2.2.6 Trdota po Brinellu

The Brinell hardness (static hardness) perpendicular to the grain on the radial face was measured according to DIN EN 1534 (2011) with a universal testing machine (Zwick Roell Z010, Zwick, Ulm, Germany). Ten samples obtained from five slats (i.e. two samples per slat) were used per treatment. The samples were conditioned at 20 °C/ 65% RH for 14 days until constant weight. A maximum force of 500 N was exerted using a steel ball with a diameter of 10 mm applied for 25 seconds on the samples with dimensions of 20 × 50 × 200 mm^3 (r × t × l). The diameters of the residual impressions at three points on a face of each sample, with any two points being at least 50 mm apart, were automatically determined by the testing machine. The Brinell hardness was then calculated according to Equation 8:

$$BH = 2.F \ / \pi . D \left[D - \sqrt{\left(D^2 - d^2 \right)} \right] \ [N/mm^2] \qquad (8)$$

Where BH is the Brinell hardness (N/mm^2), F is the maximum force used (N), D is the diameter of the steel ball (mm) and d is the diameter of the imprint on the sample (mm).

2.2.7 Data Analysis

2.2.7 Obdelava podatkov

Descriptive statistics comprising means with standard deviations were presented for each treatment and test. A comparison of the results from the treatments was made using Analysis of Variance (ANOVA). The Least Square Deviation test was used to compare means at α = 0.05, when ANOVA revealed significant differences. The Statistical Package for Social Sciences (IBM Statistics) version 26 was used for the analyses.

3 RESULTS AND DISCUSSIONS

3 REZULTATI IN DISKUSIJA

3.1 MASS LOSS

3.1 IZGUBA MASE

Table 1 presents the mass loss, proportions of cellulose, hemicelluloses, lignin and extractives in the thermally-modified *Gmelina arborea* wood. Most properties of thermally modified wood depend to a large extent on the mass loss (Bal, 2013). This indicates that high mass loss results in more

Table 1. Mass loss, proportions of cellulose, lignin, hemicelluloses and extractives per treatment.

Preglednica 1. Rezultati izgube mase in deleži celuloze, lignina, hemiceluloz in ekstraktivov glede na uporabljen postopek termične modifikacije

Treatment	Mass Loss [%]	α-Cellulose [%]	Hemicelluloses [%]	Lignin [%]	Extractives [%]
Untreated	0.00 (0.00)	55.93^{a} (0.98)	19.16^{a} (1.13)	34.86^{a} (1.09)	7.87^{a} (0.20)
180 °C	5.44^{a} (0.86)	51.50^{b} (0.55)	18.08^{a} (0.43)	39.62^{b} (0.98)	6.35^{b} (0.34)
200 °C	10.08^{b} (0.95)	53.20^{ab} (0.14)	10.56^{b} (0.88)	40.50^{bc} (0.64)	9.11^{a} (0.23)
220 °C	15.13^{c} (2.17)	52.45^{b} (0.52)	6.63^{c} (0.23)	44.16^{c} (0.62)	6.35^{b} (0.40)

Means followed by the same letter are not significantly different at α = 0.05
Standard error values are shown in parentheses

pronounced changes in wood properties, including chemical and mechanical properties (Militz, 2002; Hill, 2006; Esteeves & Pereira, 2009). Mass loss increased with the modification temperature, from 5.44% at 180 °C to 15.17% at 220 °C. Esteeves and Pereira (2007) reported that mass loss is primarily due to thermal degradation of hemicelluloses and volatilization of extractives. This is evidenced in Table 1, where the hemicelluloses and extractives contents decreased from 19.16% and 7.87% in untreated wood to 6.63% and 6.35% at 220 °C, respectively. The influence of the reduced hemicelluloses content on mass loss is more pronounced (R^2 = 0.9197) than that of the extractives content (R^2 = 0.0369) (Fig. 1).

3.2 CELLULOSE AND HEMICELLULOSES CONTENT

3.2 VSEBNOST CELULOZE IN HEMICELULOZ

Variations in the cellulose concentrations at 180 °C and 220 °C were not significantly different, with both differing significantly from untreated wood. Yildiz et al. (2006) and Esteves et al. (2008) noted that cellulose is less affected by TM in comparison to hemicelluloses in an atmosphere without oxygen. Degradation of amorphous cellulose is principally what takes place, making the resulting cellulose more crystalline and causing a reduction of cellulose content in TM woods (Boonstra & Tjeerdsma, 2006). The hemicelluloses content decreased significantly from 19.16% when untreated

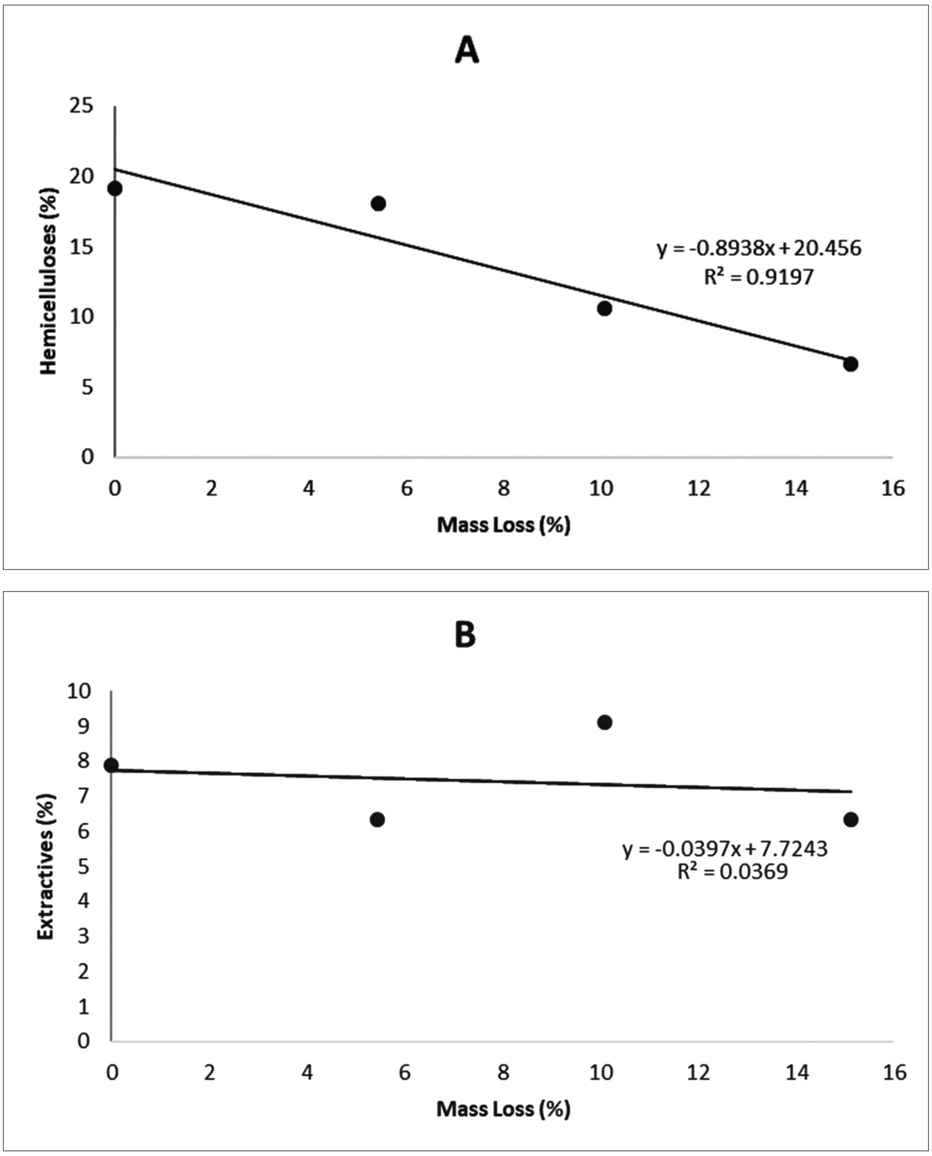

Figure 1. Relationship between mass loss and hemicelluloses (A) and extractives (B) contents [%].

Slika 1. Zveza med izgubo mase in vsebnostjo hemiceluloz (A) in ekstraktivnih snovi (B) [%].

to 6.63% at 220 °C modification temperature (Table 1). According to Sivonen et al. (2002) and Nuopponen et al. (2004), hemicelluloses are the first wood structural component to be affected during TM. They pointed out that deacetylation and the released acetic acid act as a depolymerization catalyst that further facilitates polysaccharide degradation, in line with the observations made in this study. The hemicelluloses content decreased with increased modification temperature, showing the highest reduction of 65.40% at 220 °C. On the other hand, cellulose decreased by a maximum of 8.60% at 180 °C. As such, cellulose is less degraded than hemicelluloses when wood is thermally modified.

3.3 LIGNIN CONTENT

3.3 DELEŽ LIGNINA

Table 1 shows that the lignin content increased significantly and consistently from 34.86% for untreated wood to 44.16% for a modification temperature of 220 °C. Similar results were obtained by Zaman et al.,(2000) in their study of Scots pine (*Pinus sylvestris* L.) and birch (*Betula pendula* Roth Tent. fl. Germ.) thermally modified at 205 °C and 230 °C with respective holding times of 4 and 8 hours. They recorded increased lignin content from 24.5% to 38.7% and from 21.8% to 35.8%, respectively. Several researchers have suggested that an increase in lignin content after TM could be due to polycondensation reactions of lignin with other cell wall components (Tjeerdsma & Militz, 2005; Boonstra & Tjeerdsma, 2006). This results in cross-linking of lignin, leading to an increase in lignin content. Other reports have also indicated that lignin degrades at the beginning of the modification process, although the rate of degradation is slower than that seen with polysaccharide (hemicellulose) degradation (Windeisen et al., 2007). Therefore, the higher decrease in polysaccharide content gives the apparent observed increase in lignin content.

3.4 EXTRACTIVES CONTENT

3.4 DELEŽ EKSTRAKTIVOV

Generally the extractives content (EC) was significantly reduced after TM. It decreased from the initial content of 7.87% for untreated wood to 6.35% for modification temperatures of 180 °C and 220 °C (Table 1). A spike in EC of 9.11% was, however, recorded at a modification temperature of 200 °C. Similarly, Esteves et al. (2008) reported a substantial increase in EC with increased modification temperature followed by a decrease with a further increase in temperature. Esteves and Pereira (2009) explained that although EC generally decreases with increased modification temperatures, new extractable compounds released at certain temperatures from polysaccharide degradation, such as water and ethanol, may cause it to increase. Esteves et al. (2008) indicated that the composition of the extractives changed as the original extractives disappeared and new compounds were formed in their place. In the present study, part of the inherent extractives may have been removed at 180 °C resulting in the observed decrease in EC. In contrast, the increased EC at 200 °C could have been contributed by degradation products of polysaccharides or hemicelluloses at that temperature. A further increase in temperature to 220 °C resulted in increased removal of extractible materials leading to the decreased EC and the significant mass loss observed. Consequently, the composition of the extractives may also vary from the original.

3.5 MODULUS OF ELASTICITY IN STATIC BENDING

3.5 MODUL ELASTIČNOSTI IZ STATIČNEGA UPOGIBA

Table 2 presents results of mechanical tests of thermally modified *Gmelina arborea* wood. Generally, a decreasing trend was observed in the Modulus of Elasticity (MOE) of the thermally modified wood with an increase in temperature. At 220 °C a significant reduction of 25.71% in MOE was observed compared to the untreated wood. Despite the marginal increase of 6.89% at 180 °C and a decrease of 3.50% at 200 °C compared to the untreated wood's MOE, the differences were not statistically significant. The initial marginal increase or relatively stable MOE at 180 °C (mass loss of 5.44% (Table 1)) was mainly due to crystallization of cellulose and condensation of lignin resulting from cross-linking reactions with furfurals produced from the thermal degradation of hemicelluloses (Bal & Bektas, 2013; Wang et al., 2018). However, it is noteworthy that comparing the MOE's for the various modification temperatures shows that there were significant reductions with an increase in temperature (Table 2). A similar observation has been made by other researchers. Esteves and Pereira (2007) and Inoue et al. (1993), for example, observed an increase in MOE at about 4.0% mass loss, which decreased subsequently at higher mass losses or modification temperatures. Xu et al. (2019) reported an initial increase in MOE from 9230 N/mm^2 in the control samples to 10840 N/mm^2 of TM white oak (*Quercus alba* L.) at 160 °C which decreased to 7640 N/mm^2 at TM temperature of 200 °C and 9 h holding times. Hidayat et al., (2016) also obtained significant reductions in the MOE for thermally modified wood of *Cylicodiscus gabunensis* (Harms).

3.6 MODULUS OF RUPTURE IN STATIC BENDING

3.6 TRDNOST PRI STATIČNEM UPOGIBU

Modulus of rupture (MOR) is one of the mechanical properties most affected by wood TM, and it decreases with increasing modification temperature (Esteves & Pereira, 2009). The MOR of *Gmelina arborea* wood was significantly reduced after TM, from 85.85 N/mm^2 when untreated to 38.76 N/mm^2 at 220 °C (Table 2). Decreased MOR could be caused by degradation of wood structural components, specifically cellulose and hemicelluloses. The hemicelluloses content gets degraded much more than cellulose (Table 1). When amorphous hemicelluloses are degraded, the remaining and dominant cellulose becomes more crystalline (Boonstra & Tjeerdsma, 2006). Wood then becomes increasingly brittle with increased modification temperature, lowering its MOR. One of the functions of hemicelluloses is to absorb stress transferred in wood by reinforcing cellulose microfibrils in the wood cell wall. Removal of hemicelluloses thus leads to the distribution of stress over less cell wall material which is brittle, resulting in failure with minimal stress (Winandy & Lebow, 2001).

3.7 DEGREE OF INTEGRITY (I), FINE FRACTION (F) AND RESISTANCE TO IMPACT MILLING (RIM)

3.7 STOPNJA INTEGRITETE (I), DROBNA FRAKCIJA (F) IN ODPORNOST NA UDARNO MLETJE (RIM)

The degree of integrity of *Gmelina arborea* decreased significantly from 55.30% for untreated wood to the lowest value of 42.92% at 220 °C modification temperature. The fine fraction, which shows greater discrimination between untreated and modified wood, generally increased significantly when wood was modified, from 1.32% in untreated wood to 8.17% at 220 °C (Table 2). Overall, a significant reduction in RIM was recorded between untreated and modified wood. Untreated wood recorded RIM of 87.84% reaching a lowest value of 79.60% at 220 °C (Table 2). The reduction in RIM as a result of increased modification temperature

Table 2. Results of static bending, HEMI-test and Brinell hardness per treatment.

Preglednica 2. Rezultati statičnega upogiba, HEMI-testa in trdote po Brinellu glede na postopek obdelave.

Treatment	MOE [N/mm^2]	MOR [N/mm^2]	I [%]	F [%]	RIM [%]	BH [N/mm^2]
Untreated	9562.73^{ab} (125.60)	85.85^{a} (2.00)	55.30^{a} (0.78)	1.32^{a} (0.08)	87.84^{a} (0.16)	17.39^{ab} (0.49)
180 °C	10221.82^{a} (250.97)	82.56^{a} (2.11)	53.85^{a} (0.67)	1.19^{a} (0.11)	87.57^{a} (0.22)	20.65^{c} (0.57)
200 °C	9227.88^{b} (177.32)	56.45^{b} (1.76)	47.19^{b} (0.72)	3.66^{b} (0.17)	84.05^{b} (0.26)	19.18^{ac} (0.52)
220 °C	7103.94^{c} (178.34)	38.76^{c} (1.13)	42.92^{c} (2.01)	8.17^{c} (0.35)	79.60^{c} (0.45)	15.90^{bd} (0.53)

Means followed by the same letter are not significantly different at α = 0.05
Standard error values are shown in parentheses
MOE: Modulus of Elasticity, MOR: Modulus of Rupture, I: Integrity, F: Fine fraction, RIM: Resistance to Impact Milling, BH: Brinell Hardness.

could be ascribed to the reduced microstructural integrity which underlies the recorded increase in fragmentation and decrease in fragment size (Rapp et al., 2006; Welzbacher, et al., 2011). The decline in microstructural integrity occurred as a result of reductions in hemicelluloses content (Table 1) and increased cellulose crystallinity at higher modification temperatures leading to increased wood brittleness.

3.8 BRINELL HARDNESS

3.8 TRDOTA PO BRINELLU

Table 2 shows that Brinell hardness parallel to the grain increased from 17.39 N/mm^2 up to 20.65 N/mm^2 and 19.18 N/mm^2 as *Gmelina arborea* wood was thermally modified at 180 °C and 200 °C, respectively. It reduced to a minimum of 15.90 N/mm^2 at 220 °C modification temperature. Wood hardness increased with modification temperature in the instance of 180 °C and 200 °C, due to increased cellulose crystallinity as the modification temperature increased. However, the significant decrease observed at 220 °C could be due to enhanced degradation of hemicelluloses (Table 1), which reduced the wood's ability to withstand stresses (Winandy & Lebow, 2001). A similar trend was recorded in ash (*Fraxinus spp*. L.) and tali (*Erythrophleum ivorense* A. Chev.), each thermally modified at 180 °C for 1.5 h and 210 °C for 2 h. The Brinell hardness increased from 6.79 N/mm^2 and 4.51 N/mm^2 in untreated wood to 7.01 N/mm^2 and 10.81 N/mm^2 at 180 °C and subsequently reduced to 6.95 N/mm^2 and 9.19 N/mm^2 at 210 °C (Sivrikaya et al., 2015).

4 CONCLUSIONS

4 ZAKLJUČKI

The research focused on the effects of thermal modification at temperatures of 180 °C, 200 °C and 220 °C on chemical and mechanical changes in *Gmelina arborea* wood. Generally, the cellulose and hemicelluloses content decreased significantly after TM, with only lignin recording a significant increase with TM temperature. Additionally, MOE, MOR, I, and RIM decreased with increased modification temperature, recording the highest decreases of 26.13%, 58.30%, 83.69%, 22.37%, and 9.38%, respectively, at 220 °C. The fine fraction and Brinell hardness increased up to 518.94% at 220 °C and 18.75% at 180 °C, respectively. The closely comparable strength properties between untreated wood and that modified at 180 °C makes this particular temperature acceptable for modification of this species, especially when used for structural purposes and other such applications, where the strength properties are critical. However, for purposes other than structural ones a modification temperature of 200 °C could be adopted to offer the added advantage of other improved wood properties. The results obtained in this study are generally useful as a reference for applications of thermally-modified *Gmelina arborea* wood.

5 SUMMARY

5 POVZETEK

Wood is a very important environmentally friendly, renewable and accessible material, which is widely preferred around the globe. However, the sustainable supply of tropical timber on the world market is increasingly threatened (Boonstra, 2008). In its 2016 report, the Forestry Commission (FC) of Ghana indicated that about 80% of the country's forest resources under state management had been lost to illegal logging activities (FC, 2016a). To reduce over-exploitation of primary forests and ensure a sustainable supply of timber on local and export markets, Ghana has embarked on the establishment of plantations covering about 190,000 ha as of 2015. The FC further plans to cultivate 100,000 ha of plantations from 2016 – 2040. Among the many species to be cultivated is *Gmelina arborea*, which is an exotic species (FC, 2016b). To complement the effort of the FC to curb deforestation in Ghana and reduce the focus on traditional primary timber species, there is a need to research the properties of the plantation species to establish their suitability for furniture and other such applications. Successful adoption of plantation species such as *G. arborea* for utilization on both local and export markets will depend on their properties. This study therefore investigated the impact of TM applied to enhance the properties of *G. arborea* wood from Ghana with regard to its basic chemical and mechanical properties.

Four *Gmelina arborea* trees were bucked into 2.5 m length bolts with a chainsaw, which were further processed using a portable Wood-Mizer sawmill (LT 30) into boards of 25 mm thickness. The boards were air-dried until moisture content (MC) below 20% was achieved. Boards were selected from within 15 cm radius of the pith to ensure the use of heartwood. These boards were further processed into slats of dimension 20 × 50 × 650 mm^3 for the TM process. The slats were sorted according to weight and those in the range of 300 – 400 g were used for the study. The pre-sorting was necessary to homogenize the lot and minimize the effect of the initial density on the results.

An open TM system similar to the ThermoWood® process (Mayes & Oksanen, 2002) was employed. Peak temperatures of 180, 200, and 220 °C were adopted. Each peak temperature was held for 3 h. The mass loss of the wood slats after the TM process was determined according to Metsa-Kortelainen et al. (2006). The chemical composition of the wood was also conducted based on TAPPI and Wise et al. (1946). Static bending (MOE and MOR) was determined in accordance with DIN 52186 (1978). High Energy Multiple Impact (HEMI) testing was performed using methods outlined by Brischke (2017). The degree of integrity (I), fine fraction (F) and resistance to impact milling (RIM) were determined. Brinell (static) hardness were also carried out according to DIN EN 1534 (2011). Descriptive statistics comprising means with standard deviations were presented for each treatment and test. Comparison of the results from the treatments was made using Analysis of Variance (ANOVA). Least Square Deviation was used to compare means at $\alpha = 0.05$ when ANOVA revealed significant differences. The Statistical Package for Social Sciences (IBM Statistics) version 26 was used for the analysis.

Table 1 presents the mass loss, proportions of cellulose, hemicelluloses, lignin and extractives in the thermally modified *Gmelina arborea* wood. Mass loss increased along with the TM temperature from 5.44% at 180 ºC to 15.17% at 220 ºC. According to Esteves and Pereira (2007), mass loss is primarily due to thermal degradation of hemicelluloses and volatilization of extractives (Fig. 1). Variations in the cellulose concentrations at 180 °C (51.50%) and 220 °C (52.45%) were not significantly different, with both differing significantly from untreated wood (55.93%). Hemicelluloses content decreased significantly from 19.16% when untreated to 6.33% at 220 °C (Table 1). Esteves et al. (2008) noted that cellulose is less affected by TM in comparison to hemicelluloses. According to Sivonen et al. (2002) and Nuopponen et al. (2004), hemicelluloses are the first wood structural component to be affected during TM. The highest reductions in cellulose and hemicelluloses were 8.60% at 180 °C and 65.40% at 220 °C respectively. Lignin content increased consistently from 34.86% for untreated wood to 44.16% for a modification temperature of 220 °C. Windeisen et al. (2007) reported that lignin degrades at the beginning of the modification process, but the rate of degradation is slower than polysaccharide (hemicelluloses) degradation, giving the apparent observed increase in lignin content. Generally, the extractive content (EC) was significantly reduced after TM. It decreased from the initial content of 7.87% for untreated wood to 6.35% at 180 °C and 220 °C (Table 1).

Table 2 presents the results of mechanical tests of thermally modified *Gmelina arborea* wood. Generally, a decreasing trend was observed in the modulus of elasticity (MOE) with an increase in temperature. At 220 °C, a significant reduction of 25.71% in MOE was observed compared to untreated wood. In spite of the marginal increase of 6.86% at 180 °C and decrease of 3.50% at 200 °C compared to the untreated wood's MOE, the differences were not statistically significant. The modulus of rupture (MOR) is one of the mechanical properties that are most affected by wood TM and decreases with increasing modification temperature (Esteves & Pereira, 2009). The MOR of *Gmelina arborea* wood was significantly reduced after TM, from 85.85 N/mm^2 when untreated to 38.76 N/mm^2 at 220 °C (Table 2). When amorphous hemicelluloses are degraded, the remaining and dominant cellulose become crystalline (Boonstra & Tjeerdsma, 2006). Removal of hemicelluloses thus leads to the distribution of stress over less cell wall material which is brittle, resulting in failure with minimal stress (Winandy & Lebow, 2011). The degree of integrity (I) of *Gmelina arborea* decreased significantly from 55.30% for untreated wood to a low of 42.92% at 220 °C modification temperature. The fine fraction, which shows greater discrimination between untreated and modified wood, generally increased significantly when wood was modified, from 1.32% in untreated wood to 8.17% at 220 °C (Table 2). Overall, a significant reduction in RIM was recorded between untreated and modified wood. Untreated wood recorded RIM of 87.84% reaching a lowest value of 79.60% at 220 °C (Table 2). The reduction in RIM as a result of increased modification temperature could be ascribed to the reduced microstructural integrity which underlies the recorded increase in fragmentation and decrease in fragment size (Rapp et al., 2006; Welzbacher et al., 2011). Brinell hardness parallel to the grain increased from 17.39 up to 20.65 and 19.18 N/mm^2 as *Gmelina arborea* wood was thermally modified at 180 °C and 200 °C, respectively. It fell to a minimum of 15.90 N/mm^2 at 220 °C.

Generally, the cellulose and hemicelluloses contents decreased significantly after TM, with only lignin recording a significant increase with TM temperature. Additionally, MOE, MOR, I, and RIM decreased with increased modification temperature, recording the highest decreases of 26.13%, 58.30%, 83.69%, 22.37%, and 9.38%, respectively, at 220 °C. The fine fraction and Brinell hardness saw increases of up to 518.94% at 220 °C and 18.75% at 180 °C, respectively. Closely comparable strength properties between untreated wood and those modified at 180 °C, makes this particular temperature acceptable for modification of this species, especially for structural purposes and other such applications, where the strength properties are critical. However, for purposes other than structural ones a modification temperature of 200 °C could be adopted to offer the added advantage of other improved wood properties. The results obtained in this study are generally useful as a reference for applications of thermally modified *Gmelina arborea* wood.

Les je pomemben okolju prijazen, obnovljiv, dostopen in priljubljen material po vsem svetu. Trajnostna ponudba tropskega lesa je na svetovnem trgu vse bolj ogrožena (Boonstra, 2008). V svojem poročilu za leto 2016 je gozdarska komisija (FC) iz Gane navedla, da je bilo okoli 80 % državnih gozdnih virov izgubljenih zaradi nezakonite sečnje (FC, 2016a). Da bi zmanjšala prekomerno izkoriščanje primarnih gozdov in zagotovila trajnostno oskrbo z lesom za porabo doma in za izvoz, je Gana v letu 2015 zasnovala nasade v obsegu približno 190.000 ha. FC nadalje načrtuje gojenje lesa na 100.000 ha nasadov v obdobju 2016–2040. Med številnimi vrstami, primernimi za gojenje, je *Gmelina arborea*, ki na območju predstavlja eksotično vrsto (FC, 2016b). Da bi dopolnili prizadevanja FC, omejili krčenje gozdov v Gani in zmanjšali prekomerno izkoriščanje tradicionalnih primarnih lesnih vrst, želijo raziskati lastnosti lesa plantažnih vrst in njihovo primernost za pohištvo in podobne namene. Uspešno uvajanje plantažnih vrst, kot je *G. arborea*, za uporabo doma in za izvoz, bo odvisno od lastnosti lesa. Ta študija zato vključuje raziskave vpliva postopka termičnega modificiranja (TM), ki se uporablja za izboljšanje relevantnih lastnosti lesa *G. arborea* iz Gane, in vpliva na njegove osnovne kemijske in mehanske lastnosti. Štiri drevesa vrste *Gmelina arborea* so bila z motorno žago razžagana na hlode dolžine 2,5 m, ki so bili nato s prenosno žago Wood-Mizer (LT 30) razžagani v deske debeline 25 mm. Deske smo osušili na zraku, do lesne vlažnosti (MC) pod 20 %. V nadaljevanju smo izbrali deske znotraj polmera 15 cm od stržena na območju jedrovine in jih razžagali na letve dimenzij 20 mm × 50 mm × 650 mm za postopek TM. Letve smo sortirali glede na maso lesa in za raziskavo uporabili tiste z maso od 300 do 400 g. Predhodno razvrščanje je bilo potrebno za homogenizacijo vzorca in zmanjšanje učinka gostote lesa na rezultate. Uporabili smo odprti sistem termične modifikacije, podoben postopku ThermoWood® (Mayes & Oksanen, 2002). Najvišje temperature tretiranja so bile 180, 200 in 220 °C, trajanje delovanja vsake od navedenih temperatur pa je bilo 3 ure. Izguba mase lesa je bila po TM določena v skladu z Metsa-Kortelainen et al. (2006). Kemijsko sestavo lesa smo določili na podlagi TAPPI in Wise et al. (1946). Statično upogibno trdnost in modul elastičnosti (MOR in MOE) smo določili v skladu z DIN 52186 (1978). Preskus večkratnih visokoenergijskih udarcev (HEMI) je bil izveden z uporabo metod, ki jih je opisal Brischke (2017). Določena je bila stopnja integritete (I), drobne frakcije (F) in odpornost na udarno mletje (RIM). (Statična) trdota po Brinellu je bila določena v skladu z DIN EN 1534 (2011). Za rezultate posamičnega postopka in testiranja smo izračunali osnovno statistiko, srednje vrednosti s standardnim odklonom. Primerjavo rezultatov po postopkih smo ocenili z analizo variance

(ANOVA). LSD test mnogoterih primerjav smo uporabili za primerjavo povprečij pri stopnji zaupanja α = 0,05, ko je ANOVA pokazala statistično značilne razlike. Za analize smo uporabili Statistični paket za družbene vede (IBM Statistics) različice 26.

V preglednici 1 so predstavljeni deleži celuloze, hemiceluloz, lignina, ekstraktivnih snovi in izgube mase v termično modificiranem lesu vrste *G. arborea*. Razlike v deležih celuloze pri 180 °C (51,50 %) in 220 °C (52,45 %) niso bile statistično značilne, v obeh primerih pa smo zabeležili bistvene zmanjšanje deleža celuloze v primerjavi z neobdelanim lesom (55,93 %). Vsebnost hemiceluloz se je znatno zmanjšala z 19,16 % (neobdelan les), na 6,33 % pri 220 °C (preglednica 1). Esteves et al. (2008) so ugotovili, da TM bolj zmanjšuje delež hemiceluloz kot celuloze. Sivonen et al. (2002) in Nuopponen et al. (2004) so objavili, da TM najbolj vpliva na deleže hemiceluloz. Najvišje znižanje deležev celuloze in hemiceluloz je bilo 8,60 % pri 180 °C in 65,40 % pri 220 °C. Vsebnost lignina se je sistematično povečala s 34,86 % pri neobdelanem lesu na 44,16 % pri lesu, obdelanem pri temperaturi 220 °C. Windeisen et al. (2007) so poročali, da se lignin razgrajuje na začetku postopka modifikacije, vendar je stopnja razgradnje počasnejša od razgradnje polisaharidov (hemiceluloze), kar ima za posledico znatno povečanje vsebnosti lignina. Na splošno se je vsebnost ekstraktivnih snovi (EC) po TM znatno zmanjšala. Z začetne vsebnosti 7,87 % v neobdelanem lesu se je zmanjšala na 6,35 % pri 180 °C in 220 °C (preglednica 1). Izguba mase se je povečala s povečanjem temperature TM za 5,44 % pri 180 °C do 15,17 % pri 220 °C. Esteves & Pereira (2007) poročata, da je izguba mase predvsem posledica toplotne razgradnje hemiceluloz in hlapenja ekstraktivnih snovi (slika 1).

V preglednici 2 so predstavljeni rezultati mehanskih preskusov toplotno modificiranega lesa vrste *G. arborea*. Na splošno smo opazili trend zniževanja modula elastičnosti (MOE) z višanjem temperature. Pri 220 °C smo opazili znatno zmanjšanje MOE za 25,71 % v primerjavi z neobdelanim lesom. Kljub mejnemu povečanju 6,86 % pri 180 °C in 3,50 % pri 200 °C v primerjavi z MOE neobdelanega lesa razlike niso bile statistično pomembne. Upogibna trdnost (MOR) je ena od mehanskih lastnosti, ki se najbolj zmanjša pri TM v odvisnosti od naraščanja temperature (Esteves & Pereira, 2009). Upogibna trdnost lesa vrste *G. arborea* se je po TM znatno zmanjšala, in sicer s 85,85 N/mm^2 pri neobdelanem, na 38,76 N/mm^2 pri lesu, obdelanem pri temperaturi 220 °C (preglednica 2). Ko se amorfne hemiceluloze razgradijo, postane preostala celuloza bolj kristalinična (Boonstra & Tjeerdsma, 2006). Odstranjevanje hemiceluloz vodi do porazdelitve stresa po manjši količini materiala celične stene, ki je krhek, kar povzroči porušitev že pri nizki napetosti (Winandy & Lebow, 2011). Stopnja integritete (I) lesa *G. arborea* se je znatno zmanjšala s 55,30 % pri neobdelanih do 42,92 % pri 220 °C. Drobna frakcija, ki kaže večje razlike med neobdelanim in modificiranim lesom, se je na splošno znatno povečala z modifikacijo, od 1,32 % v neobdelanem lesu na 8,17 % pri 220 °C (preglednica 2). V glavnem je bilo zabeleženo znatno zmanjšanje RIM med neobdelanim in modificiranim lesom. Neobdelani les je imel RIM 87,84 %, ki je pri 220 °C padel na 79,60 % (preglednica 2). Zmanjšanje RIM zaradi povečane temperature modifikacije bi lahko pripisali zmanjšani mikrostrukturni celovitosti, ki je podlaga za povečanje fragmentacije in zmanjšanje velikosti delcev (Rapp et al., 2006; Welzbacher et al., 2011). Trdota po Brinellu vzporedno s potekom aksialnih elementov se je povečala s 17,39 na 20,65 oz. 19,18 N/mm^2, po toplotni modifikaciji pri 180 °C oziroma 200 °C. Trdota se je znižala na 15,90 N/mm^2 pri 220 °C. Na splošno se je vsebnost celuloze in hemiceluloz po TM znatno zmanjšala, le delež lignina se je znatno povečal s temperaturo TM. Poleg tega so se MOE, MOR, I in RIM znižali s povišano temperaturo modifikacije, pri čemer smo zabeležili njihova najvišja znižanja za 26,13 %, 58,30 %, 83,69 %, 22,37 % in 9,38 % pri 220 °C. Drobna frakcija in trdota po Brinellu sta se povečali do 518,94 % pri 220 °C in 18,75 % pri 180 °C. Majhna izguba trdnosti po obdelavi pri 180 °C nakazuje, da je ta temperatura sprejemljiva za modifikacijo lesa proučene vrste, zlasti kadar jo želimo porabiti za konstrukcijske in podobne namene, kjer so trdnostne lastnosti zelo pomembne. Za uporabo lesa za nekonstrukcijske namene pa bi bila lahko sprejemljiva tudi temperatura modifikacije 200 °C, da bi dosegli dodatno izboljšanje drugih lastnosti lesa. Predstavljeni rezultati te študije bodo po pričakovanju splošno uporabni kot referenca za uporabo toplotno modificiranega lesa pogoste plantažne vrste *Gmelina arborea*.

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UPORABNOST LESNIH OSTANKOV TUJERODNIH INVAZIVNIH DREVESNIH VRST ZA PROIZVODNJO PELETOV

USEFULNESS OF NON-NATIVE INVASIVE TREE SPECIES WOOD RESIDUES FOR PELLET PRODUCTION

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Izvleček / Abstract

Izvleček: *Na laboratorijski peletirni napravi smo izdelali pelete iz petih izbranih tujerodnih invazivnih drevesnih vrst, ki rastejo na območju Slovenije in sicer: divjega kostanja* (Aesculus hippocastanum)*, amerikanskega javora* (Acer negundo)*, robinije* (Robinia pseudoacacia)*, trnate gledičevke (*Gleditsia triacanthos*) in velikega pajesena* (Ailanthus altissima) *ter mešanic surovine omenjenih tujerodnih invazivnih vrst s smrekovino* (Picea abies) *v razmerjih 70 : 30 in 50 : 50. Pod enakimi proizvodnimi pogoji smo skupno izdelali 15 različnih vrst peletov. Izdelanim peletom smo določili pomembnejše fizikalne in mehanske lastnosti (vsebnost vode, gostoto nasutja, mehansko obstojnost in vsebnost pepela). Rezultate smo primerjali z mejnimi vrednostmi, opredeljenimi v standardu SIST EN ISO 17225-2:2014. Vsebnost vode in gostota nasutja vseh izdelanih vrst peletov sta zadostili zahtevam standarda za razvrstitev v najvišji kakovostni razred A1. Mehanske obstojnosti izdelanih peletov niso dosegale zahtev standarda in niso presegale 96,5% (kar je mejna vrednost za B razred). Rezultati kažejo, da imajo med izbranimi tujerodnimi invazivnimi drevesnimi vrstami največji potencial za nadaljnjo optimizacijo peletirnega postopka robinija, trnata gledičevka in visoki pajesen.*

Ključne besede: *lesni ostanki, trdna biogoriva, tujerodne invazivne drevesne vrste, peletiranje, kakovost peletov*

Abstract: *We produced pellets from five invasive non-native tree species growing in Slovenia on a laboratory pelleting device, namely: wild chestnut* (Aesculus hippocastanum)*, boxelder maple* (Acer negundo)*, black locust* (Robinia pseudoacacia)*, thorny locust* (Gleditsia triacanthos) *and tree of heaven* (Ailanthus altissima)*, as well as mixtures of the raw material from the above non-native invasive species and spruce* (Picea abies) *in the ratios 70:30 and 50:50. Under the same production conditions, we produced a total of 15 different types of pellets. The most important physical and mechanical properties (water content, bulk density, mechanical stability and ash content) were determined for the pellets produced. The results were compared with the limits defined in the standard SIST EN ISO 17225-2:2014. The water content and bulk density of all produced pellet types met the requirements of the standard for the highest quality class A1. The mechanical durability of the pellets produced did not meet the requirements of the standard and did not exceed 96.5% (which is the limit value for quality class B). The results suggest that black locust, thorny locust and tree of heaven have the highest potential for further optimization of the pelleting process.*

Keywords: *wood residues, solid biofuels, non-native invasive tree species, pelleting, pellet quality*

1 UVOD
1 INTRODUCTION

Vse večja skrb za čisto okolje in zmanjševanje emisij toplogrednih plinov ter hkrati vse večje potrebe po energiji se kažejo v povečani rabi obnovljivih virov energije. Proizvodnja in poraba lesnih peletov iz leta v leto narašča tako v svetovnem kot tudi evropskem merilu in po podatkih European Pellet Council je proizvodnja peletov v EU-28 v letu 2019 za 5 % presegla proizvodnjo v letu 2018 in je tako dosegla 18 milijonov ton (EPC, 2021). Po podatkih Gozdarskega inštituta Slovenije konstantno narašča tudi letna proizvodnja peletov v Sloveniji in se je med letom 2010 in 2019 povečala iz 60.000 ton na 134.000 ton (GIS, 2021).

Zaradi vse večjega obsega proizvodnje in rabe peletov se bo tudi v prihodnjih letih potreba po vhodni surovini konstantno povečevala (Stelte et al., 2012; Lauri et al., 2014; Puig-Arnavat et al., 2016; Whittaker & Shield, 2017; García, 2019; Picchio et al., 2020), kar ima vpliv tudi na ceno vhodne suro-

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vine. Zato je potrebno raziskati dodatne možnosti uporabe in potenciale različnih lesnih kot tudi nelesnih virov biomase. Zelo podroben pregled raziskav o kakovosti peletov, izdelanih iz različnih virov gozdno-lesne in kmetijske biomase iz obdobja zadnjih petih let, podajajo Picchio et al. (2020).

Težave, povezane z rabo biomase za proizvodnjo peletov v energetske namene, se nanašajo na neprimerno obliko vstopne surovine, nizko nasutno gostoto in visoko vlažnost, kar povzroča probleme v obdelavi in lahko privede do degradacije med transportom in skladiščenjem ter posledično višje stroške proizvodnje (Puig-Arnavat et al., 2016). Na gospodarnost izdelave trdnih biogoriv ključno vpliva tudi razpršenost virov surovin, zato je nujno učinkovito in okolju prijazno obvladovanje sicer kompleksne biomasne oskrbovalne verige (Puig-Arnavat et al., 2016).

S procesom peletiranja ali zgoščevanja biomase dosegamo homogeno trdno gorivo z visoko gostoto, nizko vsebnostjo vlage in povišano kurilno vrednostjo. Enostaven transport, manjši skladiščni prostor, enostavna uporaba (ogrevanje s peleti je popolnoma avtomatizirano) so dodatne prednosti peletov. Med trdnimi biogorivi je povpraševanje po lesnih peletih največje. Uporabljajo se tako v industrijskih napravah za proizvodnjo toplotne in električne energije, termičnih napravah za toplotno uplinjanje, kot tudi za neindustrijsko rabo v gospodinjstvih v majhnih kurilnih napravah (Stelte et al., 2012). Pri neindustrijski rabi morajo peleti imeti najvišjo kakovost.

Na končno kakovost peletov odločilno vplivajo vrsta in lastnosti vhodne surovine (npr. kemijska sestava, vlažnost, velikost gradnikov in njihova porazdelitev); način priprave surovine in pogoji pri proizvodnji peletov (npr. temperatura matrice, dimenzije matrice, podajalna hitrost, tlak pri peletiranju, itd.) (Obernberger & Thek, 2010).

Proces proizvodnje lesnih peletov lahko v grobem razdelimo na tri dele in sicer: (I) dobava in priprava vhodne surovine, (II) izdelava peletov in (III) distribucija proizvoda (GIS, 2020). Vsi deli imajo svoje značilnosti, ki jih je za doseganje ustrezne kakovosti potrebno poznati, obvladovati in dosledno upoštevati. Obdelavo oz. postopek izdelave peletov razdelimo na več faz (npr. mletje, sušenje, kondicioniranje, stiskanje oz. peletiranje, hlajenje, sejanje in embaliranje), katerih zaporedje je odvisno predvsem od vrste in oblike uporabljene vhodne surovine za proizvodnjo peletov (npr. hlodovina slabe kakovosti, sečni ostanki, kosovni ostanki iz predelave lesa, žagovina, lesni ostružki, oblanci, lesni sekanci …).

V proizvodnji peletov se danes največ uporablja les oziroma lesni ostanki iz žagarske in lesnopredelovalne industrije (npr. lesni prah, žagovina in drobni oblanci), saj je takšen les neoporečen in kemijsko neobdelan. Za žagovino so značilni majhni delci lesa, zato priprava surovine za nadaljnje peletiranje ni energetsko potratna. Velikokrat je žagovina iz žagarskih obratov brez lubja (ali drugih nečistoč), kar omogoča proizvodnjo visokokakovostnih peletov. Uporaba manj znanih drevesnih vrst za proizvodnjo peletov je povsem smiselna, pri čemer je ključno doseganje podobne ravni kakovosti peletov kot jo imajo peleti, izdelani iz lesnih vrst, ki se običajno uporabljajo v Evropi (npr. smreka, jelka, bukev) (Castellano et al., 2015).

Tujerodne invazivne drevesne vrste (TIDV) in grmovne vrste predstavljajo določen potencial za izdelavo peletov, še posebej tiste, ki so rasle v urbanem okolju (npr. mestni parki, drevoredi, vrtovi …) in imajo zaradi rastiščnih pogojev pogosto nižjo kakovost lesa in s tem omejeno področje uporabe.

Cilj pričujoče študije je oceniti primernost manjvrednih lesnih ostankov izbranih tujerodnih invazivnih drevesnih vrst za peletiranje. Čistim lesnim ostankom divjega kostanja, amerikanskega javora, robinije, trnate gledičevke in velikega pajesena smo določili gostoto, jih zmleli in kondicionirali na ciljno vlažnost, primerno za peletiranje v laboratorijskih pogojih. Pelete smo izdelali tako iz omenjenih tujerodnih invazivnih drevesnih vrst kot tudi mešanic TIDV in smreke v razmerjih 70 : 30 in 50 : 50. Vse postopke priprave surovine in peletiranja smo izvedli pod enakimi pogoji in parametri. Izdelanim peletom smo izmerili pomembnejše kazalnike kakovosti, ki jih določa standard SIST EN ISO 17225-2:2014 in sicer mehansko obstojnost, vsebnost vode, gostoto nasutja in vsebnost pepela. Postavili smo tri hipoteze; (I) lastnosti surovine se razlikujejo glede na izbrane TIDV in mešanice, (II) lastnosti izdelanih peletov se med izbranimi vrstami in mešanicami razlikujejo, (III) peleti, izdelani iz mešanice tujerodnih invazivnih drevesnih vrst in smrekovine bodo imeli boljše lastnosti kot peleti, izdelani izključno iz lesa tujerodne invazivne drevesne vrste.

2 MATERIALI IN METODE
2 MATERIALS AND METHODS

2.1 VHODNA SUROVINA
2.1 RAW MATERIALS

V raziskavi smo uporabili kosovne ostanke izbranih tujerodnih invazivnih drevesnih vrst, ki so bile proučevane v okviru projekta Applause (UIA02-228). Kot surovino za izdelavo peletov smo uporabili les divjega kostanja (*Aesculus hippocastanum)*, amerikanskega javora (*Acer negundo*), robinije (*Robinia pseudoacacia*), trnate gledičevke (*Gleditsia triacanthos*) velikega pajesena (*Ailanthus altissima*) in smrekovine (*Picea abies*). Značilnosti lesa uporabljenih drevesnih vrst so opisane v literaturi (Torelli, 2002; Gorišek et al., 2018; Gorišek et al., 2019; Merhar et al., 2020; Smolnikar, 2020). Lesne ostanke smrekovine smo uporabili za pripravo mešanic TIDV v dveh razmerjih.

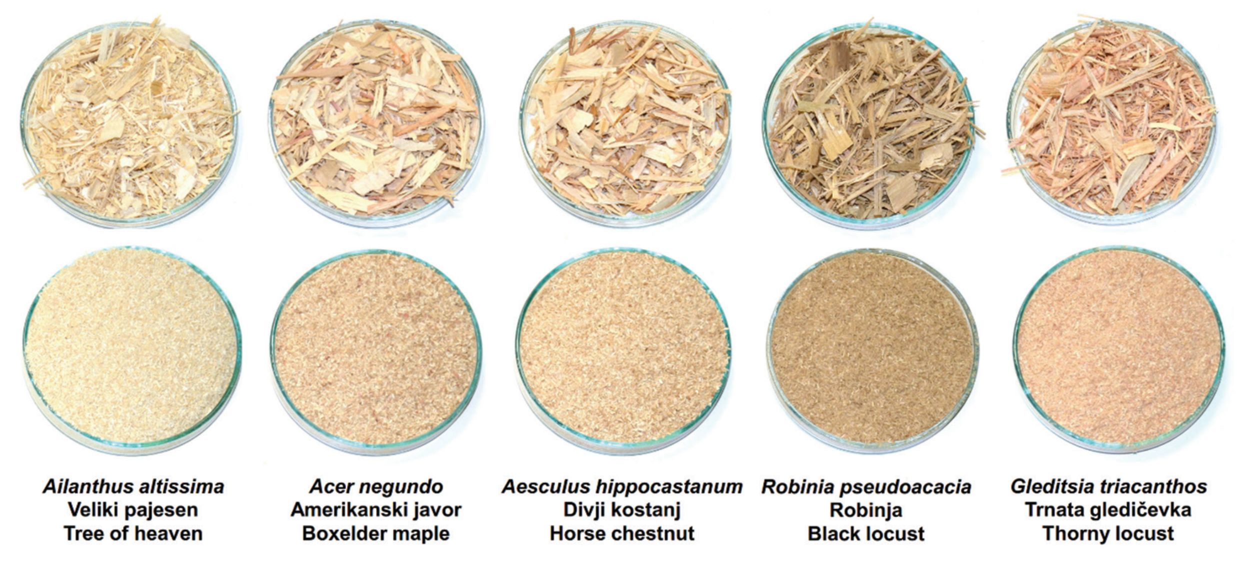
2.2 PRIPRAVA SUROVINE
2.2 SAMPLE PREPARATION

Ostanke masivnega lesa brez lubja vseh naštetih vrst smo razžagali na kose okvirnih dimenzij 20 mm × 20 mm × 30 mm in jih zmleli v dveh korakih (slika 1). Po opravljenem grobem mletju na laboratorijskem mlinu CONDUX CSK 350/N1 smo fino mletje izvedli na mlinu Retsch SM200. Pri finem mletju smo uporabili 2 mm sito za 2/3 volumna in 4 mm sito za 1/3 volumna vsake drevesne vrste. Mešanice tujerodnih invazivnih drevesnih vrst in smrekovine smo pripravili v dveh različnih volumskih razmerjih (50 % TIDV / 50 % smrekovine in 70 % TIDV / 30 % smrekovine) (preglednica 1). V nadaljevanju navajamo mešanice glede na delež tujerodne invazivne drevesne vrste. Mešanje zmlete surovine TIDV in smrekovine je potekalo ročno, v 10 l plastičnih vedrih. V vseh mešanicah smo uporabili enako volumsko razmerje velikosti gradnikov.

Vsem preučevanim TIDV smo volumetrično določili gostoto v absolutno suhem stanju pred fazo mletja na desetih naključno izbranih vzorcih in podali rezultat kot povprečje meritev.

Analizo vsebnosti pepela v pripravljenih mešanicah (preglednica 1) smo izvedli skladno s SIST EN ISO 14775:2010 z uporabo peči Nabertherm LE 14/22 B 300. Segrevanje vzorcev smo izvedli po naslednjem temperaturnem režimu: 50 min enakomerno segrevanje do 250 °C, 60 min gretje na 250 °C, enakomerno povišanje temperature na 500 °C v 30 min intervalu in vzdrževanje temperature 500 °C nadaljnjih 120 min. Za vsako mešanico smo naredili po štiri meritve in podali povprečje meritev deleža pepela.

Pripravljenim kombinacijam surovin smo določili vsebnost vode na merilniku vlažnosti BEA-MA110-1 in po potrebi surovino navlažili do ciljne vsebnosti vode med 13 % in 15 %, kar je optimalna vrednost za peletiranje na uporabljeni peletirni napravi. Pred izdelavo peletov smo izvedli tudi fazo



Slika 1. Vzorci tujerodnih invazivnih drevesnih vrst po grobem in finem mletju.
Figure 1. Chipped and ground samples of non-native invasive tree species.

Preglednica 1. Surovinska sestava peletov.
Table 1. Raw material composition of pellets.

Lesna vrsta / Species	Tujerodna invazivna vrsta : Smrekovina / Non-native invasive Species: Spruce		
	100 % : 0 %	70 % : 30 %	50 % : 50 %
Veliki pajesen / Tree of heaven	✓	✓	✓
Divji kostanj / Horse chestnut	✓	✓	✓
Trnata gledičevka / Thorny locust	✓	✓	✓
Robinija / Black locust	✓	✓	✓
Amerikanski javor / Boxelder maple	✓	✓	✓

kondicioniranja in sicer tako, da smo mešanice 20 min pred peletiranjem enakomerno poškropili z vodo, jih temeljito ročno premešali in tako dosegli navlažitev površine gradnikov.

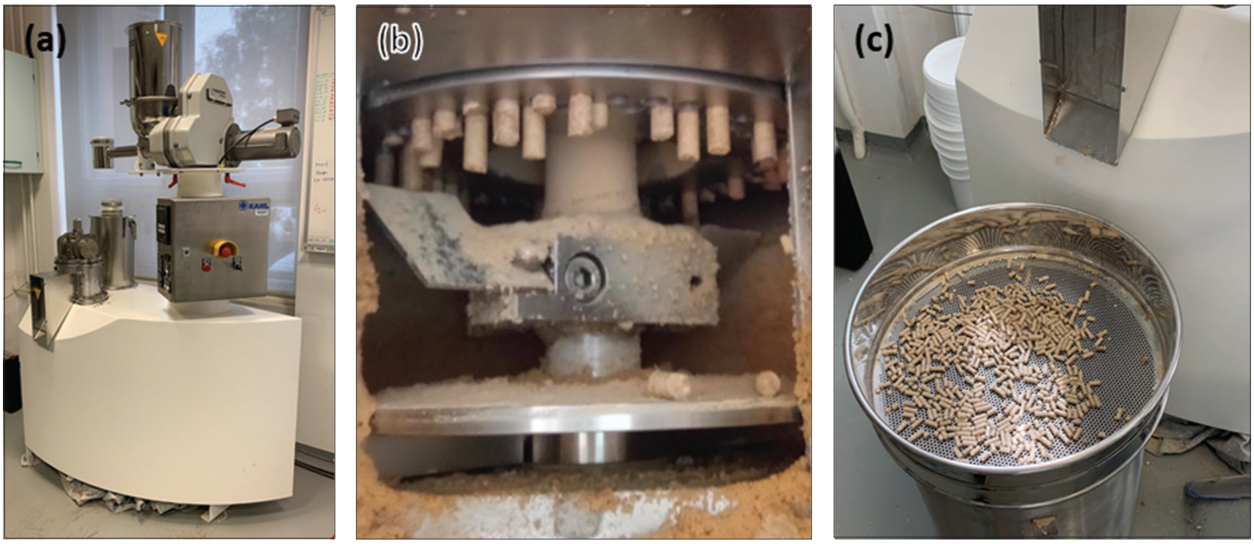
2.2 PROIZVODNJA PELETOV
2.2 PELLET PRODUCTION

Pelete smo izdelali na laboratorijski peletirni napravi Kahl 14-175 (slika 2a) z matrico v obliki diska (plošče), s kanali premera 6 mm, dolžine 22 mm, oziroma s stiskalnim razmerjem 0,27 (slika 2b). Podroben postopek peletiranja je opisal Smolnikar (2020). Med peletiranjem smo natančno beležili pogoje peletiranja (temperaturo matrice, vrtilno hitrost dozirnega polža in maso izdelanih peletov v časovnih intervalih). Pelete smo po vsakem stiskanju ohladili na rešetki (slika 2c).

2.3 ANALIZA PELETOV
2.3 ANALYSIS OF PELLETS

Vsem izdelanim peletom smo skladno s standardom SIST EN ISO 17225-2:2014 določili naslednje lastnosti:

- Vsebnost vode po standardni gravimetrični metodi, opredeljeni v standardu SIST EN ISO 18134-1:2015 ter z merilnikom vlažnosti BEAMA110-1.
- Gostoto nasutja, skladno s postopkom, opredeljenim v standardu SIST EN ISO 15103:2010, s tem, da smo namesto 5 l posode uporabili posodo z volumnom 0,5 l.
- Mehansko obstojnost skladno s standardom SIST EN ISO 15210:2010. Postopek določanja mehanske obstojnosti temelji na kontrolirani obrabi pelet. Med testiranjem peleti trkajo



Slika 2. Laboratorijska peletirna naprava Kahl 14-175 (a), proizvodnja peletov (b) in ohlajevanje peletov (c).
Figure 2. Laboratory pellet press Kahl 14-175 (a), pellet production (b) and cooling of the pellets (c).

drug ob drugega ter ob stene testirne naprave, katere delovanje in oblika je natančno opredeljena v standardu SIST EN ISO 15210-1:2010. Manjša obraba peletov pomeni manjšo količino nastalih finih delcev in posledično boljšo mehansko obstojnost. Za vsako vrsto (mešanico) peletov (preglednica 1) smo mehansko obstojnost določili dvema vzorcema, in sicer peletom, ki smo jih izdelali po približno 10 min peletiranja, in peletom, ki smo jih naredili na koncu peletiranja. Rezultati mehanske obstojnosti za posamezno mešanico so podani kot povprečje obeh meritev.

3 REZULTATI IN RAZPRAVA

3 RESULTS AND DISCUSSION

V raziskavi smo ugotavljali, ali so lesni ostanki tujerodnih invazivnih drevesnih vrst primerni za peletiranje in kakšna je kakovost peletov, če ne spreminjamo pogojev priprave surovine in peletiranja glede na drevesno vrsto. Peletiranje smo izvajali v laboratorijskih pogojih na laboratorijski peletirni napravi in kakovost peletov določali s standardnimi metodami.

3.1 LASTNOSTI IN PRIPRAVA VHODNE SUROVINE

3.1 PROPERTIES AND PREPARATION OF RAW MATERIALS

Eden ključnih dejavnikov, ki vplivajo na kakovost peletov, je homogena in ustrezno pripravljena vhodna surovina. V raziskavi smo različne oblike lesnih ostankov tujerodnih invazivnih drevesnih vrst razžagali na enotne kose in jih nato v dveh korakih zmleli do želene velikosti gradnikov. S sejalno analizo smo določili velikostne razrede gradnikov, kar je predstavil Smolnikar (2020). Ciljna velikost gradnikov je odvisna od načina peletiranja oziroma izvedbe matrice in peletirne naprave (Obernberger & Thek, 2010; Döring, 2013). V naši raziskavi so bile ciljne dimenzije gradnikov v velikostnem razredu med 2 mm in 4 mm.

V raziskavi smo izdelali pelete iz 15 različnih vhodnih surovin oziroma mešanic (preglednica 1). Za vsako mešanico smo pripravili cca 16 dm^3 surovine s ciljno vsebnostjo vode 13 % (realno izmerjene med 13 % in 15 %) in ustrezno sestavo gradnikov ter s temeljitim mešanjem in uravnovešanjem zagotovili homogenost vzorca. Različni raziskovalci navajajo različne ciljne vlažnosti za različne vrste vstopne surovine: med 8 % - 12 % (Obernberger & Thek, 2010) med 6 % - 13 % (Whittaker & Shield, 2017), Lehtinkangas (2001) navaja za smrekovino vlažnost med 13 % - 15 %. Optimalno vlažnost je potrebno določiti za vsako vrsto vhodne surovine, saj pomembno vpliva na lastnosti izdelanih peletov kot je npr. mehanska obstojnost. Višja vlažnost surovine namreč zmanjšuje trenje v procesu peletiranja in povečuje relaksacijo peletov po izdelavi in s tem negativno vpliva na mehansko obstojnost. Po drugi strani pa višja vlažnost znižuje temperaturo steklastega prehoda (Tg) lignina, kar pospešuje povezovanje gradnikov. Glede na dejstvo, da je Tg pri listavcih na splošno nekoliko višja kot pri iglavcih, ima višja vlažnost surovine lahko pozitiven vpliv na mehansko obstojnost peletov (Stelte, 2011), kar smo upoštevali v naši raziskavi.

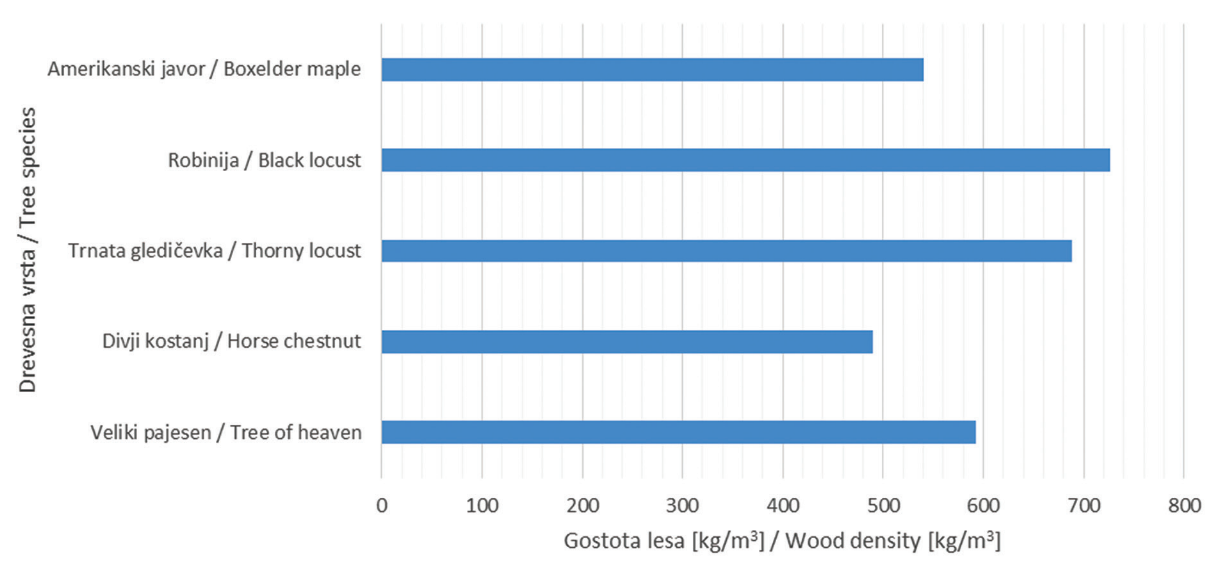
Raziskali smo tudi vpliv gostote lesa na potek peletiranja. Variabilnost gostote med izbranimi invazivnimi vrstami je razmeroma velika, najvišje gostote so imeli vzorci robinije (726 kg/m^3), najnižje pa divjega kostanja (490 kg/m^3) (slika 3). Vpliv gostote lesa na hitrost doziranja in temperaturo matrice komentiramo v poglavju 3.2.

Rezultati analize vsebnosti pepela v pripravljeni surovini za izdelavo peletov kažejo, da imajo najnižjo vsebnost pepela peleti, izdelani iz 100 % lesa robinije (delež pepela je 0,30 %) ter njeni 70 % in 50 % mešanici s smrekovino (slika 4). Omenjeni peleti izpolnjujejo kriterije standarda za uvrstitev v najvišji kakovostni razred A1, ki predpisuje zgornjo mejo 0,7 %. Najvišjo vsebnost pepela pa imajo peleti, izdelani iz lesa amerikanskega javorja, in sicer 1,35 % ter njegova 70 % mešanica s smrekovino (slika 4).

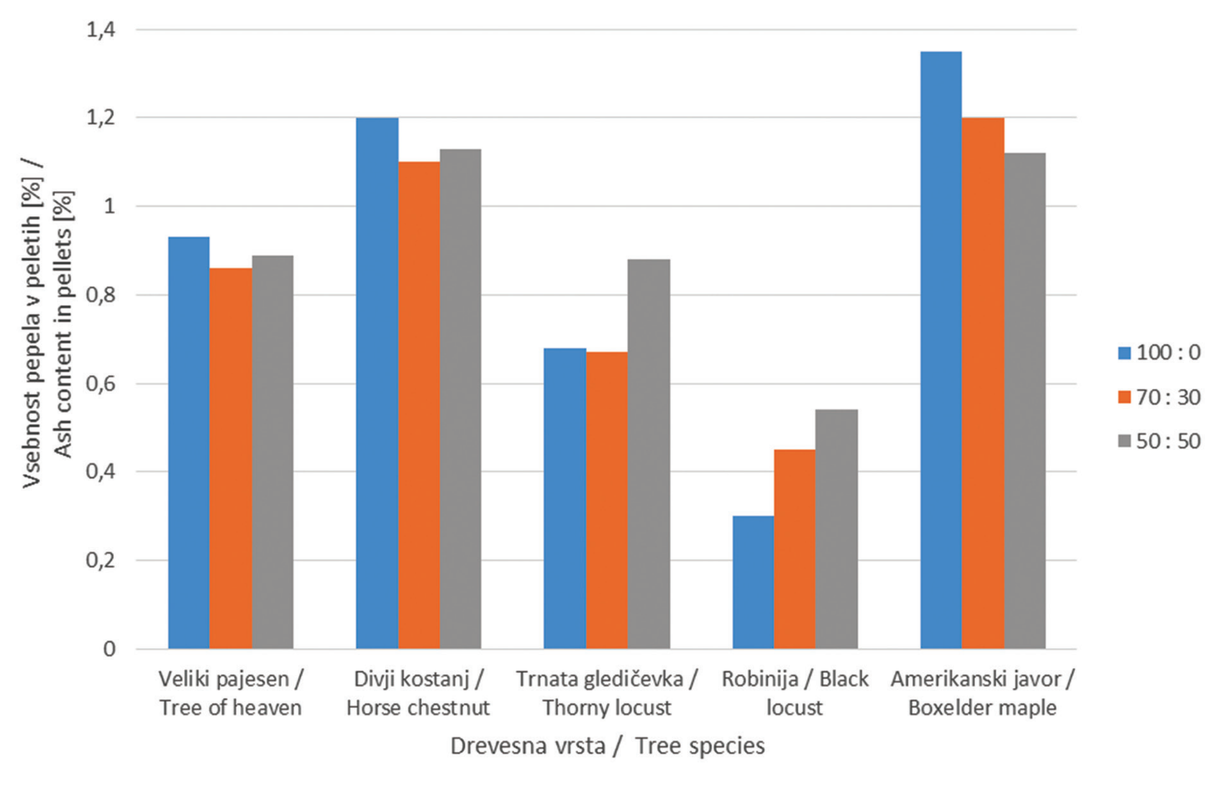
3.2 ANALIZA POGOJEV PELETIRANJA

3.2 CONDITIONS OF PELLET ANALYSIS

Za kakovost peletov je ključnega pomena ustrezna hitrost peletiranja, ki jo uravnavamo s hitrostjo doziranja vhodne surovine in vrtilno hitrostjo gnanih valjev. Vrtilna hitrosti valjev na matrici je bila v naši raziskavi konstantna, spreminjali pa smo vrtilno hitrost dozirnega polža. To smo konstantno nadzorovali in jo prilagajali glede na potek peletiranja. Vrtilno hitrost dozirnega polža smo postopno povečevali iz začetnih 50 vrt/min po 10 vrt/min do končnih 260 vrt/min. V primeru, da je poraba električnega toka



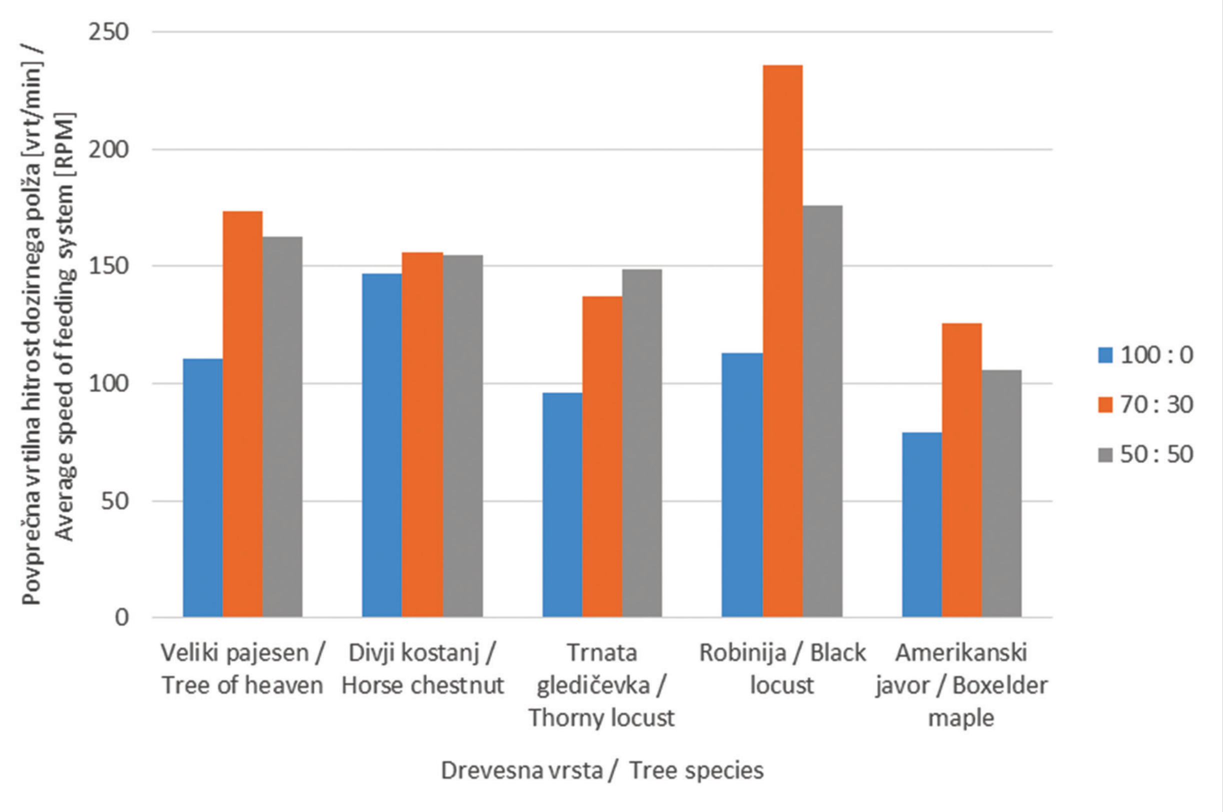
Slika 3. Gostote lesa izbranih tujerodnih invazivnih drevesnih vrst.
Figure 3. Density of selected non-native invasive tree species.



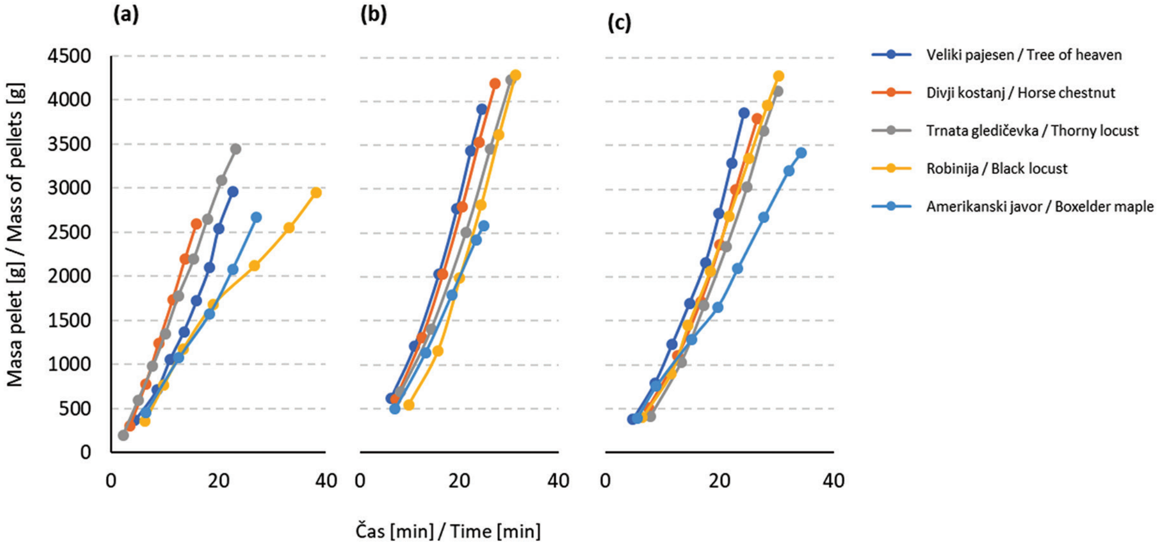
Slika 4. Vsebnost pepela v peletih izbranih TIDV in mešanicah.
Figure 4. Ash content in pellets of selected non-native invasive species in mixtures.

presegla normalno (nazivno) vrednost, smo vrtilno hitrost dozirnega polža zmanjšali in na ta način preprečili zabitje matrice. Povprečne hitrosti doziranja pri posameznih mešanicah prikazuje slika 5. Pri 100 % TIDV je prišlo do najvišje povprečne vrtilne hitrosti dozirnega polža pri divjem kostanju, kar bi lahko pripisali nizki gostoti surovine. Po zgornji trditvi bi pričakovali, da bo vrtilna hitrost z večanjem deleža smreke v mešanicah (70 % in 50 %) višja, kar lahko potrdimo pri mešanicah lesnih vrst divjega kostanja, trnate gledičevke in amerikanskega javorja.

Na sliki 6 je prikazana količina proizvedenih peletov v odvisnosti od časa, kar smo v naši raziskavi opredelili kot hitrost peletiranja različnih vrst surovine. Ta parametra nam lahko služita za kvalitativno primerjavo poteka peletiranja različnih surovin (Gornik Bučar et al., 2019). Iz vsake mešanice surovine smo naredili med 2,5 in 4,5 kg peletov. Hitrost peletiranja je vsekakor odvisna od uporabljene peletirne naprave, značilnosti vstopne surovine in pogojev peletiranja, in nikakor ne sme biti razumljena kot absolutna vrednost. Na podlagi hitrosti peletiranja lahko sklepamo o »enostavnosti« oziroma »težavnosti« peletiranja določene surovine. Če smo pri določeni vrtilni hitrosti dozirnega polža zaznali preobremenitve elektromotorja gnanih valjev, smo dotok surovine zmanjšali in s tem preprečili zamašitev matrice, kar je vplivalo tudi na količino proizvedenih peletov v določenem časovnem intervalu. Pri peletiranju čiste (100 % TIDV) surovine (slika 6a), smo najvišjo hitrost peletiranja (proizvedena masa peletov v enoti časa) dosegli pri divjem kostanju (167 g/min) najnižjo pa pri robiniji (78 g/min), kljub temu, da ima največjo gostoto med izbranimi vrstami. V primeru peletiranja 70 % mešanic (slika 6b) je bila največja hitrost peletiranja pri visokem pajesenu (160 g/min), sledi divji kostanj, najnižjo pa ima amerikanski javor (104 g/min), ki odstopa od ostalih. Zanimivo je, da je ob mešanju TIDV s smrekovino pri nekaterih primerih prišlo do povišanja hitrosti peletiranja (visoki pajesen, robinija in amerikanski javor) pri nekaterih pa do znižanja (divji kostanj in trnata gledičevka). Podobna opa-



Slika 5. Povprečna vrtilna hitrost dozirnega polža pri peletiranju izbranih TIDV in mešanicah.
Figure 5. Average speed of feeding system for pelleting of selected non-native invasive tree species inmixtures.



Slika 6. Količina proizvedenih peletov, izdelanih iz: (a) 100 % lesa TIDV, (b) 70 % mešanic in (c) 50 % mešanic.
Figure 6. Quantity of produced pellets during pelletization produced from: (a) 100% of non-native invasive tree species, (b) 70% of non-native invasive tree species and (c) 50% of non-native invasive tree species.

žanja kot pri 70 % mešanicah opazimo tudi pri 50 % mešanicah (slika 6c). Najvišjo hitrost peletiranja ima ponovno veliki pajesen (160 g/min), najnižjo pa amerikanski javor (100 g/min).

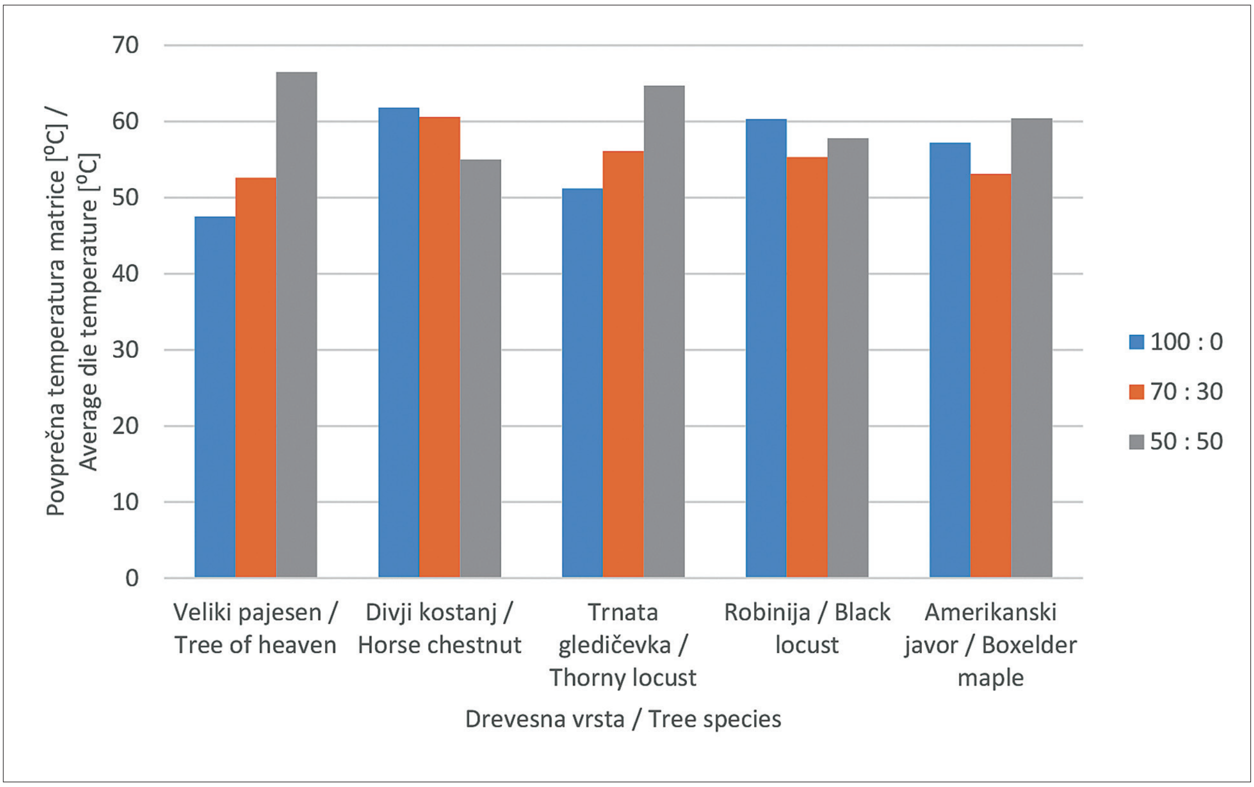
Med peletiranjem oz. zgoščevanjem (ang. densification) biomasne surovine se med gradniki in stenami matrice zaradi trenja generira toplota, ki je ključnega pomena za kakovost peletov. Optimalna temperatura, ki omogoča izdelavo najkakovostnejših peletov, je odvisna od vrste biomase in načina priprave biomase, njene kemijske sestave in vsebnosti vode kot tudi značilnosti peletirne naprave. Zgoščevanje biomase je torej zelo kompleksen proces, na katerega imajo vpliv številni dejavniki, ki se v večini primerov določajo s poskušanjem (ang. »Trial and error«) in izkustvenimi spoznanji, kar je časovno zelo potratno (Pugi-Arnavat, 2016). Temperature peletiranja se glede na vrsto biomase in peletirne naprave gibljejo v širokem območju med 20 °C in 120 °C (Pugi-Arnavat, 2016). Kot je razvidno iz slike 7, se je povprečna temperatura matrice oz. peletiranja v naši raziskavi gibala med 50 °C in 67 °C, kar se odraža tudi v relativno nizki mehanski obstojnosti peletov (slika 11). Naj pri tem poudarimo, da je raziskava preliminarna in da smo želeli proizvesti pelete iz različnih TIDV oziroma mešanic, pod kar se da enakimi pogoji, tako pri pripravi surovine kot peletiranju. Glede na to bi bilo smiselno v nadaljnjih raziskavah izvajati peletiranje pri višjih temperaturah matrice.

3.3 LASTNOSTI PELETOV
3.3 PROPERTIES OF PELLETS

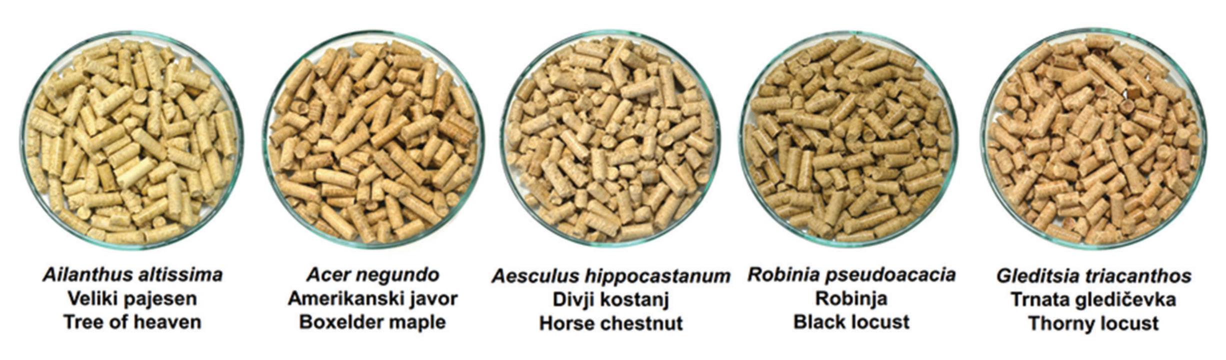
Pri lesni biomasi se vlažnost običajno podaja kot voda v lesu (preračunana glede na mokro maso), kar pomeni, da govorimo o absolutni vlažnosti (Prislan et al., 2020). Vsebnost vode odločilno vpliva na potek peletiranja kot tudi na kalorične vrednosti peletov. Vsi izdelani peleti (slika 8) so imeli povprečno vsebnost vode pod 10 % (slika 9), kar izpolnjuje kriterije standarda za uvrstitev v najvišji kakovostni razred peletov A1. Meritve povprečne vsebnosti vode, izmerjene po metodi, opisani v standardu SIST EN ISO 18134-1:2015, se gibajo med 5,8 % in 7,8 %. Najnižjo vsebnost vode smo izmerili za čisto mešanico trnate gledičevke, najvišjo pa pri 50 % mešanici robinije.

Povprečne gostote nasutja izdelanih peletov (slika 10) variirajo od 611 kg/m^3 do 703 kg/m^3 in ustrezajo zahtevam za kakovostni razred pelet A1, kot je določeno v standardu SIST EN ISO 17225-2:2014, ki navaja mejno vrednost 600 kg/m^3. Najvišjo gostoto nasutja je imela 70 % mešanica trnate gledičevke, najnižjo pa 70 % mešanica amerikanskega javorja. Gostota nasutja peletov je odvisna tako od gostote peletov kot tudi velikosti vmesnih prostorov med posameznimi peleti. Večja kot je gostota nasutja peletov, večja je količina akumulirane energije na prostorninsko enoto, posledično pa so transportni in skladiščni stroški nižji (Obernberger & Thek, 2010).

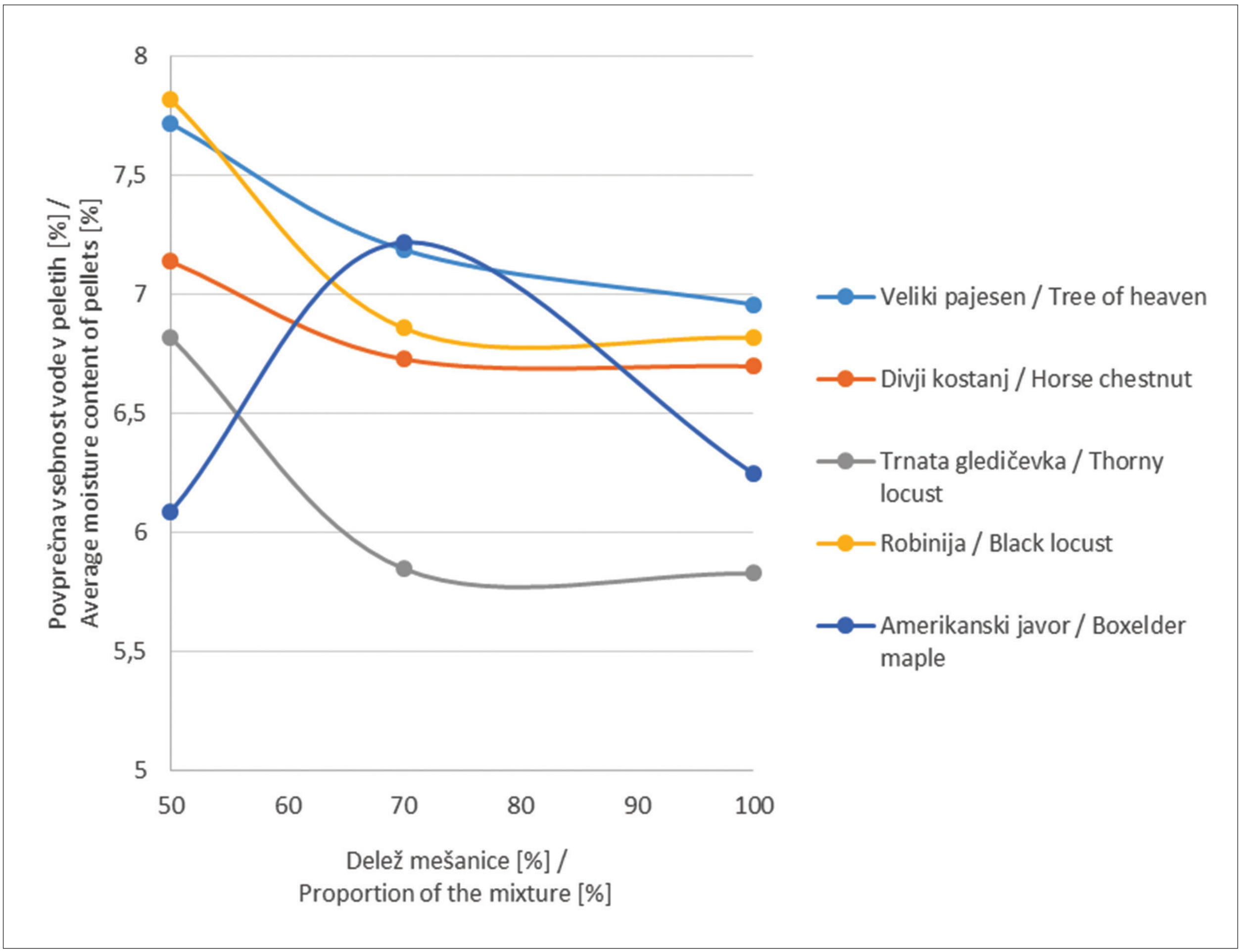
Mehanska obstojnost je eden ključnih kakovostnih kazalcev peletov. Na mehansko obstojnost ima odločilen vpliv tako vhodna surovina, njena sestava in priprava kot tudi postopek peletiranja. Najvišjo povprečno mehansko obstojnost, ki je znašala 95 %, so dosegli peleti iz čiste (100 %) surovine trnate gledičevke, odstotek nižjo pa peleti,



Slika 7. Povprečna temperatura matrice med peletiranjem izbranih TIDV in mešanic.
Figure 7. Average die temperature for pelleting of selected non-native invasive species and mixtures.

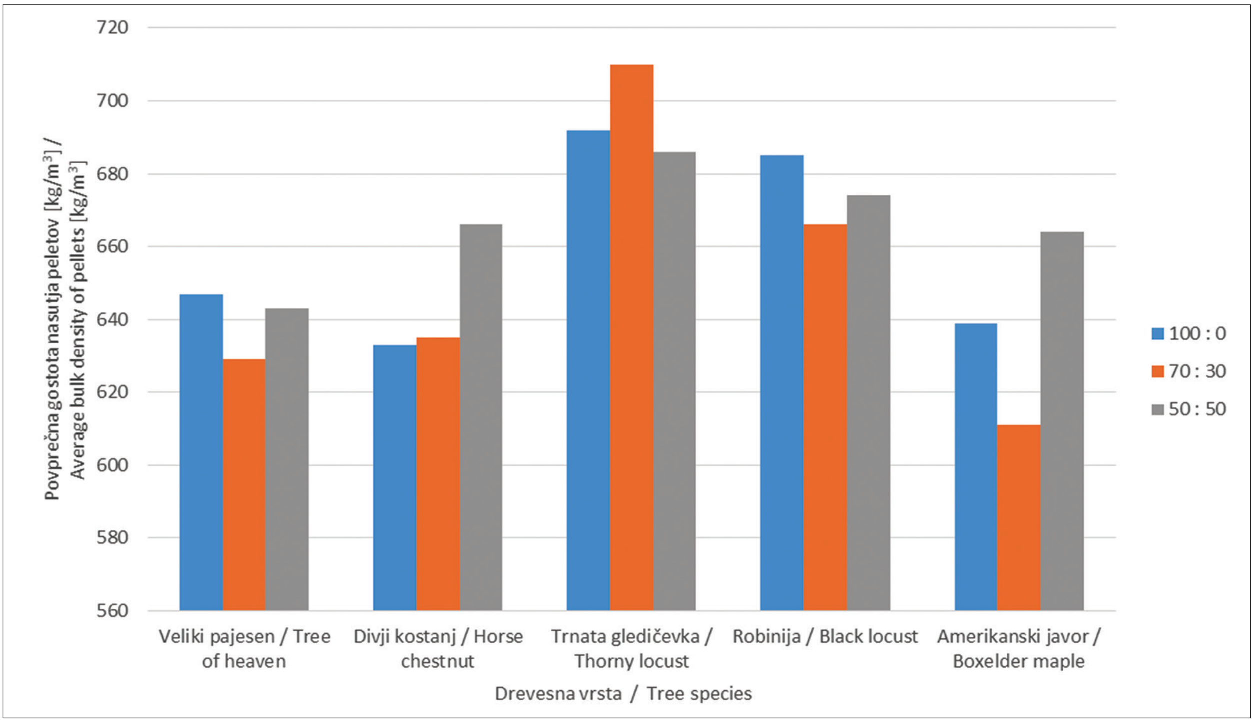


Slika 8. Peleti, izdelani iz izbranih tujerodnih invazivnih drevesnih vrst.
Figure 8. Pellets from selected non-native invasive tree species.

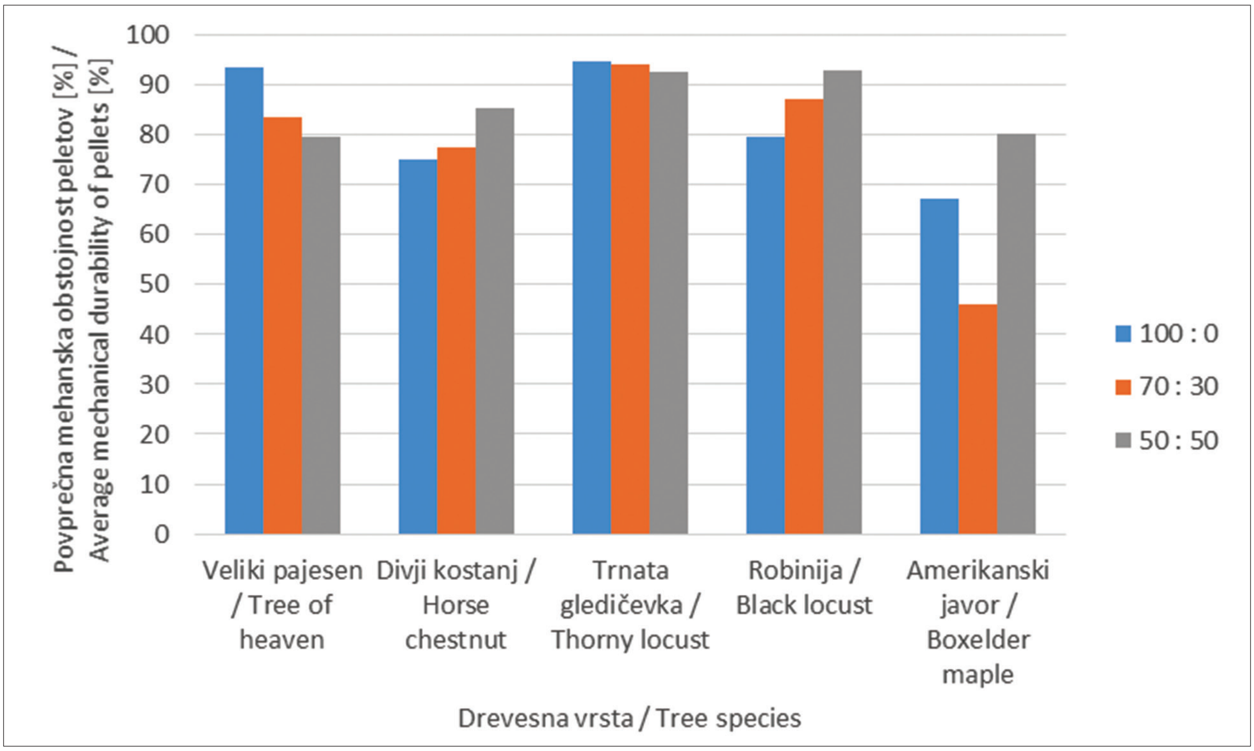


Slika 9. Povprečna vsebnost vode v peletih izbranih TIDV in mešanicah.
Figure 9. Average water content of pellets of selected non-native invasive species and mixtures.

izdelani iz velikega pajesena. Najslabšo mehansko obstojnost (46 %) so imeli peleti, izdelani iz 70 % mešanice amerikanskega javorja (slika 11). Mehanska obstojnost mešanic s smrekovino je bila v primeru velikega pajesena in trnate gledičevke z večanjem deleža smrekovine manjša; v primeru divjega kostanja in robinije pa večja kot pri čisti surovini. Takšni rezultati mehanske obstojnosti kot enega ključnih kazalnikov kakovosti peletov potrjujejo navedbe raziskovalcev (Stelte, 2011; Puig-Arnavat et al., 2016; Picchio et al., 2020), da je peletiranje zelo kompleksen proces, na katerega imajo vpliv tako surovina kot postopek, in mora biti optimiziran za posamezno vrsto biomasne surovine. Nizka mehanska obstojnost vodi do številnih nezaželenih dogodkov že med samo proizvodnjo peletov kot tudi med distribucijo, transportom, skladiščenjem in uporabo. Velike emisije prahu tako povzročajo moteno delovanje avtomatiziranih sistemov kot tudi nevarnost požara in eksplozije med prevozom ter skladiščenjem in imajo negativen vpliv na zdravje. Relativno nizko doseženo mehansko obstojnost izdelanih peletov lahko pripišemo tudi relativno nizki temperaturi matrice pri peletiranju, saj se med postopkom zgoščevanja ni generirala zadostna toplota, ki bi pozitivno vplivala na tvorjenje vezi med delci. Predvidevamo, da bi višja temperatura peletiranja imela pozitiven vpliv na mehansko obstojnost peletov. Izboljšanje mehanske trdnosti bi poleg povišane temperature peletiranja najverjetneje dosegli tudi z optimizacijo vrtilne hitrosti gnanih valjev in stiskalnim razmerjem, ki bi ga dosegli z izbiro druge debeline matrice.



Slika 10. Povprečna gostota nasutja peletov izbranih TIDV in mešanic.
Figure 10. Average bulk density of pellets of selected non-native invasive species and mixtures.



Slika 11. Povprečna mehanska obstojnost peletov izbranih TIDV in mešanic
Figure 11. Average mechanical durability of pellets made from selected non-native invasive species and mixtures.

4 ZAKLJUČKI
4 CONCLUSIONS

Raba lesne biomase v energetske namene še vedno predstavlja določen izziv, tako iz okoljskega kot tudi gospodarsko-ekonomskega stališča. Z zgoščevanjem lesne biomase (peletiranjem) dosegamo večjo gostoto energije, izboljšamo pa tudi enostavnost transporta, skladiščenja in rokovanja s trdnim gorivom. Pri izdelavi peletov je nujno upoštevati pogoj, da se za ta namen uporabi samo lesna masa, ki nima druge možnosti uporabe, kajti raba v druge namene podaljšuje krožni gospodarski cikel (Zule et al., 2017), poleg tega pa je potencialna dodana vrednost v nadaljnjih predelavah v primeru rabe lesa kot energenta nizka (Kropivšek & Gornik Bučar, 2017). Zaradi vse večje rabe peletov, katerih vstopna surovina je les in pa ostanki iz lesnopredelovalne industrije, se iščejo možnosti uporabe različnih drevesnih in grmovnih lesnih vrst, saj povečana raba lesa v energetske namene negativno vpliva tudi na ceno vhodne surovine. Eden od potencialnih virov je tudi manjvreden les tujerodnih invazivnih drevesnih vrst, ki rastejo predvsem v urbanem okolju in ima zaradi rastiščnih pogojev in slabše kakovosti omejeno področje uporabe. Tovrstna raba lesa TIDV bi lahko imela tudi pozitiven vpliv na omejevanje intenzivnosti širjenja le-teh, ker ogrožajo avtohtone drevesne vrste.

V laboratorijskih pogojih smo izdelali pelete iz izbranih tujerodnih invazivnih drevesnih vrst: divjega kostanja (*Aesculus hippocastanum)*, ameriškanskega javora (*Acer negundo*), robinije (*Robinia pseudoacacia*), trnate gledičevke (*Gleditsia triacanthos*) in velikega pajesena (*Ailanthus altissima*) ter avtohtone smreke (*Picea abies*), ki smo jo dodajali TIDV v različnih razmerjih. Izdelanim peletom smo določili nekatere pomembnejše fizikalne in mehanske lastnosti (vsebnost vode, nasipno gostoto, vsebnost pepela in mehansko obstojnost). Na osnovi rezultatov lahko zaključimo, da imajo od preučevanih tujerodnih invazivnih drevesnih vrst potencial za energetsko izrabo v obliki peletov predvsem robinija, trnata gledičevka in veliki pajesen. Ugotavljamo, da se nekatere uporabljene vrste »lažje« peletirajo (t.j. z višjo hitrostjo peletiranja ob nižjih temperaturah) in da to ni odvisno od gostote lesne vrste. Tako lahko na osnovi rezultatov prvo in drugo zastavljeno hipotezo potrdimo. Za potrditev oziroma zavrnitev hipoteze, da imajo peleti, izdelani iz lesnih ostankov izbranih tujerodnih invazivnih drevesnih vrst in smrekovine boljše lastnosti kot izdelani samo iz lesa TIDV, bi morali izvesti obsežnejšo raziskavo in optimizirati postopke izdelave.

Ker smo nekatere uporabljene tujerodne invazivne drevesne vrste za izdelavo pelet uporabili prvič in tudi v nam dostopni literaturi nismo našli podatkov o rabi v tovrstne namene, smo želeli pridobiti okvirne informacije o možnosti rabe izbranih TIDV za proizvodnjo peletov. Raziskava je preliminarna, zato naš cilj ni bil proizvesti peletov, ki izpolnjujejo zahteve standardov, kar tudi pomeni, da postopkov priprave surovine in peletiranja nismo optimizirali. Dobljeni rezultati in pridobljene izkušnje so zagotovo dragocene za nadaljnje raziskovanje možnosti rabe tovrstnega vira lesnih ostankov za proizvodnjo peletov.

5 POVZETEK
5 SUMMARY

The energetic use of wood biomass remains a challenge, both from an ecological and economic point of view. With the densification of wood biomass (pelletization), a higher energy density can be achieved, which can lead to optimized transport, storage and handling conditions of solid fuels. It should be emphasized that only low-quality wood biomass should be considered for this purpose in order to meet the requirements of a sustainable circular economy.

As the use of pellets continues to increase, the possibility of using various tree and shrub wood species is being explored in addition to traditional sources of raw materials such as the wood processing industry. One of the potential sources is also low-quality wood of invasive tree species, growing mainly in urban environments.

The objective of this preliminary study is to evaluate the suitability of low-quality wood residues from invasive non-native tree species for pelleting. We produced pellets from five selected invasive non-native tree species growing in Slovenia on a laboratory pelleting device, namely: wild chestnut (*Aesculus hippocastanum*), boxelder maple (*Acer negundo*), black locust (*Robinia pseudoacacia*), thorny locust (*Gleditsia triacanthos*) and tree of heaven (*Ailanthus altissima*), as well as mixtures of the raw materials from the above invasive alien species and spruce *(Picea abies*) in the ratios 70:30 and 50:50. Raw material preparation and pelleting procedures were carried out under the same conditions and parameters. The laboratory Kahl press (flat die with 0.27 press ratio) was used for pelleting, and the feed rate as well as the temperature at the die were continuously recorded during the pelleting process.

The main quality parameters of the pellets produced were measured (i.e., water content, bulk density and mechanical durability). The methods and procedures for determining the properties of the pellets were carried out according to the relevant standards.

The results of the tested pellet properties were compared with the limits for quality class A1, A2 and B defined in the international standard SIST EN ISO 17225-2:2014. All types of pellets produced met the requirements of the standard for classification in the highest quality class A1 in terms of water content and bulk density. The mechanical durability of the pellets did not meet the requirements of the standard and did not exceed 96.5% (which is the limit for class B). We conclude that the reason for the low mechanical durability is the relatively low temperature of the die during pelleting. We find that the type of input raw material and the pelleting conditions affect the quality of the pellets, especially the mechanical durability. The results suggest that black locust, thorny locust and tree of heaven have the highest potential for further optimization of the pelleting process. We have observed that some of the wood species studied have a higher pelletization rate at lower temperatures, but this is not related to the density of the wood species.

Since we used some of the non-native invasive tree species for the first time for pellet production and did not find data on their use in the relevant literature, we evaluated the possibility of using selected invasive species for pellet production. As this is a preliminary study, it was not our aim to produce pellets that meet the requirements of the standards, which also means that we have not optimized the raw material preparation and the pelleting process. The results obtained and the experience gained are certainly valuable for further research into the possibilities of using such a source of wood residues for pellet production.

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FORMATION OF SILVER NANOPARTICLES ON LIGNIN AND TWO OF ITS PRECURSORS

TVORBA SREBROVIH NANODELCEV NA LIGNINU IN NJEGOVIH DVEH PREKURZORJIH

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Abstract / Izvleček

Abstract: *Metastable Induced Electron Spectroscopy, Ultraviolet Photoelectron Spectroscopy (He I and He II), X-ray Photoelectron Spectroscopy, and Atomic Force Microscopy were employed to study the interaction of silver with lignin as well as with two of its natural precursors, coniferyl alcohol and sinapyl alcohol. For all three of them, no chemical interaction between the adsorbed silver and the organic substrate was found before contact with air. Nevertheless, silver nanoparticles were found in all three cases after contact with air. Thus, a process of silver nanoparticle formation during the decomposition of the organic molecules is suggested, similar to the previously found catalytic decomposition of cinnamyl alcohol by water in the presence of silver atoms.*

Keywords: *Coniferyl Alcohol; Lignin; Metastable Induced Electron Spectroscopy; Nanoparticle; Silver; Sinapyl Alcohol; Ultraviolet Photoelectron Spectroscopy; X-Ray Photoelectron Spectroscopy; Atomic Force Microscopy*

Izvleček: *Za preučevanje interakcije srebra z ligninom ter z njegovima naravnima prekurzorjema koniferil alkoholom in sinapil alkoholom smo uporabili metastabilno elektronsko spektroskopijo, ultravijolično fotoelektronsko spektroskopijo (He I in He II), rentgensko fotoelektronsko spektroskopijo in mikroskopijo na atomsko silo. V vseh treh primerih nismo ugotovili kemijske interakcije med adsorbiranim srebrom in organskim substratom. Kljub temu pa smo v vseh treh primerih po stiku z zrakom detektirali nanodelce srebra. Na podlagi teh opažanj smo predlagali proces tvorbe srebrovih nanodelcev med razgradnjo organskih snovi, ki je podoben predhodno ugotovljenemu katalitskemu razkroju cinamil alkohola z vodo v prisotnosti atomov srebra.*

Ključne besede: *koniferil alkohol; lignin; metastabilna elektronska spektroskopija; nanodelci; srebro; sinapil alkohol; ultravijolična fotoelektronska spektroskopija; rentgenska fotoelektronska spektroskopija; mikroskopija na atomsko silo*

1 UVOD
1 INTRODUCTION

The preservation of wood structures against all kinds of aging and degradation is a highly topical area of research around the world. The improvement of hitherto existing processes and the development of new ones with higher efficiencies and better sustainability are currently being investigated, employing a variety of chemical and physical means. Numerous novel materials (Maggini et al., 2012), different forms of modifications like thermal treatment (Calonego et al., 2012) or chemical bonding (Namyslo & Kaufmann, 2009), as well as the use of nanostructures, were already studied and presented in the literature (Ding et al., 2011). Even though the classic preservation approach using chromated copper arsenate (CCA) or copper chrome boric acid (CCB) is designated hazardous, similar approaches with reduced leaching (Treu et al., 2011; Lesar et al., 2011) and reutilization (Humar et al., 2011) have been investigated.

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The modification of inorganic materials employing silver is carried out for many applications, like corrosion protection, RF shielding, reflective coatings and many more. Silver nanoparticles have drawn special interest due to additional features like enhancing the efficiency of organic light emitting devices (Yang et al., 2009) and photocatalytic reaction rates on TiO_2 nanoparticles (Kato et al., 2005; Chuang & Chen, 2009), as well as their antibacterial properties (Lok et al., 2006; Ilic et al., 2010). The last one may be particularly useful for the preservation of wood surfaces against the attack of microorganisms like bacteria and fungi, which are part of the main mechanisms of wood aging and degradation. Furthermore, a silver nanoparticle based functionalization may be much more sustainable than current procedures such as lacquering or impregnation.

The presented investigation is part of a research project concerning the interaction of metals (Ag, Ti) with wood surfaces. Based on the results obtained here, we will come closer to understanding such complex systems as the interactions of metals and wood. Furthermore, the reactions with atmospheric gases as well as typical volatile organic compounds are investigated, with a view to future applications. A fundamental understanding of the interactions between Ag and wood surfaces is developed, using measurements on the major compounds of wood, i.e. lignin and cellulose. While starting with lignin, several model systems are used which resemble the organic groups of lignin. The natural precursors of lignin are mainly the two monolignols coniferyl alcohol and sinapyl alcohol (Klarhöfer et al., 2007; Klarhöfer et al., 2008; Klarhöfer et al., 2010). Since these two are derived from cinnamyl alcohol (also known as phenylallyl alcohol), we have also used this less complex molecule as an additional model system for lignin in preliminary investigations (Dahle et al., 2012). The adsorbed silver was first found to be chemically inert, while nanoparticles were found after contact with air. Bringing the cinnamyl alcohol into contact with water after the adsorption of silver was then discovered to yield the decomposition of the cinnamyl alcohol (Dahle et al., 2014). Furthermore, the decomposition reaction has been proven to include the catalytical influence of silver atoms at their distinctive adsorption sites (Dahle et al., 2014). The effect of plasma treatment on the presented molecules was also studied previously (Klarhöfer, 2009).

2 MATERIALS AND METHODS

2 MATERIALI IN METODE

An ultra-high vacuum (UHV) apparatus with a base pressure of 5 × 10^{-11} hPa, which has been described in detail previously (Klarhöfer et al., 2008; Dahle et al., 2012), is used to carry out the experiments. All measurements were performed at room temperature.

Electron spectroscopy is performed using a hemispherical analyser (Leybold EA 10) in combination with a source for metastable helium atoms (mainly He* $^3S_1$) and ultraviolet photons (HeI line). A commercial non-monochromatic X-ray source (Fisons XR3E2-324) is utilized for XPS.

During XPS, X-ray photons hit the surface under an angle of 80° to the surface normal, illuminating a spot of several mm in diameter. For all measurements presented here, the Al K_α line with a photon energy of 1486.6 eV is used. Electrons are recorded by the hemispherical analyser with an energy resolution of 1.1 eV for detailed spectra and 2.2 eV for survey spectra, respectively, under an angle of 10° to the surface normal. All XPS spectra are displayed as a function of binding energy with respect to the Fermi level. For quantitative XPS analysis, photoelectron peak areas are calculated via mathematical fitting with Gauss-type profiles using CasaXPS (Casa Software Ltd., Florida, USA) with a Shirley-type background, which applies Levenberg-Marquardt algorithms to achieve the best agreement between experimental data and fit. To optimize our fitting procedure, Voigt profiles have been applied to various oxidic and metallic systems but for most systems the Lorentzian contribution converges to 0. Therefore all XPS peaks are fitted with Gaussian shapes. When calculating stoichiometries, the following variables are taken into account: photoelectric cross sections, as calculated by Scofield (1976) with asymmetry factors after Powell and Jablonski (2010a), taking into account asymmetry parameters after Reilman et. al. (1976) and

Jablonski (1995), as well as inelastic mean free paths from the NIST database (Powell & Jablonski, 2010b) (using the database of Tanuma, Powell and Penn for elementary contributions and the TPP-2M equation for molecules), and the energy dependent transmission function of our hemispherical analyser. As a quantitative measure, layer thicknesses d_{ox} were calculated for each step of preparation from the attenuation of the intensities of peaks of the material covered below the adlayer with the inelastic mean free paths in the adlayer λ taking into account the angle between surface normal and the direction of the emitted electrons θ = 10°, using the following formula (Ertl & Küppers, 1985)

$$d = \cos\theta \bullet \lambda \bullet \ln\frac{I_0}{I(d)}$$

MIES and UPS were performed applying a cold cathode gas discharge via a two-stage pumping system. A time-of-flight technique is employed to separate electrons emitted by He* (MIES) from those caused by HeI (UPS) interaction with the surface. The combined He*/HeI beam strikes the sample surface under an angle of 45° to the surface normal and illuminates a spot of approximately 2 mm in diameter. The spectra are recorded simultaneously by the hemispherical analyser with an energy resolution of 220 meV under normal emissions within 140 seconds.

MIES is an extremely surface sensitive technique probing solely the outermost layer of the sample, because the He* atoms typically interact with the surface 0.3 to 0.5 nm in front of it. This may occur via a number of different mechanisms depending on surface electronic structure and work function, as is described in detail elsewhere (Harada et al., 1997; Morgner, 2000; Ertl & Küppers, 1985). Only the processes relevant for the spectra presented here shall be discussed below, namely Auger Deexcitation and Auger Neutralization.

During Auger Deexcitation (AD), an electron from the sample fills the 1s orbital of the impinging He*. Simultaneously, the He 2s electron is emitted carrying the excess energy. The resulting spectra reflect the Surface Density of States (SDOS) directly. AD-MIES and UPS can be compared and allow a distinction between surface and bulk effects. AD takes place for all organic systems shown here.

Auger Neutralization (AN) occurs on pure and partly oxidized metal surfaces with work functions beyond about 3.5 eV, like silver surfaces, as long as the surface shows a metallic behaviour. As a result the impinging He* atom is ionized in the vicinity of the surface by resonant transfer (RT) of its 2s electron into unoccupied metallic surface states. Afterwards, the remaining He^+ ion is neutralized by a surface electron thus emitting a second surface electron carrying the excess energy. The observed electron spectrum is rather structureless and originates from a self-convolution of the surface density of states (SDOS).

All MIES and UPS spectra are displayed as a function of the electron binding energy with respect to the Fermi level, thus being able to compare MIES and UPS spectra more easily. Obviously, the binding energy scale is only valid for the AD process. Nevertheless, all spectra including structures originating in the AN process have also been displayed in this particular manner. The surface work function can be determined from the high binding energy onset of the MIES or the UPS spectra with an accuracy of ± 0.1 eV.

Atomic force microscopy (AFM) is applied to study the surface topography of the samples after silver adsorption and determine the size of the silver nanoparticles on lignin films. A Veeco Dimension 3100 SPM is used to perform the AFM measurements in tapping mode. Silicon cantilevers (NSC15 with Al backside coating from Micromasch) with resonance frequencies of about 325 kHz and spring constants in the range of 40 N/m are used. All images are recorded with a line-scan frequency of 1 Hz consisting of 512 × 512 pixels. SPIP (Image Metrology A/S) is employed for the depiction of the AFM images and the determination of the particle size. The RMS roughness calculations are performed according to ISO 4287/1.

The experiments on coniferyl alcohol and sinapyl alcohol were carried out on inert Au(111) substrates (Goodfellow, 99.999 %). These substrates were cleaned prior to the experiments by Ar^+ sputtering at 4 kV for 20 min and subsequent heating up to 1000 K. Coniferyl alcohol (Sigma-Aldrich Co., 98.0 %) and sinapyl alcohol (Sigma-Aldrich Co., technical grade 80.0 %) were evaporated in a preparation chamber (base pressure < 10^{-9} hPa) using a temperature controlled evaporator (Kentax TCE-BS). The preparation chamber is directly connected to the UHV chamber separated by an UHV valve. During the experiments, coniferyl alcohol has been evaporated at 75 °C for 5 min, leading to a film with a thickness of about 3 nm (Klarhöfer, 2009), whereas sinapyl alcohol has been evaporated at 60 °C for 5 min, leading to a film with a thickness of about 4 nm (Klarhöfer, 2009).

For the experiments on lignin, an Au/Mica substrate (Sigma-Aldrich, 200 nm Au 99.999 % layer on mica) was used to most closely reproduce the protocol according to Klarhöfer (2009). The Au/mica substate was etched using peroxymonosulfuric acid (from sulphuric acid ≥97% and hydrogen peroxide 30%, both Sigma-Aldrich) and rinsed with distilled water prior to the film preparation. Lignin (Sigma-Aldrich, organosolv lignin) was dissolved in dimethyl sulfoxide (DMSO, Sigma-Aldrich, ≥99.9 %), dispersed using an ultrasonic cleaning system and afterwards spin-coated for 120 s at 17500 rpm.

Silver (Sigma-Aldrich, 99%) was evaporated with a commercial UHV evaporator (Omicron EFM3) onto the samples. On a clean Si(100) target (Sigma-Aldrich, Si wafer, single-side polished, N-type with phosphorous dopant), oxide-free metallic silver films grow at a rate of 0.23 nm min^{-1} at room temperature when evaporated with an Ag^{+} ion flux of 1 µA at the fluxmeter of the EFM3. This flux is a measure for the number of Ag atoms moving towards the sample per second. The film growth rate for Ag has been estimated from the Si 2p peak attenuation in XPS, while the growth mode has been verified to be of the Frank-van der Merwe-type via AFM measurements.

3 RESULTS AND DISCUSSION

3 REZULTATI IN RAZPRAVA

The experiments on the interaction of silver with the three molecules described in the introduction, i.e. coniferyl alcohol, sinapyl alcohol and lignin, are presented following the increasing molecule size. The spectroscopic results are directly followed up with the corresponding microscopic images, whereas the discussion is done afterwards for the entire set of results. The silver films were prepared with 8 min of evaporation, leading to a film thickness of about 1.8 nm (Dahle et al., 2012). The only exception was made for lignin, where additionally a film of about 9 nm after 40 min of evaporation was produced according to the growth rate determined earlier (see sect. 2).

3.1 ADSORPTION OF SILVER ON CONIFERYL ALCOHOL

3.1 ADSORPCIJA SREBRA NA KONIFERIL ALKOHOL

Fig. 1 shows the MIES (left), UPS He I (middle) and UPS He II (right) spectra of a coniferyl alcohol film before (black lines) and after silver adsorption (red lines). The shoulder between 2 eV and 5 eV corresponds to p states of the ring system, which can most clearly be seen in the UPS He II spectrum. The intensity between 6 eV and 8 eV is mainly related to hydroxyl groups, whereas methoxyl groups cause multiple peaks between 6 eV and 12 eV (Kimura et al., 1981; Klarhöfer et al., 2008; Klarhöfer, 2009). These features are severely broadened in the MIES and UPS He I spectra, and can most clearly be seen in the UPS He II spectrum, again. For comparison, the corresponding molecular orbitals in benzene and phenol have been summarized in tab. 1 together with the respective binding energies in the valence band spectrum and the noted markings provided in the MIES and UPS spectra. The phenol molecular orbitals 14a' through 21a' are equal to the respective MOs in benzene, and are expected to remain similar through larger molecules including lignin. Moreover, MO 14a' in the marked region (d) includes phenolic hydroxyl groups, as opposed to those hydroxyl groups attached to aliphatic chains, which are more commonly found at lower binding energies.

The adsorption of silver onto the coniferyl alcohol film leads to a decrease in the work function from 4.5 eV to 4.1 eV and thus an increase of the background noise. With regard to this background signal, no significant change in the valence band states of the hydroxyl and methoxyl groups is visible. The increased intensity below 5 eV binding energy most probably does not belong to a change corresponding to the p states of the ring system, but can rather be attributed to silver structures originating from an AN process as described in sect. 2. The AN structure at 2.8 eV involves two electrons from the Ag 5s orbital during the deexcitation process (Dahle et al., 2012), whereas the structure around 7 eV involves one electron each from the

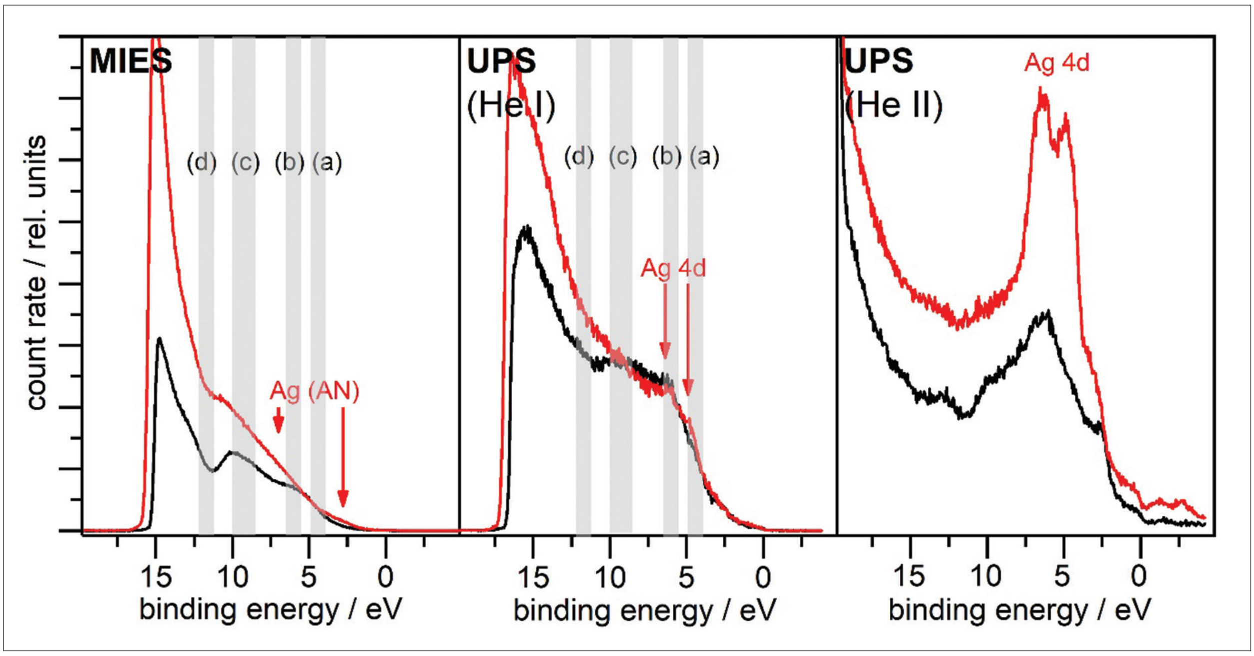

Figure 1. MIES (left), UPS He I (middle) and UPS He II (right) spectra of a coniferyl alcohol film before (black lines) and after silver adsorption (red lines).

Slika 1. MIES (levo), UPS He I (sredina) in UPS He II (desno) spektri filma koniferilnega alkohola pred (črne črte) in po adsorpciji srebra (rdeče črte).

Table 1. Binding energies of valence structures for molecular orbitals (MO) in benzene and phenol (Klarhöfer, 2009; Kimura et al., 1981).

Preglednica 1. Energije vezave valentnih struktur za molekularne orbitale (MO) v benzenu in fenolu (Klarhöfer, 2009; Kimura et al., 1981).

Mark	Binding energy	MO in benzene	MO in phenol
(a)	4.4 eV	$1e_{1g}$ (πCH)	3a”, 4a”
(b)	7.0 eV	$3e_{2g}$ (σCH) $1a_{2u}$ (πCH)	21a’, 20a’, 19a’ 2a”
(c)	8.5 – 10.5 eV	$3e_{1u}$ (σCH) $1b_{2u}$ (σCC) $2b_{1u}$ (σCH)	1a“, 18a‘, 17a‘, 16a‘
(d)	11.7 eV	$3a_{1g}$ (σCH)	15a’, 14a’

Ag 4d and Ag 5s states (Stracke et al., 2001). The UPS spectra reveal a double peak at 4.9 eV and 6.4 eV, which corresponds to the Ag 4d orbital (Dahle et al., 2012). The previous features from the coniferyl alcohol are still present, and except for slight broadening and attenuation the structures appear unaffected by the silver adsorption.

Fig. 2 depicts the XPS spectra of the C 1s region (left), the O 1s region (middle) and the Ag 3d region (right) of a coniferyl alcohol film before (black lines) and after silver adsorption (red lines). The C 1s region resembles the known spectrum for coniferyl alcohol (Klarhöfer, 2009). The intensity gets attenuated upon silver adsorption, following the signal of the underlying gold substrate (not shown). The shape of the C 1s feature does not change notably, which indicates the coniferyl alcohol to remain unaffected by the silver in the first place. The O 1s peak remains nearly unchanged in both, the shape and intensity. The Ag 3d feature is exactly like the

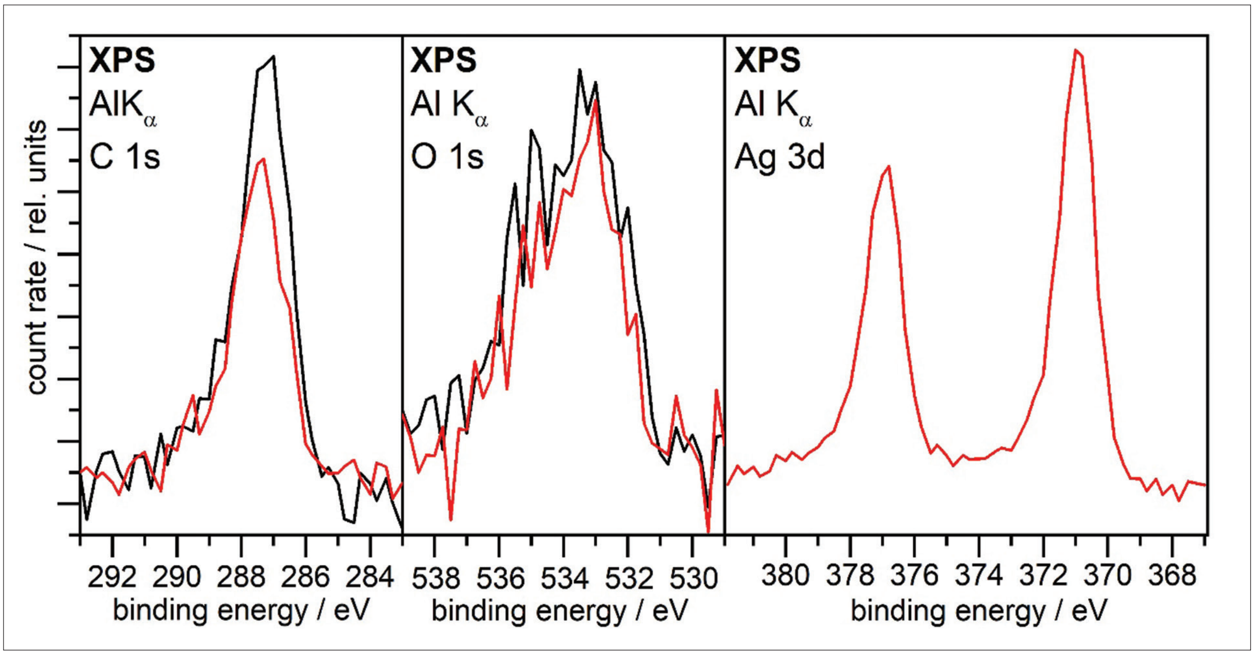

Figure 2. XPS spectra of the C 1s region (left), the O 1s region (middle) and the Ag 3d region (right) of a coniferyl alcohol film before (black lines) and after silver adsorption (red lines).
Slika 2. XPS spektri območja C 1s (levo), območja O 1s (sredina) in območja Ag 3d (desno) filma koniferilnega alkohola pred (črne črte) in po adsorpciji srebra (rdeče črte).

Table 2. Sample stoichiometries and film thicknesses evaluated from XPS survey spectra.
Preglednica 2. Vzorčne stehiometrije in debeline filma, ovrednotene iz XPS anketnih spektrov.

Sample system	Carbon	Oxygen	Silver	C/O ratio	Adlayer thickness
Coniferyl alcohol	75.9 %	24.1 %	-	3.16	1.3 nm
Silver on Coniferyl alc.	51.1 %	24.8 %	24.1 %	2.06	0.8 nm
Sinapyl alcohol	75.7 %	24.3 %	-	3.11	0.4 nm
Silver on Sinapyl alc.	51.4 %	9.8 %	38.8 %	5.22	0.6 nm
Lignin	77.1 %	22.9 %	-	3.37	-
Silver on Lignin	69.1 %	18.5 %	12.4 %	3.73	0.7 nm
Silver on Lignin	46.9 %	9.1 %	44.0 %	5.15	0.9 nm

shape of metallic silver (Dahle et al., 2012), pointing out the absence of any complexation processes or other chemical interactions with the coniferyl alcohol film. The film thickness evaluation from the attenuation of the Au substrate's intensity yields an organic adlayer thickness of 1.3 nm, whereas a silver adlayer thickness of 0.8 nm can be calculated from the attenuation of the coniferyl adlayer's intensity. The silver adlayer thickness is in discrepancy with the calibrated evaporation amounts. Further potential influences are a difference in sticking coefficients as well a possible desorption of organic material induced by the absorption of the silver atoms. An absorption of silver into the organic film is further sustained by a change in C/O ratio from 3.2 before silver adsorption to 2.1 afterwards, whereas the conservation of peak shapes for both C 1s and O 1s indicate no chemical change of the material. Thus, a geometrical rearrangement of the organic film upon silver adsorption can be deduced. The stoichiometries of the samples, C/O ratios and calculated film thicknesses are provided in tab. 2,

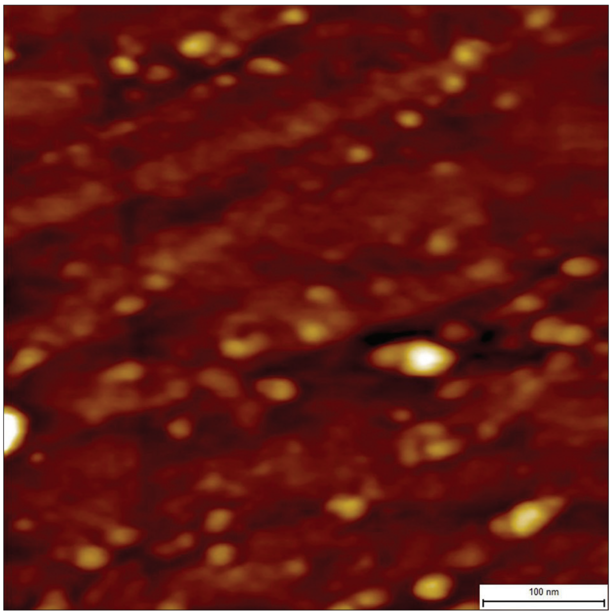

Figure 3. AFM image (500 × 500 nm^2) of a coniferyl alcohol film on Au(111) after silver adsorption and air contact.

Slika 3. AFM-slika (500 × 500 nm^2) filma koniferilnega alkohola na Au (111) po adsorpciji srebra in stiku z zrakom.

whereas the complete set of original and analysed data are made openly available online (Dahle et al., 2021).

Fig. 3 shows an AFM image of adsorbed silver on a film of coniferyl alcohol, which has been exposed to ambient air. The surface is very smooth (R_q = 0.34 nm) except for sporadic particles, probably representing silver clusters. Those islands exhibit a wide size distribution with a mean value of (30 ± 5) nm in diameter and 1.2 nm in height.

3.2 ADSORPTION OF SILVER ON SINAPYL ALCOHOL
3.2 ADSORPCIJA SREBRA NA SINAPIL ALKOHOL

Fig. 4 shows MIES (left), UPS He I (middle) and UPS He II (right) spectra of a sinapyl alcohol film before (black lines) and after silver adsorption (red lines). The valence band states of the sinapyl alcohol contribute in the same binding energy ranges as coniferyl alcohol (see sect. 3.1), as discussed by Klarhöfer (2009). The work function decreases from 4.2 eV to 4.1 eV upon silver adsorption, while the background noise decreases, too. The sinapyl alcohol valence band features are significantly broadened after silver adsorption, especially in the MIES

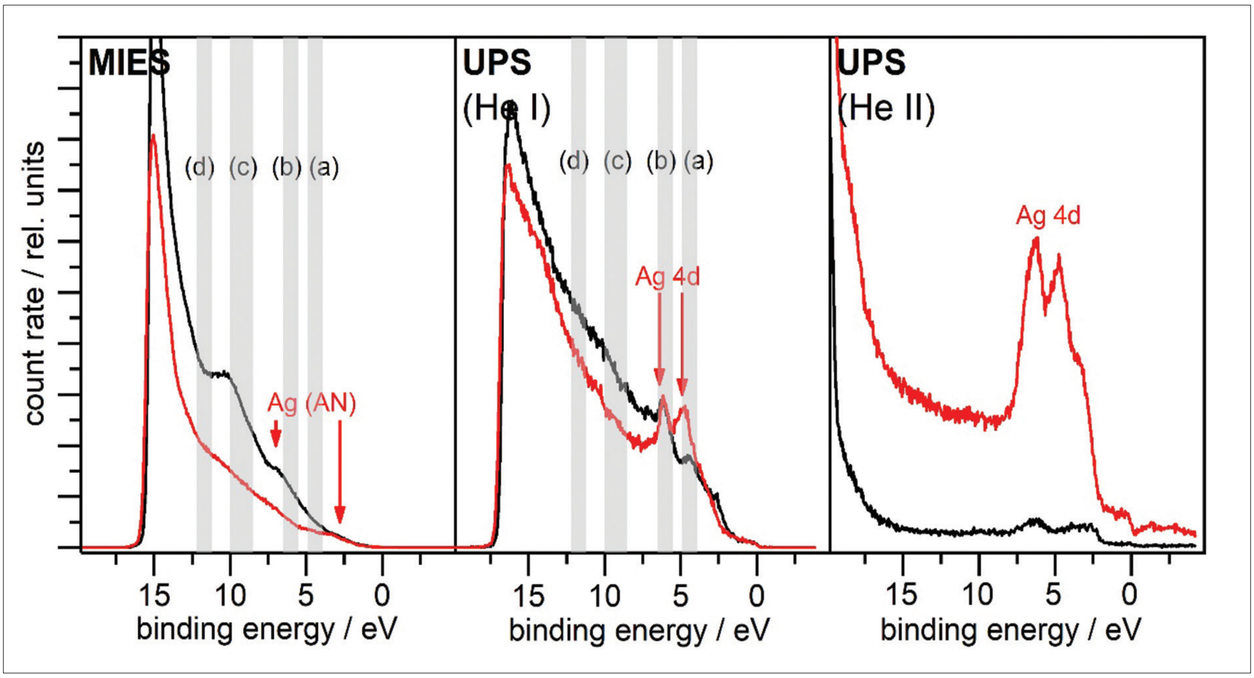

Figure 4. MIES (left), UPS He I (middle) and UPS He II (right) spectra of a sinapyl alcohol film before (black lines) and after silver adsorption (red lines).

Slika 4. MIES (levo), UPS He I (sredina) in UPS He II (desno) spektri filma sinapilnega alkohola pred (črne črte) in po adsorpciji srebra (rdeče črte).

spectrum. No silver AN contributions are visible in the MIES spectrum (reference positions marked by red arrows), whereas UPS reveals the presence of peaks corresponding Ag 4d states.

Fig. 5 contains XPS spectra of the C 1s region (left), the O 1s region (middle) and the Ag 3d region (right) of a sinapyl alcohol film before (black lines) and after silver adsorption (red lines). The C 1s spectrum is similar to the spectra in the literature (Klarhöfer, 2009) and gets just attenuated after silver adsorption without any change in shape. The O 1s structure consists of two peaks, the main feature at 535.5 eV and a shoulder at 533.5 eV. The feature at a higher binding energy is erased after the silver adsorption, whereas the shoulder gets attenuated similar to the C 1s structure. The Ag 3d peak yields no signs of any other chemical state than the pure metal. The calculated film thicknesses amount to 0.4 nm of sinapyl alcohol as well as to an additional silver adlayer thickness of 0.6 nm. Similar to coniferyl alcohol, this is most likely due to an absorption of the metal into the organic film. The C/O ratio changed from 3.1 before silver adsorption to 5.2 afterwards, thus again indicating a restructuring

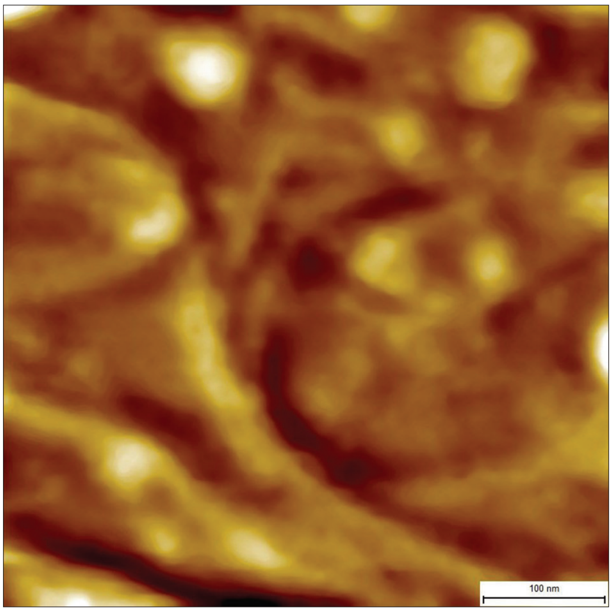

Figure 6. AFM image (500 × 500 nm²) of a sinapyl alcohol film on Au(111) after silver adsorption and air contact.

Slika 6. AFM slika (500 × 500 nm²) filma sinapilnega alkohola na Au (111) po adsorpciji srebra in stiku z zrakom.

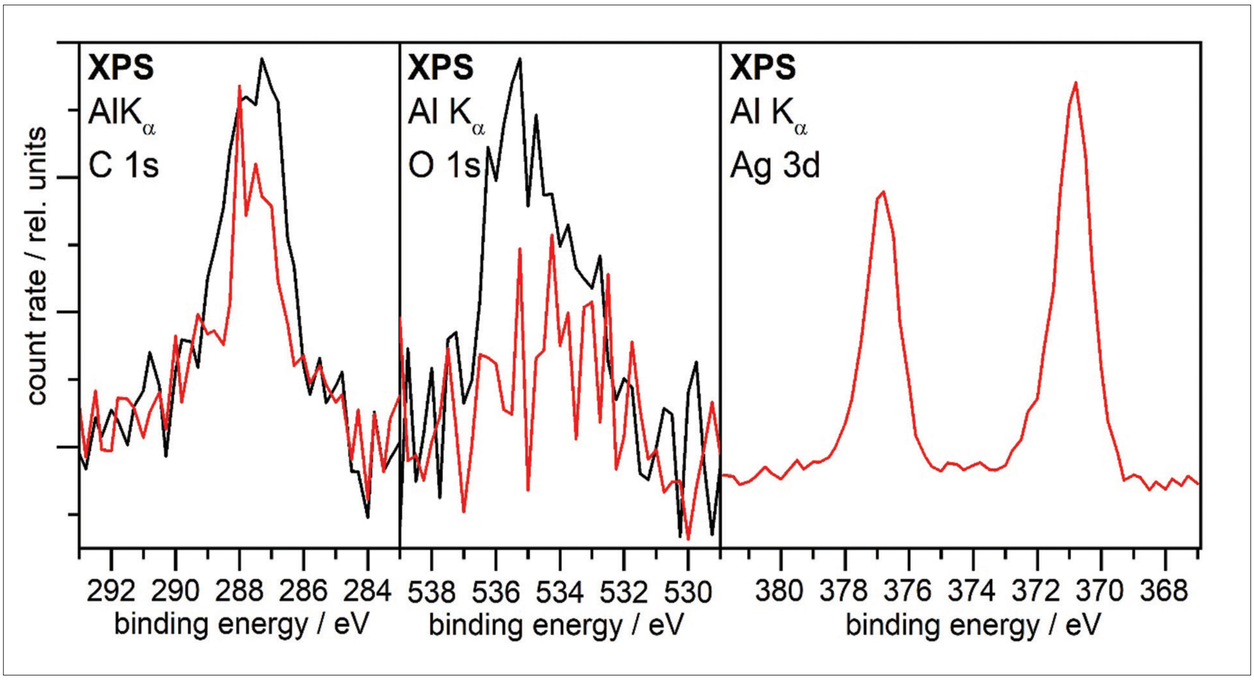

Figure 5. XPS spectra of the C 1s region (left), the O 1s region (middle) and the Ag 3d region (right) of a sinapyl alcohol film before (black lines) and after silver adsorption (red lines).

Slika 5. XPS spektri območja C 1s (levo), območja O 1s (sredina) in območja Ag 3d (desno) filma sinapilnega alkohola pred (črne črte) in po adsorpciji srebra (rdeče črte).

or rearrangement of the film during the silver absorption. The stoichiometries of the samples, C/O ratios and calculated film thicknesses are provided in tab. 2, whereas the complete set of original and analysed data are made openly available online (Dahle et al., 2021).

Fig. 6 depicts the AFM results concerning a sinapyl alcohol film after silver adsorption and air exposure. The smooth surface (R_q = 0.16 nm) exhibits only a few randomly distributed bumps with diameters ranging from 40 nm to 70 nm and an average height of approximately 0.5 nm.

3.3 ADSORPTION OF SILVER ON LIGNIN

3.3 ADSORPCIJA SREBRA NA LIGNIN

Fig. 7 shows the MIES (left left), UPS He I (left middle) and UPS He II (left right) spectra of a pristine lignin film (black lines), after adsorption of 1.8 nm silver (red lines) and after adsorption of 9 nm silver (green lines). A shoulder at the high binding energy side for the lignin spectra, which is due to electrical charging through the lignin film, prevents the analysis of the work functions. The spectra of the pristine lignin film resemble the spectra from the literature quite well (Klarhöfer et al., 2008; Haensel et al., 2012), even though peak broadening conditioned by charging complicates the distinction of the different states. The adsorption of 1.8 nm silver leads to a reduction of the charging induced broadening as well as the shoulder at the high binding energy side of the secondary electron peak. Furthermore, the background noise of inelastic scattered electrons gets reduced significantly after the adsorption of silver. Both UPS spectra, He I and He II, already exhibit a considerable conduction band. Nevertheless, no silver contributions can clearly be distinguished in the He I spectrum, while the He II spectrum contains a large Ag 4d structure. Further adsorption of silver up to a total amount of 9 nm leads to an increase in intensity of the Ag 4d structure in the He II spectrum, whereas the lignin contributions are diminished. After the adsorption of such an amount of silver, the UPS He I spectrum also clearly exhibits the same silver state. The MIES spectrum shows a further reduction of the charging effects, but also exhibits some intensity due to the silver AN process.

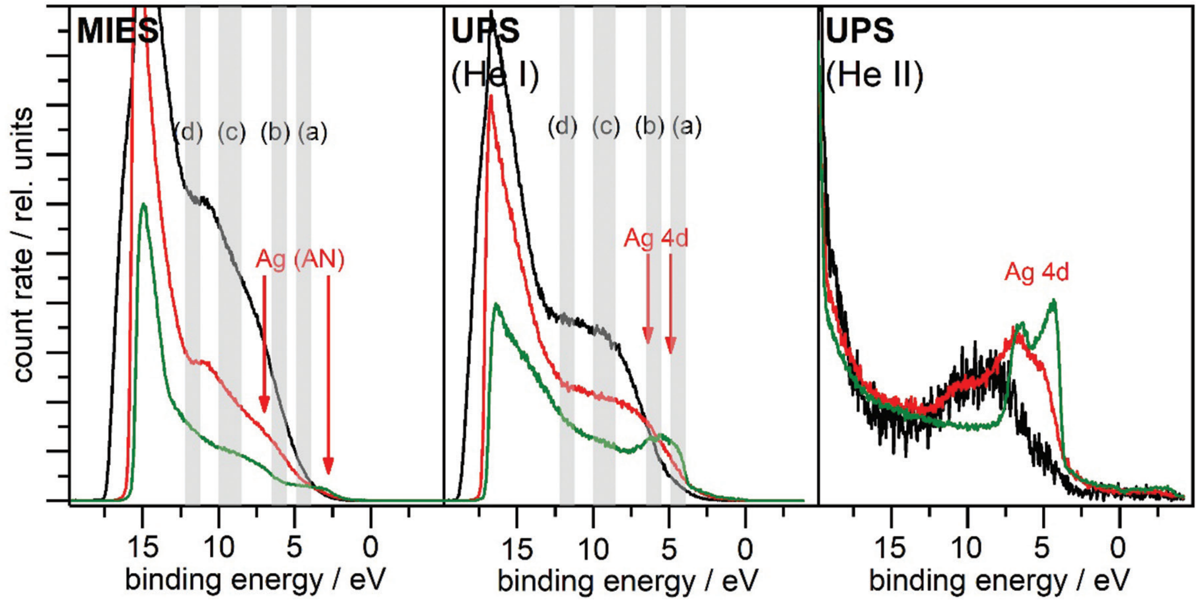

Figure 7. MIES (left left), UPS He I (left middle) and UPS He II (left right) spectra of a pristine lignin film (black lines), after adsorption of 1.8 nm silver (red lines) and after adsorption of 9 nm silver (green lines).
Slika 7. MIES (levo levo), UPS He I (levo sredino) in UPS He II (levo desno) spektri neokrnjenega ligninskega filma (črne črte), po adsorpciji 1,8 nm srebra (rdeče črte) in po adsorpciji 9 nm srebra (zelene črte).

Fig. 8 exhibits XPS spectra of the C 1s region (left), the O 1s region (middle) and the Ag 3d region (right) of a pristine lignin film (black lines), after adsorption of 1.8 nm silver (red lines) and after adsorption of 9 nm silver (green lines). The O 1s structure reveals two peaks as previously found for sinapyl alcohol. Without any change in the shape, the intensity of the O 1s structure is attenuated according to the amount of adsorbed silver. Correspondingly, the intensity of the Ag 3d structure increases while resembling the peak shapes of pure metallic silver (Dahle et al., 2012). The C 1s structure decreases similarly to the O 1s structure upon silver adsorption. The peak shape after the adsorption of 9 nm silver matches the shape of the pristine lignin. There against, the peak shape after adsorption of 1.8 nm silver yields an asymmetrically larger contribution at the high binding energy side. This might indicate a momentary adsorption of carbon oxide within the lignin film. The C/O ratio changed from 3.4 before silver adsorption to 3.7 after adsorption of small amounts of silver, and further to 5.2 after further silver adsorption. The film thickness of the spin-coated lignin film cannot be

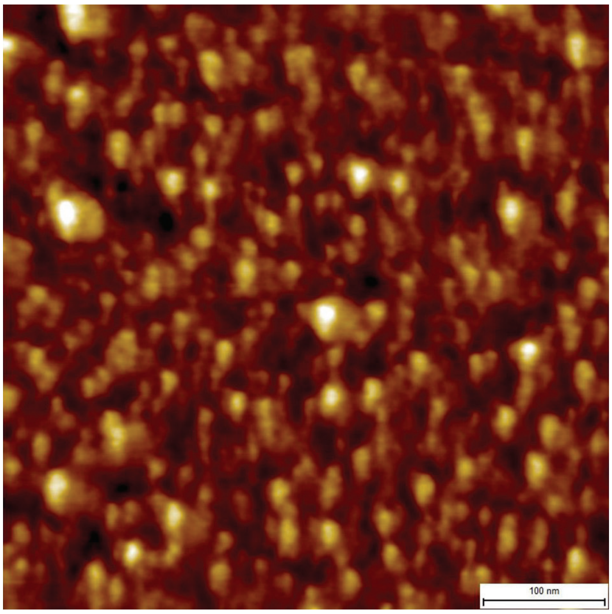

Figure 9. AFM image (500 × 500 nm^2) of a lignin film on Au/Mica after adsorption of 9 nm silver and subsequent air contact.

Slika 9. AFM slika (500 × 500 nm^2) ligninskega filma na Au/sljuda po adsorpciji 9 nm srebra in nadaljnjem stiku z zrakom.

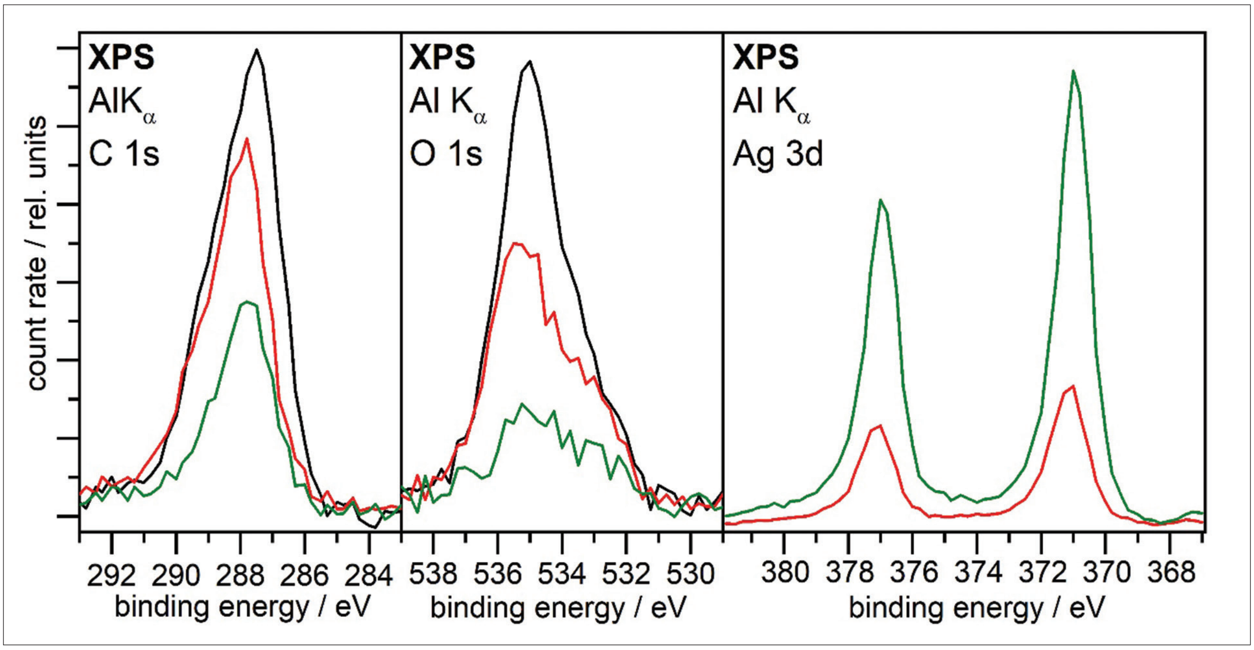

Figure 8. XPS spectra of the C 1s region (left), the O 1s region (middle) and the Ag 3d region (right) of a pristine lignin film (black lines), after adsorption of 1.8 nm silver (red lines) and after adsorption of 9 nm silver (green lines).

Slika 8. XPS spektri območja C 1s (levo), območja O 1s (sredina) in območja Ag 3d (desno) neokrnjenega ligninskega filma (črne črte), *po adsorpciji 1,8 nm srebra (rdeče* črte) *in po adsorpciji 9 nm srebro (zelene* črte).

evaluated from peak attenuation. For the silver adsorption, film thicknesses over the lignin of 0.7 nm and 0.9 nm are calculated. This is consistent with the absorption of the metal into the organic film, similar to the results above for coniferyl and sinapyl alcohol. The stoichiometries of the samples, C/O ratios and calculated film thicknesses are provided in tab. 2, whereas the complete set of original and analysed data are made openly available online (Dahle et al., 2021).

Fig. 9 shows an AFM image of a spin-coated lignin film on Au/Mica. The surface has been covered with the equivalent of 9 nm silver by evaporation and exposed to ambient air, resulting in an RMS-roughness of 0.52 nm. A dense layer of particles covers the surface. Their diameters vary from 11 nm to 45 nm ($d_{average}$= 23.4 nm), the average height is 1.3 nm.

4 CONCLUSIONS

4 SKLEPI

All MIES and UPS valence band spectra reveal a work function of 4.1 eV after silver adsorption for all organic films. The values in the literature for the work function of silver vary between 4.1 eV (Chelvayohan & Mee, 1982) and 4.7 eV (Dweydari & Mee, 1975). The very low value found for the silver-coated organic systems may hence be due to the rough surface structure (Wan et al., 2012), due to a weak surface atom density of the silver with the work function following the Smoluchowski correlation (Dweydari & Mee, 1975), or due to an Ag(110)-oriented crystal face at the top of the nanoparticles (Chelvayohan & Mee, 1982). The valence band features of the organic molecules did not get modified upon silver adsorption for all three systems. The silver structures just slightly appeared in the MIES spectra directly after the adsorption, while the Ag 4d structure and the conduction band already became clearly visible in the UPS spectra. Thus, the silver seems to occupy adsorption sites closely beneath the surface of the organic film, where they are mostly screened by protruding orbitals of the organic films.

The XPS C 1s and O 1s results resembled the molecule spectra from the literature quite well, and the peak shapes did not change notably upon silver adsorption, while their intensities get attenuated by the adlayer. The Ag 3d feature well resembles the reference and literature results for pure metallic silver without any sign of chemical interactions between the adsorbed silver and the organic molecule films. However, a notable change in C/O ratios for all three organic films despite the unchanged peak shapes indicates a restructuring or rearrangement of the organic films upon silver adsorption. This further supports the interpretation of the valence spectra such that the silver for the most part is adsorbed within the organic films. Furthermore, this is sustained by the calculation of adlayer thicknesses of silver over the organic layer, which is much too low for the amount of deposited material. These findings are well in line with the results for cinnamyl alcohol and support the interpretation of those results published earlier (Dahle et al., 2012).

The AFM images reveal structures of agglomerated silver after the sample got in contact with air. The statistical evaluation yielded average diameters of about 30 nm for coniferyl alcohol, 23 nm for sinapyl alcohol and 28 nm for lignin at an average height of 1.2 nm for coniferyl alcohol, 0.5 nm for sinapyl alcohol and 1.3 nm for lignin. This is comparable to the results for cinnamyl alcohol, where nanoparticles with an average diameter of 12 nm at a height of 1.8 nm were found (Dahle et al., 2012).

The results from more in-depth investigations on the interactions of the silver-coated cinnamyl alcohol films with gas molecules revealed a catalytic decomposition of the organic film upon the first contact with oxidizing gases like water or oxygen (Dahle et al., 2014). Furthermore, these investigations yielded strong evidence for this decomposition reaction to be responsible for or at least proceeding simultaneously with the formation of the nanoparticles. A plasma treatment of the cinnamyl alcohol preliminary to the silver adsorption did not influence the formation of the nanoparticles at all (Dahle et al., 2012), even though it caused the attached propylenol chain to be oxidized or reduced according to the chosen process gas. Therefore, the initial adsorption site of the silver atoms within the organic film must be related to the benzene ring rather than the attached chain. As such, a similar decomposition is suggested to be responsible for the nanoparticle formation upon air contact analogous to the findings for cinnamyl alcohol.

In summary, the adsorption of silver on coniferyl alcohol, sinapyl alcohol and lignin yielded chemically inert silver atoms occupying adsorption sites closely beneath the surface of the organic film. No chemical interaction was measurable, but a structural rearrangement of the organic films upon absorption of silver is apparent from the spectra. After the samples were brought into contact with air, nanoparticles with an average diameter between 20 nm and 30 nm were found on all organic films. Due to all of the similarities, the adsorption process is proposed to proceed in a similar manner as to that on cinnamyl alcohol (Dahle et al., 2012; Dahle et al., 2014).

5 SUMMARY

5 POVZETEK

Vsi spektri valentnega pasu MIES in UPS razkrivajo delovno funkcijo 4,1 eV po adsorpciji srebra za vse organske filme. Vrednosti v literaturi za delovno funkcijo srebra se gibljejo med 4,1 eV (Chelvayohan & Mee, 1982) in 4,7 eV (Dweydari & Mee, 1975). Zelo nizka vrednost, ugotovljena za srebrno prevlečene organske sisteme, je zato lahko posledica grobe površinske strukture (Wan et al., 2012), zaradi šibke površinske gostote atomov srebra z delovno funkcijo po korelaciji Smoluchowskega (Dweydari & Mee, 1975) ali zaradi kristalne površine, usmerjene v Ag (110) na vrhu nanodelcev (Chelvayohan & Mee, 1982). Značilnosti valentnega pasu organskih molekul se pri adsorpciji srebra za vse tri sisteme niso spremenile. Signali struktur srebra so bili na spektrih MIES komaj zaznavni, neposredno po adsorpciji, medtem ko sta struktura Ag 4d in prevodni pas že postala jasno vidna v spektrih UPS. Zdi se, da srebro zaseda mesta adsorpcije blizu pod površino organskega filma, kjer jih večinoma pregledujejo štrleče orbitale organskih filmov.

Rezultati XPS C 1s in O 1s so bili precej podobni molekularnim spektrom iz literature in oblike vrhov se pri adsorpciji srebra niso bistveno spremenile po obliki, medtem ko njihovo intenzivnost oslabi adsorbirana plast. Funkcija Ag 3d je zelo podobna referenčnim in literaturnim rezultatom za čisto kovinsko srebro brez kakršnih koli znakov kemičnih interakcij med adsorbiranim srebrom in filmi organskih molekul. Vendar opazna sprememba razmerja C / O za vse tri organske filme kljub nespremenjeni obliki vrhov kaže na prestrukturiranje ali prerazporeditev organskih filmov po adsorpciji srebra. To nadalje podpira razlago valenčnih spektrov na osnovi večinske adsorbcije v organske filme. Poleg tega to potrjujejo tudi izračuni debeline adsorbirane plasti srebra nad organsko plastjo, ki je bistveno prenizka za količino naloženega materiala. Te ugotovitve se popolnoma ujemajo z rezultati o cinamil alkoholu in podpirajo razlago prej objavljenih rezultatov (Dahle et al., 2012).

Slike AFM razkrivajo strukture aglomeriranega srebra, ko je vzorec prišel v stik z zrakom. Statistična ocena je dala povprečni premer približno 30 nm za koniferilni alkohol, 23 nm za sinapilni alkohol in 28 nm za lignin pri povprečni višini 1,2 nm za koniferilni alkohol, 0,5 nm za sinapilni alkohol in 1,3 nm za lignin. To je primerljivo z rezultati na cinamil alkoholu, kjer so našli nanodelce s povprečnim premerom 12 nm na višini 1,8 nm (Dahle et al., 2012).

Rezultati poglobljenih raziskav interakcij posrebrenih filmov cinamil alkohola z molekulami plinov so pokazali katalitično razgradnjo organskega filma ob prvem stiku z oksidativnimi plini, kot sta voda ali kisik (Dahle et al., 2014). Poleg tega so te preiskave dale trdne dokaze, da je bila ta reakcija razgradnje odgovorna za nastanek nanodelcev ali pa je vsaj potekala hkrati z njo. Obdelava cinamil alkohola s plazmo pred adsorpcijo srebra sploh ni vplivala na tvorbo nanodelcev (Dahle et al., 2012), čeprav je povzročila, da se pritrjena veriga propilenolov oksidira ali zmanjša glede na izbrani procesni plin. Zato mora biti začetno adsorpcijsko mesto atomov srebra v organskem filmu povezano z benzenskim obročem in ne s pritrjeno verigo. Na osnovi tega ugotavljamo, da je podoben razkroj odgovoren tudi za tvorbo nanodelcev ob stiku z zrakom, analogno ugotovitvam o cinamil alkoholu.

Ugotavljamo, da so pri adsorpciji srebra na koniferilnem alkoholu, sinapilnem alkoholu in ligninu nastali kemično inertni atomi srebra, ki so zasedli mesta adsorpcije blizu površine organskega filma. Z meritvami ni bilo mogoče izmeriti nobene kemične interakcije, vendar je iz spektrov razvidna strukturna preureditev organskih filmov po absorpciji srebra. Ko so prišli vzorci v stik z zrakom, smo na vseh organskih filmih zaznali nanodelce s povprečnim premerom med 20 nm in 30 nm. Zaradi vseh podobnosti naj bi postopek adsorpcije potekal na podoben način kot pri cinamil alkoholu (Dahle et al., 2012; Dahle et al., 2014).

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SUPPLEMENTAL INFORMATION AND RAW DATA
DODATNE INFORMACIJE IN NEOBDELANI PODATKI

Supplemental material and all raw data can be accessed openly via the author’s institutional repository at https://repozitorij.uni-lj.si/IzpisGradiva.php?lang=eng&id=124499, and cited as Dahle et al. (2021).

Raziskovalni podatki, obravnavani v članku, ki so na voljo v odprtem dostopu prek avtorjevega institucionalnega repozitorija na spletni strani https://repozitorij.uni-lj.si/IzpisGradiva.php?lang=slv&id=124499, naj bodo citirani Dahle et al. (2021).

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THERMAL CONDUCTIVITY OF DIFFERENT BIO-BASED INSULATION MATERIALS

TOPLOTNA PREVODNOST RAZLIČNIH BIO-IZOLACIJSKIH MATERIALOV

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Abstract / Izvleček

Abstract: *To achieve the zero-waste goal as well as sustainability, the use of the raw materials, especially those from nature, and wood in particular, has to be smart, meaning that the resource has to be used to its full potential. Since wood-based industry is associated with high intensity and the generation of a relatively large amount of residues, those residues should be used for the production of useful products, otherwise they will easily be classified as waste and afterwards used as a source of energy. To present a possible solution for wood residues like wood chips, wood particles and bark, we investigated the possibility of using wood and bark residues as constituents for the production of single layer insulation panel with a target thickness of 40 mm and target density of 0.2 g·cm^{-3}. Thermal conductivity was determined using the steady state principle at three different temperature settings. The average thermal conductivities were determined between 49 mW·m^{-1}·K^{-1} and 74 mW·m^{-1}·K^{-1}. The highest values were determined at boards made from bark, which also had the highest density (0,291 g·cm^{-3}), while the lowest thermal conductivity was observed for boards made from spruce wood particles.*

Keywords: *thermal conductivity, insulation, particleboard, wood particles, bark*

Izvleček: *Doseganje cilja „nič odpadkov" in načela trajnostne rabe je kompleksen proces, ki zahteva učinkovito rabo surovine, še posebej naravne, zlasti lesa. Pomembno je, da je raba surovine celostna, da se uporabi v celoti za izdelavo trajnostnih produktov. Lesnopredelovalna industrija je visoko intenzivna proizvodnja, kar pomeni, da pri tem nastanejo tudi velike količine ostankov, primernih za izdelavo uporabnih proizvodov. V primeru, ko ostankov ne bi smotrno uporabili, pa se to surovino opredeli kot odpadek in uporabi za proizvodnjo energije (kar pa ni najbolj trajnostna in tudi optimalna rešitev). S ciljem predstavitve možne uporabe lesnih ostankov, kot so npr. sekanci, iveri in skorja, smo raziskali možnost njihove uporabe za izdelavo enoslojnih izolacijskih plošč ciljne debeline 40 mm in gostote 0.2 g·cm^{-3}. Toplotno prevodnost smo določili pri treh različnih temperaturnih pogojih. Ugotovljene vrednosti povprečne toplotne prevodnosti so bile med 49 mW·m·1·K^{-1} in 74 mW·m·1·K^{-1}. Najvišje vrednosti so bile izmerjene pri ploščah iz skorjete so imele tudi najvišjo gostoto (0.291 g·cm^{-3}), najnižje pa so izmerili na ploščah iz smrekovih iveri.*

Ključne besede: *toplotna prevodnost, izolativnost, sekanci, iveri, skorja*

1 UVOD

1 INTRODUCTION

One of the important advantages of wood and wood-based industry is its ability to utilize the whole potential of this material created by nature. Trees are perennial plants with roots, stems and branches, and the wood that we get from tree stems is one of the best materials for sustainable development. But when considering sustainable development, the circular economy and zero waste society, there is still much more we can do with wood, as a significant amount of the tree stays unused or its usability is limited,

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and thus is wasted or burned, with the latter causing the emission of greenhouse gasses, and thus pollution.

Particleboard made from wooden particles can be classified as a thermal insulating material. Sonderegger and Niemz (2009) reported a thermal conductivity value for three-layer particleboard between 99 $mW \cdot m^{-1} \cdot K^{-1}$ and 118 $mW \cdot m^{-1} \cdot K^{-1}$ (density between 0.62 and 0.76 $g \cdot cm^{-3}$).

The availability of reports on the usability of wood chips for thermal insulation is limited. Wang and Fukuda (2016) presented the possibility of using vinyl packed wood chips as "loose-fill" insulation material for roofs. Skogsberg and Lundberg (2005) showed the positive impact of wood chips in order to prevent snow melting when snow is used as a cooling agent.

The most unused part of a tree is also the part that is most visible, the bark, which serves as a protective barrier for wood. Bark constitutes between 10% and 20% of total tree mass, although this varies regarding wood species and age of tree. Bark is the outer part of a tree occurring outside of the vascular cambium and can be divided into outer bark (dead tissue) and inner part (tissue with living cells). The majority of harvested bark is currently used for energy, which is, according to Deppe and Hoffmann (1972) and Gupta et al. (2011), not the best solution due its low calorific value and high CO_2 emissions. A smaller amount of bark is used in special applications like horticulture (for landscaping), in pharmacy (Miranda et al., 2012), for leather tanning (Pizzi, 2008), for insulation panels (Kain et al., 2014), foams (Tondi & Pizzi, 2009; Čop et al., 2015), decorative panels for flooring (Tudor et al., 2018), or as a substance to produce fire-resistant wood-based composites for construction purposes (Tondi et al., 2014). Most of the bark obtained in the debarking process in the forest or at a sawmill is unused, however, which creates an industrial and environmental problem. The complex structure of bark presents a problem when considering its usage, but it also opens wide range of possible approaches. In the literature some information can be found about utilizing bark for particleboards, with such work presented by Dost (1971), Deppe and Hoffman (1972), Maloney (1973), Lehmann and Geimer (1974), Place and Maloney (1977), Muszynski and McNatt (1984), Suzuki et al. (1994), Blanchet et al. (2000), Nemli and Colakoglu (2005), and Yemele et al. (2008). Despite the use of different adhesives, they determined that excessive bark content in the particleboard lowers mechanical properties and resistance against water (increases thickness swelling and water uptake). Ružiak et al. (2017) used bark as a filler (a flour substitute) and also determined its influences on thickness swelling. The main reason for such behaviour was due to differences in the size of constituents and the chemical structure of bark compared to wood. The chemical composition of bark shows that it contains a higher content of ash, extractives and lignin, and a lower content of polysaccharides cellulose and hemicelluloses (Antonović et al., 2010; Antonović et al., 2018). Due to the presence of phenol like components in bark, which react with formaldehyde (Cameron & Pizzi, 1985; Prasetya & Roffael, 1991; Nemli & Çolakoğlu, 2005; Takano et al., 2008; Medved et al., 2019), the addition of bark resulted in lower formaldehyde emissions. Bark, although an underutilized bio- or lignocellulose-based resource, is also a very interesting material, which has a versatile role in the tree. As summarized by Rossel et al. (2014), the main functions of bark are protection, transportation and storage of nutrients, insulation, and mechanical support of the stem. Due to undesired particle morphology and the fact that a lot of dirt, stones, and other unwanted contaminants, picked up during logging, can have a negative impact on processing, bark has generally been of low interest for wider industrial use, especially for the production of wood-based panels (Deppe & Hoffman, 1972; Pásztory et al., 2016).

The utilization of bark relies mostly on its physical and chemical properties, hence it is no surprise that the majority of reports in last decade(s) are focused on using bark as insulation material (Martin, 1963; Suzuki et al., 1994; Sato et al., 2009; Kain et al. 2014).

Martin (1963) determined that dry bark has almost 20% better insulation properties than solid wood at the same density, and that the conductivity of boards made from bark was lower than that obtained for boards made from wood.

The usage of low quality wood is nowadays mostly related to particleboard and fibreboard production, and also with energy production, while bark is used mostly for energy. In the present investigation we focus on an important wood property, on its thermal insulation ability. Wood and bark residues created by the wood processing industry are mostly in already crushed shape, meaning they are either in shape of particles or chips. The aim of this paper is thus to present the possibility to using wood and bark residues for the production of boards with low thermal conductivity.

2 MATERIALS AND METHODS
2 MATERIALI IN METODE DELA

To carry out this study Norway spruce (*Picea abies* Karst.) residues and a mixture of coniferous barks were used. Solid wood and bark were processed in a laboratory using a Prodeco M-0 chipper (Figure 1) with an output screen with openings of 25 mm in diameter, along with a Condux CSK 350/N1 ring chipper (Figure 2), with the gap between the blade and beating bar of 1.25 mm.

A ring chipper was used only to obtain wood particles (Figure 3a), while a chipper was used for the primary breakdown of wood (Figure 3b) and bark (Figure 3c).

The resulting constituents were dried at 70°C for 16 hours to achieve a moisture content below 4 %. After drying, an appropriate mass of particles was put into a blending machine, where the particles were blended with melamine-urea-formaldehyde adhesive (blending ratio 15%, solid/solid ratio) produced by Melamin Kočevje, Slovenia. The total blending time was 10 minutes (5 minutes adhesive spraying and mixing + 5 minutes only mixing). Blended particles were then hand formed into

Figure 1. Prodeco M-0 chipper
Slika 1. Prodeco M-0 sekirostroj

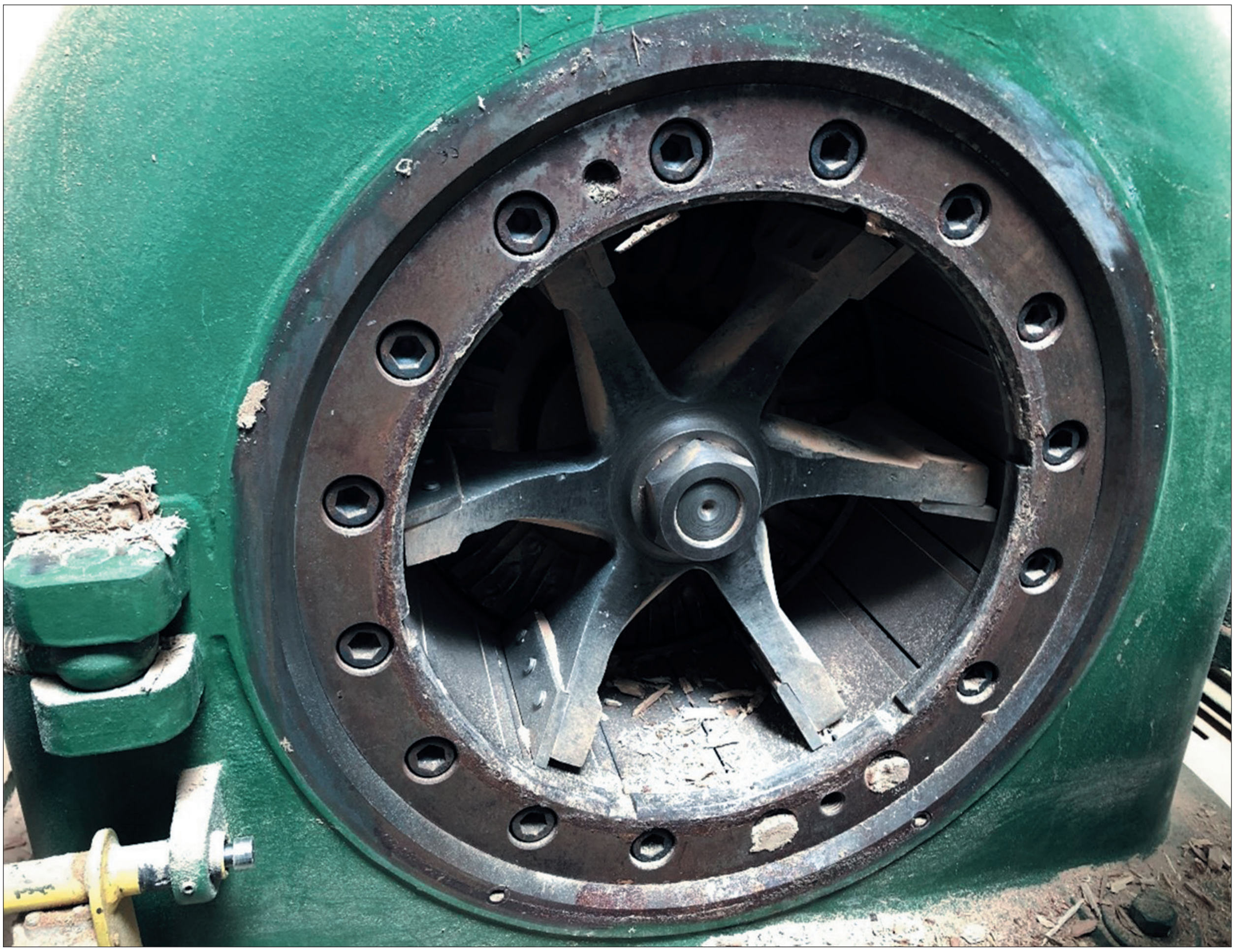

Figure 2. Condux CSK 350/N1 ring chipper

Slika 2. Condux CSK 350/N1 obročasti iverilnik

Figure 3. Constituents used in the investigation (spruce wood particles (3a), spruce wood chips (3b) and coniferous bark (3c))

Slika 3. Gradniki, uporabljeni v raziskavi (iveri lesa smreke (3a), sekanci lesa smreke (3b) in skorja iglavcev (3c))

a frame with the dimensions 500×500 mm^2 (Figure 4), which was placed on a steel plate.

The target density of the produced panels was 0.2 $g{\cdot}cm^{-3}$, with a thickness of 40 mm. The mat was pressed at 180°C and a pressure of 2 $N{\cdot}mm^{-2}$. The pressing time was set to 9 minutes followed by a 1 minute degassing phase. Three boards per composition were made.

The boards were then left at room conditions to cool down (for 60 minutes), and later placed in

Figure 4. Wood and bark mat before pressing
Slika 4. Natreseni gradniki lesa in skorje pred stiskanjem

a climate chamber at a temperature of 23±1°C and 55±5% relative air humidity. After 21 days the thermal conductivity of the boards was measured.

Thermal conductivity was determined according to EN 12667, 2002 in an LM.305 heat flow meter (Stirolab, Sežana, Slovenia). The technique used is based on the measuring of the heat conductivity in the heating chamber until steady state conditions are achieved. The measuring area was 290×260 mm^2. The measurements were conducted at three different conditions, as shown in Table 1.

The determination of heat conductivity lasted 240 minutes. Thermal conductivity λ in $W \cdot m^{-1} \cdot K^{-1}$ was calculated according to equation 1.

$$\lambda = \frac{t \times Q}{S \times \Delta T} \qquad [1]$$

where t is the thickness of sample in m, Q is the generated heat flow in W (for upholding steady state conditions – linear temperature gradient), S is the sample surface area in m^2 and ΔT is the temperature difference between hot and cold sides in K.

3 RESULTS AND DISCUSSION
3 REZULTATI IN RAZPRAVA

The thickness and density of the produced boards depends on the board composition (Table 2).

Even though all boards had same target density and thickness it can be observed that there were differences between the boards. The differences in thickness and density between boards made from wood chips and wood particles are due the differences in the compressibility of the constituents (bigger constituents, i.e. chips, being less compressible, and hence a higher thickness and lower density). The higher density and lower thickness of bark board could be the result of the reaction during compression, and thus it can be assumed that bark constituents were damaged (crushed and broken) during pressing, which enabled repositioning of the crushed parts into voids, hence creating a higher density. This higher density bark board also resulted in higher thermal conductivity, and so lower resistance against heat conductivity (Figure 5).

Comparing the density values (Table 2) and thermal conductivity (Figure 5) reveals a correlation, and namely that an increase in density causes

Table 1. Thermal conductivity measurements settings
Preglednica 1. Merilna območja za določanje toplotne prevodnosti

	Lower plate / Spodnja plošča	**Upper plate / Zgornja plošča**	**Average temperature / Povprečna temperatura**	**Temperature difference / Temperaturna razlika**
	°C	**°C**	**°C**	**K**
Set 1 / Območje 1	10	20	15	10
Set 2 / Območje 2	10	35	22.5	25
Set 3 / Območje 3	10	50	30	40

Table 2. Thickness and density of the produced boards
Preglednica 2. Debeline in gostote izdelanih plošč

	Thickness / Debelina	Density / Gostota
	mm	g·cm^{-3}
Wood chips / Lesni sekanci	42.42	0.232
Wood particles / Lesne iveri	42.01	0.252
Bark / Skorja	37.06	0.291

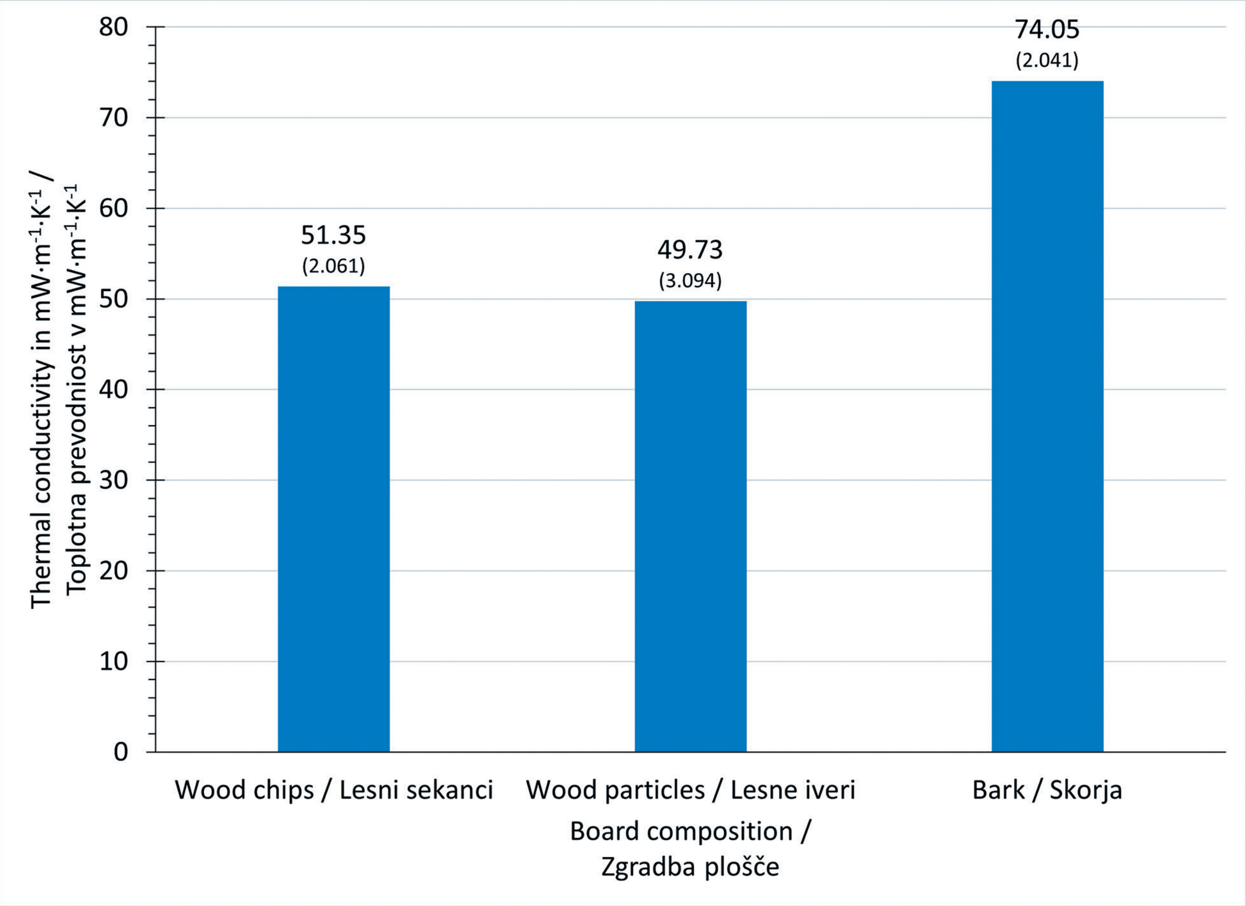

Figure 5. Average thermal conductivity with regard to the board composition (numbers in parentheses represent standard deviation values)
Slika 5. Povprečne vrednosti toplotne prevodnosti glede na zgradbo plošče (vrednosti v oklepajih so vrednosti standardnih odklonov)

an increase in thermal conductivity. The increase in thermal conductivity of denser materials is related to the higher number of constituents in contact with each other and lower number of "air" filled voids, which present a barrier to heat conductivity, while constituents in contact conduct heat from hot towards cold areas. Heat transfer will occur when the area in question reaches the maximum potential (with regard to neighbouring conditions), and the area next to it is cooler and able to accept the energy. When all areas are heated to maximum potential (according to available conditions) permanent heat flow occurs, and that flow also means a loss in energy. Higher thermal conductivity was also observed in the board made of wood chips (compared to wood particles),

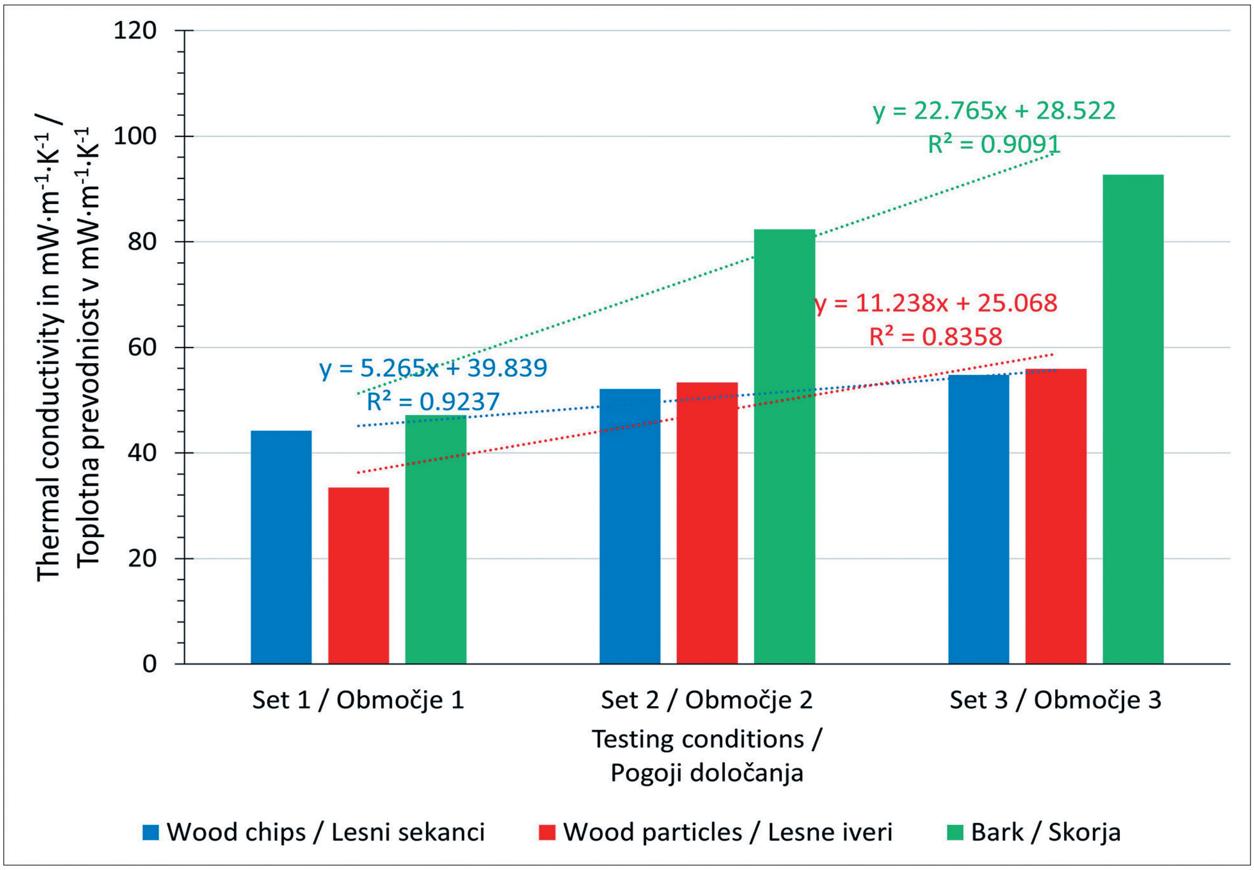

Figure 6. Thermal conductivity with regard to board composition and testing conditions
Slika 6. Toplotna prevodnost glede na zgradbo plošče in pogoje določanja toplotne prevodnosti

which could be related to the constituents' morphology. Although "stacking" bigger constituents leaves larger voids, those larger voids do not help much in lowering thermal conductivity. This is because bigger constituents enable the creation of bigger contact areas between constituents, which results in more efficient heat transfer.

A detailed analysis of the boards' behaviour at different settings shows more clearly the difference, especially between boards from wood-based constituents (Figure 6).

In all boards, the increase in average temperature/temperature differences resulted in an increase in thermal conductivity, but when comparing the relations related to board composition it reveals that boards made from wood chips are less influenced by the change in temperature conditions, while bark boards show higher sensitivity towards an increase in temperature.

Since heat resistance is determined as the relation between thickness and thermal conductivity, there is no surprise that the lowest resistance was recorded in boards containing bark (Table 3), supporting the earlier observations that bigger constituents and higher density offer lower heat flow resistance.

Table 3. Heat resistance of the produced boards (the numbers in parentheses represent standard deviation values)
Preglednica 3. Toplotna upornost izdelanih plošč (v oklepajih so vrednosti standardnih odklonov)

	Heat resistance / Toplotna upornost
	$m^2·K·W^{-1}$
Wood chips / Lesni sekanci	0.84 (0.047)
Wood particles / Lesne iveri	0.88 (0.084)
Bark / Skorja	0.55 (0.033)

4 CONCLUSIONS
4 SKLEPI

An analysis of the different residues (chips, particles, bark) shows that it is possible to make a stable board with densities between 0.23 g·cm^{-3} and 0.29 g·cm^{-3} for insulation purposes. The highest thermal conductivity values and the lowest heat resistance were determined in bark boards. The highest insulation effect was found when wood (spruce) particles were used, but the sensitivity towards the testing conditions was lowest in boards made from wood (spruce) chips.

5 SUMMARY
5 POVZETEK

Pomembna prednost lesnopredelovalne industrije je sposobnost izkoriščanja celostnega potenciala lesa kot dragocene naravne surovine. Celostna raba naravnih virov je pomemben vidik krožnega gospodarstva, nizkoogljične družbe in družbe z „nič odpadki“. Čeprav je področje rabe lesa zelo široko, od različnih proizvodov iz masivnega lesa do različnih lesnih ploščnih kompozitov, pa vseeno opažamo, da relativno velika količina lesa ostane neuporabljenega oz. njegov celotni potencial ni izkoriščen. Številne raziskave pričajo o možnostih rabe lesnih ploščnih kompozitov ne samo za izdelavo pohištva ali nosilnih konstrukcijskih elementov ampak tudi za izdelavo izolacijskih materialov (Sonderegger & Niemz, 2009; Wang & Fukuda, 2016; Skogsberg & Lundberg, 2005), pri čemer so bile plošče oz. izolatorji izdelani iz iveri ali pa iz sekancev. Skupina avtorjev pa je za izdelavo izolatorjev uporabila tudi skorjo (Martin, 1963; Suzuki et al., 1994; Sato et al., 2009; Kain et al., 2014).

Uporaba lesne surovine slabše kakovosti je danes omejena predvsem na izdelavo ivernih in vlaknenih plošč oz. na proizvodnjo energije, medtem ko se skorja uporablja predvsem kot energent. Ker se tako les kakor tudi skorja nahaja že v zdrobljeni obliki (sekanci, iveri), je cilj pričujoče raziskave predstaviti možnost rabe takšne surovine za izdelavo izolacijskih plošč.

Za izdelavo izolacijskih plošč smo uporabili lesne ostanke navadne smreke (*Picea abies* Karst.) in skorjo iglavcev, ki smo jo predelali v sekance oz. iveri na laboratorijskem sekirostroju Prodeco M-0 in laboratorijskem obročastem iverilniku Condux CSK 350/N1. Izdelane gradnike smo nato pri temperaturi 70 °C posušili do vlažnosti pod 4 %, čemur je sledilo oblepljanje z melamin-urea-formaldehidnim lepilom (delež lepila 15 %). Oblepljene gradnike smo nato ročno natresli v lesen okvir z dimenzijami 500 × 500 mm^2. Ciljna debelina plošč je bila 40 mm, ciljna gostota pa 0.2 g·cm^{-3}. Stiskali smo 9 minut pri temperaturi 180 °C in tlaku 2 N·mm^{-2}. Po 21dnevni klimatizaciji pri normalnih pogojih (temperaturi 23±1 °C in 55±5 % relativni zračni vlažnosti) smo določili toplotno prevodnost plošč po standardu EN 12667, 2002. Toplotno prevodnost smo določili pri treh različnih pogojih in sicer:

- ΔT10: temperatura spodnje plošče 10 °C, temperatura zgornje plošče 20 °C
- ΔT25: temperatura spodnje plošče 10 °C, temperatura zgornje plošče 35 °C
- ΔT40: temperatura spodnje plošče 10 °C, temperatura zgornje plošče 50 °C

Meritev je trajala 240 minut.

Debelina izdelanih plošč je bila 37,06 mm (skorja), 42,01 mm (iveri) oz. 42,42 mm (sekanci), gostota pa 0.232 g·cm^{-3} (sekanci), 0.252 g·cm^{-3} (iveri) oz. 0.291 g·cm^{-3} (skorja) (preglednica 2). Tudi toplotna prevodnost je odvisna od zgradbe plošče oz. velikosti uporabljenih gradnikov. Pri plošči, izdelani iz smrekovih sekancev, je bila povprečna toplotna prevodnost 51.35 mW·m^{-1}·K^{-1} (44.23 mW·m^{-1}·K^{-1} pri ΔT10; 55.12 mW·m^{-1}·K^{-1} pri ΔT25 in 54.76 mW·m^{-1}·K^{-1} pri ΔT40), iz smrekovih iveri 49.73 mW·m^{-1}·K^{-1} (33.43 mW·m^{-1}·K^{-1} pri ΔT10; 53.30 mW·m^{-1}·K^{-1} pri ΔT25 in 55.91 mW·m^{-1}·K^{-1} pri ΔT40) in pri skorji 74.05 mW·m^{-1}·K^{-1} (47.13 mW·m^{-1}·K^{-1} pri ΔT10; 82.37 mW·m^{-1}·K^{-1} pri ΔT25 in 92.66 mW·m^{-1}·K^{-1} pri ΔT40). Ugotovili smo, da je toplotna prevodnost odvisna od velikosti uporabljenih gradnikov (manjša pri manjših gradnikih) ter gostote (večja pri ploščah z večjo gostoto). Raziskava je pokazala, da je mogoče iz različnih ostankov (lesnih sekancev, lesnih iveri in skorje) izdelati stabilne plošče z gostoto med 0.23 g·cm^{-3} in 0.29 g·cm^{-3}, ki se lahko uporabijo kot izolacijske plošče, saj je toplotna prevodnost nižja od 75 mW·m^{-1}·K^{-1}.

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ZAHVALE

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Oddelek za lesarstvo sodeluje kot partner v ciljnem raziskovalnem projektu za izboljšanje konkurenčnosti lesa listavcev

Tina Drolc

Ciljni raziskovalni projekt (CRP) z naslovom »Možnosti rabe lesa listavcev v slovenskem biogospodarstvu«(LesGoBio) v okviru vodilne inštitucije, Gozdarskega Inštituta Slovenije vodi dr. Peter Prislan. Partner v projektu je tudi Biotehniška fakulteta Univerze v Ljubljani. Koordinator projekta izr. prof. dr. Aleš Straže s Katedre za tehnologijo lesa na Oddelku za lesarstvo je povedal: »Projekt naslavlja aktualna vprašanja glede možnosti širjenja in izboljšanja rabe lesa listavcev, razvoja novih tehnologij ter tržnih in makroekonomskih posledic. Posebej izpostavlja pomen opredelitve obetavnih, okoljsko sprejemljivih in ekonomsko izvedljivih postopkov obdelave in predelave in presojo sodobnih inovativnih lesnopredelovalnih tehnologij. Pomemben cilj projekta je razviti scenarije in strateška priporočila za implementacijo sodobnih tehnologij za proizvodnjo izdelkov z visoko dodano vrednostjo, ki bi omogočale racionalno in trajnostno rabo lesa listavcev.«

V projektu se bodo raziskave osredotočale na najbolj zastopane domače lesove listavcev kot so bukovina, hrastovina in javorovina ter ostale bolj zastopane, kot so topolovina, gabrovina in brezovina.

Širši cilj projekta je spodbuditi proizvodnjo izdelkov z višjo dodano vrednostjo, kar navadno z narodnogospodarskega vidika pomeni v prihodnje pozitivno prestrukturiranje lesnopredelovalne industrije, s tem pa vzporedno spremembo strukture porabljenih virov za proizvodnjo.

Projekt v vrednosti 170.000 EUR financirata Ministrstvo za kmetijstvo, gozdarstvo in prehrano Republike Slovenije (MKGP) in Javna agencija za raziskovalno dejavnost Republike Slovenije (ARRS), izvajal pa se bo 3 leta, od novembra 2020 do 2023.

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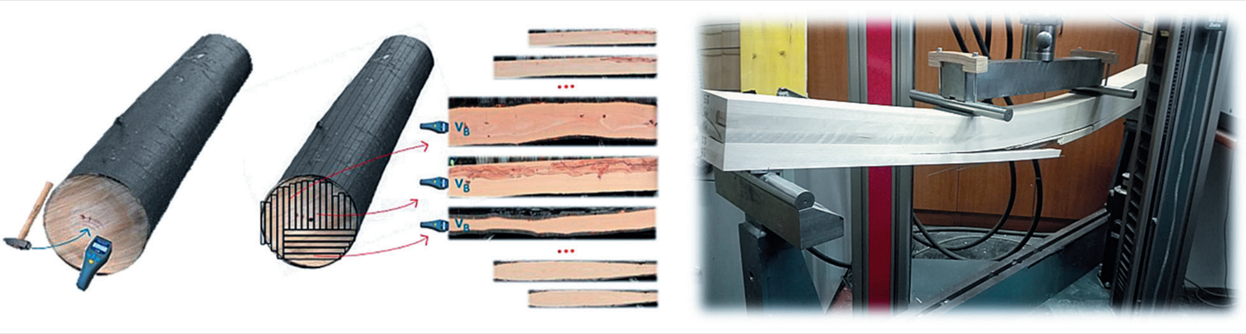

In memoriam: prof. dr. Jože Kovač (1930–2021)

prof. dr. Jože Resnik, zaslužni profesor v pokoju
Biotehniška fakulteta UL, Oddelek za lesarstvo

Konec januarja 2021 se je v enaindevetdesetem letu poslovil prof. dr. Jože Kovač, priznan strokovnjak tako v lesni industriji kot v univerzitetnem izobraževanju na področju lesarstva. Prof. dr. Jože Kovač je bil rojen v Ljubljani 18. marca 1930. Leta 1949 je z odliko končal gimnazijo v Ljubljani. Študij na gozdarskem oddelku takratne Fakultete za agronomijo, gozdarstvo in veterino Univerze v Ljubljani je zaključil leta 1956. Za diplomsko delo z naslovom Upoštevanje naravne dediščine pri gospodarjenju z gozdovi v krajinskem parku Zgornja Idrijca je prejel fakultetno Prešernovo nagrado.

Po koncu študija se je kot pripravnik zaposlil na Okrajni upravi za gozdarstvo OLO Ljubljana, leta 1958 pa kot asistent na Agronomsko-gozdarski fakulteti na gozdarskem oddelku, kjer je ostal do leta 1961. Nato je kot upravitelj gozdnega obrata Kamniška Bistrica delal v Silva-gozdnem in lesnem gospodarstvu Agronomske in gozdarske fakultete v Ljubljani, od leta 1963 pa kot referent za napredek proizvodnje v Gozdnem gospodarstvu Ljubljana. Pomembna sprememba se je zgodila v letu 1968, ko je postal generalni direktor v Lesnem podjetju Hoja v Ljubljani. V času njegovega vodenja je podjetje Hoja vidno napredovalo. Organizacijsko je združeval lesne obrate in jih selil v industrijsko cono Vič, kar je poleg drugih prednosti omogočalo bolj optimalen razvoj podjetja in bolj ekonomično poslovanje, ki se je kazalo tudi v višanju plač zaposlenih v primerjavi s plačami v slovenskem gospodarstvu. Zaposlitev v podjetju Hoja je prekinil leta 1974 in se isto leto zaposlil na Biotehniški fakulteti kot predavatelj na gozdarsko-lesarskem oddelku Univerze v Ljubljani, kjer je ostal do upokojitve leta 1994.

Leta 1976 je na Gozdarski fakulteti Univerze v Zagrebu uspešno zagovarjal doktorsko delo z naslovom Proučevanje zastojev v avtomatiziranem delovnem procesu proizvodnje lesno-cementnih gradbenih plošč in istega leta postal izredni, leta 1982 pa redni profesor za organizacijo proizvodnih procesov v lesarstvu. Njegove organizacijske sposobnosti potrjuje tudi funkcija prvega predstojnika novoustanovljenega VTOZD-a za lesarstvo (1975–1979). Veliko truda je vlagal v nadgradnjo raziskovalnega in pedagoškega delovnega področja ter optimalne in učinkovite organizacije proizvodnje v lesni industriji. V tem okviru je izdal več študijskih gradiv za področje, ki se je pričelo z njegovim prevzemom strokovno in znanstveno oblikovati. Uspešnost njegovega pedagoškega dela na področju lesne proizvodnje v praksi potrjujejo tudi številna diplomska dela s tega področja. Kot zunanji sodelavec-svetovalec je neposredno sodeloval tudi pri oblikovanju organizacijskih razvojnih politik v pomembnih lesnih tovarnah tistega časa, kot so bile Hoja Ljubljana, Bor Laško, Stilles Sevnica, Meblo Nova Gorica, Novoles Novo mesto in Savinja Celje. Bogate izkušnje iz dela v gozdarstvu in lesni industriji je znal prenesti v pedagoški proces in delo s študenti in študentkami, v izbiro tem diplomskih del in raziskovalnih tematik. Seveda smo iz tako celovitega znanja in dodanih praktičnih izkušenj veliko pridobili tudi njegovi sodelavci in sodelavke.

V zgodovino razvoja Oddelka za lesarstvo Biotehniške fakultete UL se je zagotovo zapisal kot izkušen poznavalec organizacije in pogojev za uspešen razvoj neke dejavnosti, kar zelo nazorno potrjuje njegovo prizadevanje za ustanovitev samostojnega Oddelka za lesarstvo na Biotehniški fakulteti UL leta

1975, vodenje študijskega predmeta organizacija poslovanja v lesarstvu, bogata in obširna bibliografija ter mentorstva dodiplomskim in podiplomskim študentom in študentkam magistrskega in doktorskega študija na tem področju. Bil je mentor pri dvainsedemdesetih diplomskih delih, dveh magistrskih delih in dveh doktorskih disertacijah.

Bil je avtor in soavtor številnih znanstvenih, strokovnih in poljudnih člankov ter prispevkov na domačih in mednarodnih strokovnih konferencah, posvetih in stanovskih srečanjih. Med njegovimi uredniškimi deli velja izpostaviti Spominski zbornik Biotehniške fakultete Univerze v Ljubljani 1988–1992.

Prof. dr. Jože Kovač je pustil neizbrisen pečat tudi kot predsednik Odbora za izgradnjo 1. faze Oddelka za lesarstvo v letih od 1974 do 1984. Desetletno delo, o katerem je bila izdana posebna publikacija, je bilo zelo zahtevno, saj se je ves čas soočal s številnimi formalnimi, organizacijskimi in izvedbenimi težavami.

Posebno priznanje mu je bilo izkazano tudi s strani Biotehniške fakultete in Univerze v Ljubljani, ko mu je bilo v letih 1992–1994 zaupano vodenje Habilitacijske komisije Univerze v Ljubljani.

Prof. dr. Jože Kovač je prejel decembra 1993 Svečano listino in zlato plaketo Univerze v Ljubljani za področje vzgojno-izobraževalnega in znanstvenoraziskovalnega dela. Leta 1994 je prejel Jesenkovo priznanje Biotehniške fakultete UL za pomembne zasluge, kot je bilo prizadevanje za ustanovitev samostojnega Oddelka za lesarstvo leta 1975, vodenje študijskega predmeta organizacija poslovanja v lesarstvu, bogata bibliografija, mentorstvo bodočim magistrom in doktorjem znanosti ter predsedovanje odboru za izgradnjo novih pedagoških in raziskovalnih prostorov na Oddelku za lesarstvo BF UL. Uspešno je bilo tudi njegovo vodenje Biotehniške fakultete, kjer je bil dekan v letih od 1989 do 1992, v čas katerega sodijo tudi zaključne potrditve o selitvi Dekanata in dela prostorov za pedagoški proces s Krekovega trga v centru Ljubljane na Jamnikarjevo 101.

Omeniti je potrebno tudi njegovo vseskozi aktivno in prizadevno sodelovanje pri delovanju posameznih delov strokovnih društev in revij. Tako je bil v letih od 1975 do 1977 predsednik Zveze društev inženirjev in tehnikov gozdarstva in lesarstva Slovenije, leta 1978 je bil imenovan za njenega zaslužnega, leta 1991 pa za častnega člana. Od leta 1982 je petnajst let vodil uredniško politiko uveljavljene strokovne in znanstvene revije LES, pri čemer je bil prvih deset let tudi njen glavni in odgovorni urednik. Pod njegovim vodstvom je revija pomembno izboljšala svoje mesto med tovrstnimi mednarodnimi strokovnimi in znanstvenimi revijami.

Prof. dr. Jože Kovač je bil velik ljubitelj in poznavalec narave, posebno gozdnih območij Slovenije. Morda je tudi zato postal vnet pohodnik, ki je z veseljem predajal svoje znanje in krepil spoštovanje do narave. To so bile vedno posebne učne ure o naravi, različnih rastlinah in gozdu v njej, za kar smo mu vsi, ki smo ga spremljali na teh poteh, globoko hvaležni.

In memoriam: Alojz Leb – starosta slovenskega lesarstva!

Mirko Geršak

Alojz (uradno Alojzij) Leb se je rodil leta 1929 na Kapli na Kozjaku. Učil se je za mizarja in tesarja. Kot tesar je delal pri graditvi Litostroja. Pot ga je vodila mimo šole na Aškerčevi, vstopil je iz radovednosti in se nato vpisal na novoustanovljeni Lesnoindustrijski odsek Tehniške šole za kemijsko, metalurško, rudarsko, lesno in papirno stroko v Ljubljani (šolsko leto 1949/50).

Izobraževanje je nadaljeval na Ekonomski fakulteti, kjer je leta 1962 diplomiral.

Naredil je tudi vse izpite za magistrski študij. Magistriral ni zato, ker je mentor vztrajal, da v njegovi diplomski nalogi ugotovitve niso v skladu s Kardeljevo socialistično doktrino. Alojz Lep pa jih ni hotel spremeniti.

V svoji 41-letni karieri je opravljal različna dela. Bil je mizar, tesar, sušilničar (Kopitarna Sevnica), laborant, vodja oddelka za pospeševanje proizvodnje (Stol Kamnik), tehnolog, tehnični vodja in v.d. direktorja (Melodija Mengeš). Bil je direktor Biroja za lesno industrijo, direktor organizacijsko-kadrovskega sektorja v ljubljanski Lesnini, svetnik generalnega direktorja Slovenijalesa in sekretar Splošnega združenja lesarstva Slovenije. Privlačilo ga je tudi pedagoško delo, zato je za hobi poučeval tudi na poklicnih in vajeniških šolah na Duplici, Mengšu in Domžalah. Nekaj časa je honorarno učil tudi na Srednji tehniški šoli v Ljubljani in na višji šoli Lesarskega oddelka Biotehniške fakultete. Kasneje je na Zavodu za šolstvo RS nadzoroval delovanje poklicnih lesarskih šol, zastopal Gospodarsko zbornico Slovenije v Svetu za šolstvo RS in sodeloval pri reformah v šolskem sistemu, še posebej, ko se je uvajalo t.i. usmerjeno izobraževanje. Opravljal je delo tajnika Posebne izobraževalne skupnosti lesarstva v Sloveniji. Kot sekretar Splošnega združenja lesarstva Slovenije ima precejšnje zasluge za gradnjo objekta Oddelka za lesarstvo Biotehniške fakultete Univerze v Ljubljani.

Bil je izredno plodovit pisec, saj je napisal številne strokovne članke s področja lesarstva.

Zapisal:
M. Geršak s pomočjo Zbornika ob 120-letnici Srednje lesarske šole v Ljubljani

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PROF. DR. KATARINA ČUFAR JE PREJELA JESENKOVO NAGRADO ZA ŽIVLJENJSKO DELO

PROF. DR. KATARINA ČUFAR RECEIVED THE JESENKO LIFETIME ACHIEVEMENT AWARD

Marko Petrič[1*], Milan Šernek[1]

Izvleček / Abstract

Izvleček: *Prof. dr. Katarina Čufar je v marcu 2021 prejela Jesenkovo nagrado za življenjsko delo, ki je najprestižnejše priznanje Biotehniške fakultete Univerze v Ljubljani. To je tretja nagrada za Katarino Čufar v kratkem času, saj je konec leta 2020 prejela tudi Zlato plaketo Univerze v Ljubljani in Zoisovo priznanje Republike Slovenije za pomembne znanstvenoraziskovalne dosežke in uspešno pedagoško delo.*

Ključne besede: *Jesenkova nagrada, življenjsko delo, Biotehniška fakulteta, Univerza v Ljubljani*

Abstract: *In March 2021, Prof. Dr. Katarina Čufar received the Jesenko Lifetime Achievement Award, the most prestigious prize of the Biotechnical Faculty, University of Ljubljana. This is the third award for Katarina Čufar within just a few months, as at the end of 2020 she also received the Golden Plaque from the University of Ljubljana and the Zois Prize of the Republic of Slovenia for significant scientific achievements and exemplary teaching.*

Keywords: *Jesenko Award, lifetime achievement, Biotechnical Faculty, University of Ljubljana*

Slika 1. Jesenkovo nagrado za življenjsko delo je nagrajenki prof. dr. Katarini Čufar podelila prva dekanja Biotehniške fakultete prof. dr. Nataša Poklar Ulrih.
Figure 1. Presentation of the Jesenko Lifetime Achievement Award to Prof. Dr. Katarina Čufar by the first female Dean of the Biotechnical Faculty, Prof. Dr. Nataša Poklar Ulrih.

Prof. dr. Katarina Čufar je v marcu 2021 prejela Jesenkovo nagrado za življenjsko delo, ki je najprestižnejša nagrada Biotehniške fakultete Univerze v Ljubljani (Slika 1, 2). To je že tretja nagrada za prof. dr. Katarino Čufar v kratkem času, saj je v letu 2020 prejela tudi Zlato plaketo Univerze v Ljubljani za zgledno znanstvenoraziskovalno in pedagoško delo in Zoisovo priznanje Republike Slovenije za pomembne znanstvenoraziskovalne dosežke (Vrhunci …, 2020; Slaček, 2021). Dosežke, na osnovi katerih

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je nagrajenka prejela Zlato plaketo in Zoisovo priznanje, sta v tej reviji predstavila prof. dr. Milan Šernek in prof. dr. Marko Petrič (Šernek & Petrič, 2020), tokrat pa na kratko povzemamo še nekaj poudarkov, ki dopolnjujejo sliko nagrajenkinega življenjskega dela.

Prof. dr. Katarina Čufar je redna profesorica na Biotehniški fakulteti Univerze v Ljubljani. Kot Katarina Pleško je bila rojena v Ljubljani, kjer je obiskovala tudi osnovno šolo in gimnazijo Šentvid. Na Oddelku za lesarstvo Biotehniške fakultete je zaključila univerzitetni, magistrski in doktorski študij, vse pod mentorstvom prof. dr. dr. h. c. Nika Torellija. Po diplomi se je leta 1981 zaposlila na Biotehniški fakulteti, Oddelku za lesarstvo kot stažistka-raziskovalka ter delovala kot asistentka in nato visokošolska učiteljica, od leta 2008 redna profesorica (Velušček, 2021).

Prof. dr. Katarina Čufar se kot vrhunska znanstvenica na področju lesarstva posveča znanosti o lesu, dendrokronologiji ter vsestranskim raziskavam lesa kot tkiva živih dreves in materiala v objektih kulturne dediščine in arheologije. Na Biotehniški fakulteti je vzpostavila novo znanstveno področje dendrokronologije, s podpodročji dendroarheologije, dendroekologije in dendroklimatologije. V Katedri za tehnologijo lesa, ki jo vodi, skrbi za razvoj laboratorijev, uvajanje novih metod in njihovo uporabo za raziskovalno in strokovno delo. S sodelavci razvija vsestranske raziskave lesa od njegovega nastanka v drevesu in gozdu preko njegove predelave v industriji in uporabe kot naravnega obnovljivega materiala. Sistematično dolgoletno raziskovalno delo je posvetila tudi lesu v kulturni dediščini in arheologiji, pri čemer so najbolj odmevne in sistematične raziskave prazgodovinskih kolišč na Ljubljanskem barju. Prof. Čufarjeva se tudi na tem področju povezuje s številnimi skupinami na Biotehniški fakulteti in na drugih institucijah (Slika 3, 4). Tako je vzpostavila in razvila sodelovanje z Inštitutom za arheologijo ZRC SAZU, enotami in centri Zavoda za varstvo kulturne dediščine Slovenije ter z muzeji nacionalnega in lokalnega pomena.

Nagrajenkino bogato znanstvenoraziskovalno delo se kaže v številnih objavah v vrhunskih mednarodnih revijah in na konferencah. Je avtorica preko 175 originalnih znanstvenih člankov v najprestižnejših znanstvenih revijah, med katerimi so npr. tudi revije Nature Communications, Science Advances, Nature Plants in številne druge z visokimi faktorji vpliva (COBISS …, 2021; Orcid …, 2021; RUL …, 2021). Bila je v organizacijskih odborih več kot 10 konferenc (posebej Eurodendro in World Dendro) ter na mednarodnih srečanjih sodelovala s številnimi vabljenimi plenarnimi predavanji. Prav tako je članica uredniških odborov več mednarodnih revij. Trenutno deluje predvsem kot zunanja urednica revije Tree-Ring Research in je glavna urednica revije Les / Wood, ki je pomembna za razvoj stroke v Sloveniji in slovenske strokovne terminologije.

Prof. dr. Katarina Čufar je izrazito vpeta v mednarodno raziskovalno in akademsko pedagoško okolje. Od leta 2016 je izvoljena članica IAWS (International Academy of Wood Science), kamor izvolijo vodilne znanstvenike s področja lesarstva in kjer je bila izvoljena kot prva članica iz Slovenije. Pedagoško je redno gostovala na tujih univerzah, večinoma v okviru programa SOCRATES / ERASMUS. Katedra, kjer deluje, redno gosti uveljavljene raziskovalce in doktorske in dodiplomske študente različnih smeri z vsega sveta. Bila je mentorica več študentom iz tujine in je koordinirala izmenjave študentov z Biotehniške fakultete.

Prof. dr. Katarina Čufar je izjemna pedagoginja, ki je v bogati pedagoški karieri poučevala predmete na področju anatomije in zgradbe ter bioloških lastnosti lesa. Med študenti je izjemno priljubljena, zato ne preseneča dejstvo, da je bila mentorica ali somentorica skoraj 100 diplomantkam in diplomantom, 4 doktorandkam in 1 doktorandu. Šest njenih diplomantk in diplomantov je prejelo fakultetno Prešernovo nagrado. Bila je članica komisije za zagovor pri več kot 15 doktoratih na univerzah Zagreb, Hamburg, Praga, Dunaj, Brno, Innsbruck, Padova, Neapelj, Montpellier, Alicante in Zaragoza. Trenutno je mentorica podoktorskemu kandidatu in mladi raziskovalki na doktorskem študiju. Zaradi svojega zglednega pedagoškega dela je prof. Čufar dvakratna prejemnica pohvale Biotehniške fakultete za najboljšo pedagoško delavko na Oddelku za lesarstvo. Snovala in oblikovala je različne študijske programe in aktivno sodelovala pri bolonjski prenovi študijskih programov lesarstva. Več kot deset let je oddelčna predsednica komisije za dodiplomski študij.

Prof. dr. Katarina Čufar je od leta 1999 vodja raziskovalne skupine Tehnologija lesa in Katedre za tehnologijo lesa. Bila je prodekanja za področje lesarstva, trenutno pa je že četrti mandat namestnica prodekana za pedagoško dejavnost. Več mandatov je bila in še vedno je članica Senata Biotehniške fakultete. Je članica Komisije za dodiplomski študij Biotehniške fakultete, kjer je bila 2 mandata predsednica.

Vrhunsko raziskovalno in pedagoško delo prof. dr. Katarine Čufar je že bilo prepoznano tako na Univerzi v Ljubljani kot tudi izven nje. Je dobitnica priznanja Biotehniške fakultete ob 50-letnici delovanja fakultete (1997), priznanja Biotehniške fakultete za zgledno pedagoško in raziskovalno delo (2013) ter priznanja Društva lesarjev Slovenije za prispevek pri povezovanju članov in uspešno vodenje ALUMNI kluba (2017), ter častnega priznanja Fakultete za gozdarstvo in tehnologijo lesa Univerze Mendel v Brnu, Republika Češka ob 100 letnici ustanovitve fakultete v letu 2019 (Drolc, 2019). V zadnjem obdobju še posebej odmeva, da je v letu 2020 prejela kar dve prestižni nagradi: Zoisovo priznanje in Zlati znak Univerze v Ljubljani.

Nagrajenka je izjemno izobražena, razgledana in modra profesorica, ki s svojim delom, vzgledom in vrednotami pozitivno vpliva tako na študente in študentke kakor tudi na sodelavke in sodelavce ter jih s svojo energijo in raziskovalno navdušenostjo spodbuja ter motivira pri delu. Poklic profesorice in znanstvenice na univerzi jemlje kot posebno poslanstvo, kar je ključno pri uspešnem izobraževanju in vzgoji študentk in študentov ter pri razvoju znanosti in raziskovalne odličnosti. Hkrati je njeno delovanje velik navdih za vse nas, ki imamo privilegij, da s profesorico tako ali drugače sodelujemo na svojih poteh.

Na osnovi navedenega ji je na predlog kolegic in kolegov z Oddelka za lesarstvo in po izboru komisije prva dekanja Biotehniške fakultete, prof. dr. Nataša Poklar Ulrih (pred tem so fakulteto vodili le dekani), v marcu 2021 podelila Jesenkovo nagrado za življenjsko delo. Za slovesnost podelitve je bil o nagrajenki posnet tudi kratek film, kjer je na kratko povzela in predstavila svoje raziskovalno delo in se

Slika 2. Zahvalni nagovor ob podelitvi.
Figure 2. Acknowledgement speech at the award ceremony.

zahvalila vsem za sodelovanje in podporo (Katarina Čufar - Jesenkova ..., 2021), predstavljena pa je bila tudi v Raziskovalnih novicah Univerze v Ljubljani (Z raziskovanjem lesa ..., 2021).

Nagrajenka se je ob podelitvi zahvalila sodelavkam in sodelavcem, ki so njene dosežke prepoznali, jo predlagali in izbrali, predvsem pa vsem, ki so z njo sodelovali in še sodelujejo. V prepričanju, da priznanje za življenjsko delo temelji na rezultatih skupnega dela, se je za sodelovanje in vsestransko podporo posebej zahvalila sodelavkam in sodelavcem ter kolegicam in kolegom doma in v tujini, vsem ki so jo učili in se od nje učili, ter ožji in širši družini za nenehno spodbudo in podporo. Poudarila je, da je hvaležna za uspehe in izkušnje ter da si želi, da bi lahko svoje znanje, povezave in dobre prakse še naprej izmenjevala z vsemi, ki jih veselijo les, znanje o lesu, znanost in pomembne življenjske teme. Vsi, ki smo imeli in še imamo čast sodelovati s prof. dr. Katarino Čufar, niti najmanj ne dvomimo v to, da bomo pri njej vedno dobrodošli, ko bomo potrebovali znanstveni ali strokovni nasvet s področja njenega delovanja, pa tudi kak bolj splošen nasvet ali mnenje, ki temelji na njenih bogatih znanstveno-raziskovalnih izkušnjah in življenjski modrosti.

Prof. Dr. Katarina Čufar received the Jesenko Lifetime Achievement Award

Prof. Dr. Katarina Čufar is the recipient of the Jesenko Lifetime Achievement Award 2021, the most prestigious award of the Biotechnical Faculty at the University of Ljubljana. This is the third award she has received in just a few months, as at the end of 2020 she also received the Golden Plaque of the University of Ljubljana and the Zois Prize of the Republic of Slovenia for significant scientific achievements as a researcher and teacher (Figure 1, 2). Her research was recently presented in this journal (Šernek & Petrič, 2020), but here we briefly summarise some of the highlights that complete the picture of the laureate‘s life work.

The laureate was born Katarina Pleško in Ljubljana, where she also graduated at elementary and secondary school and completed her studies at the Biotechnical Faculty, Department of Wood Science and Technology at the Bachelor‘s, Master‘s and PhD levels with theses supervised by Prof. Niko Torelli, Dr. Dr. h. c. In 1981 she was employed as a young researcher at the Biotechnical Faculty and worked as a teaching assistant, then as a university lecturer, becoming a full professor in 2008.

Prof. Dr. Katarina Čufar, is recognised as the top scientist in the field of wood research. She is dedicated to wood science, dendrochronology and general research of wood as a tissue of living trees and widely used material in numerous products, including objects of cultural heritage and archaeology. At the Biotechnical Faculty she introduced the new scientific field of dendrochronology with its subfields of dendroarchaeology, dendroecology and dendroclimatology. At the Chair of Wood Technology, which she heads, she helped to set up the laboratories, introduced new methods and their application for research and applied work. With her team, she researches wood from its origin in the tree, through its selection and processing in industry, to its use as a natural, renewable material. She has also devoted many years to the systematic study of wood in cultural heritage and archaeology, especially and systematically the prehistoric pile dwellings in the Ljubljansko barje, which existed more than 4,500 years ago. Prof. Čufar cooperates actively with research teams at the Biotechnical Faculty and other institutions in Slovenia and worldwide (Figure 3, 4).

The rich scientific and research work of the awardee is reflected in numerous publications in top scientific journals, where she has (co-)authored over 175 original scientific articles, among them *Nature Communications*, *Science Advances*, *Nature Plants* and other journals with high impact factors (COBISS..., 2021; Orcid..., 2021; RUL..., 2021). She has presented her work at numerous conferences and was a member of the scientific and organisational committees of more than 10 conferences, especially Eurodendro and World Dendro. She has served on the editorial boards of several journals. Currently she serves in the editorial board of *Tree-Ring Research* and as the editor-in-chief of *Les/Wood*, which is important for the development of wood science and of Slovene professional terminology.

Prof. Dr. Katarina Čufar is heavily involved in the international research and academic teaching environment. She has regularly been a guest teacher and researcher at foreign universities, mainly in the frame of the SOCRATES / ERASMUS programme. The research group she leads regularly hosts international guests, including renowned researchers and graduate and undergraduate students from around the world. She has been a mentor for numerous outgoing and incoming students in the framework of mobility programmes, and coordinated studies at foreign universities for students from the Biotechnical Faculty.

Slika 3. Življenjsko delo temelji na sodelovanju in medsebojni podpori. Na slikah Prof. Dr. Katarina Čufar s sodelavkami in sodelavci z Oddelka za lesarstvo (a, d), Gozdarskega inštituta Slovenije (b), Inštituta za arheologijo ZRC SAZU (c) in Univerze Zaragoza (b, d).

Figure 3. A life time of successful work is based on the good cooperation and mutual support of colleagues on national and international levels. In the pictures Prof. Dr. Katarina Čufar with researchers from the Department of Wood Science and Technology (a, d), Slovenian Forestry Institute (b), Institute of Archaeology of the ZRC SAZU (c), and University Zaragoza (b, d).

Prof. Dr. Katarina Čufar is an excellent teacher who has taught courses on wood anatomy, structure and biology during her teaching career. She is extremely popular among students. She has been (co-)supervisor of nearly 100 graduation theses and five doctoral dissertations. She has been a member of the defence committees of about 15 PhD theses at universities in Zagreb, Hamburg, Prague, Vienna, Brno, Innsbruck, Padova, Napoli, Montpellier, Alicante and Zaragoza. She is currently mentoring a postdoctoral researcher and a young researcher-doctoral student. Due to her exemplary work, Prof. Čufar has twice received the award for the best teacher of the year in the Department of Wood Science, Biotechnical Faculty. She has designed and developed various study programmes. For more than ten years she has been the department chairperson of the committee for Student Affairs.

Since 1999, Prof. Dr. Katarina Čufar has been the head of the Chair of Wood Science. She has been Vice-Dean for Wood Science and Technology and is currently in her fourth term as Deputy Vice-Dean for Teaching Affairs. She has been a member of the Senate of Biotechnical Faculty for several

Slika 4. Nagrajenka s študenti Oddelka za lesarstvo (a, b) in z mednarodnimi udeleženkami in udeleženci srečanja Historical Wood Utilization v Sloveniji.

Figure 4. With colleagues and students of the Department of Wood Science and Technology (a, b) and with international participants of the meeting Historical Wood Utilisation in Slovenia.

terms. She is the member of the Undergraduate Studies committee of the Biotechnical Faculty, where she has been chair for two terms.

Prof. Dr. Katarina Čufar's outstanding research and teaching activities have already been recognised both inside and outside the University of Ljubljana. Furthermore, she has been an elected member of IAWS (International Academy of Wood Science) since 2016 and a member of the IAWS Board since 2018. She received the Biotechnical Faculty Award on the occasion of the 50th anniversary of the Faculty (1997), the Biotechnical Faculty Award for exemplary teaching and research (2013), the Slovenian Woodworkers‘ Association‘s Award for her contributions to member networking and successful leadership of the ALUMNI Club (2017), and the Honorary Award of the Faculty of Forestry and Wood Technology of Mendel University of Brno, Czech Republic, on the occasion of the 100th anniversary of the Faculty‘s foundation (2019). As mentioned above, she received two prestigious awards in 2020: the Zois Prize and the Golden Plaque of the University of Ljubljana.

Prof. Dr. Katarina Čufar is an exceptionally knowledgeable, insightful and wise professor who positively influences both students and colleagues through her work, example and values, encouraging and motivating them in their work with her energy and enthusiasm for research. She sees the profession of professor and scientist at the University as a special mission that is crucial for the successful education of students and for the development of teaching and research excellence. At the same time, her work is a great inspiration to all of us who have had, and continue to have, the privilege of working with her in one way or another on our own journeys.

Prof. Dr. Katarina Čufar received the Jesenko Lifetime Achievement Award in March 2021 from the first female Dean of the Biotechnical Faculty, Prof. Dr. Nataša Poklar Ulrih. The short documentation (in Slovenian) briefly summarised her life‘s work (Katarina Čufar - Jesenkova..., 2021).

At the award ceremony, she thanked all those who had recognised her achievements, nominated her and awarded her. She gave special thanks to all her colleagues, teachers, students and her family, as she believes that the Lifetime Achievement Award is based on the results of cooperation and mutual support.

All of us who have the privilege of working with Prof. Dr. Katarina Čufar believe that we will continue to meet with her, whether for professional or general discussions or opinions, or just for fun.

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