

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

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

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Abstract / Izvleček

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

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

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

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

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1 INTRODUCTION

1 UVOD

Frequent availability, versatility, aesthetics and many other favourable characteristics made wood the most important biomass derived material for a varied range of end uses. The diverse nature of several wood species has resulted in their specific uses, which in the present day is difficult to maintain due to the depleting supplies of timber resources worldwide. Wood modification is the most suitable solution to this, where using various methods secondary and non-durable wood can be made suitable for specific uses and its sustainability thus ensured for longer periods. Among these techniques, thermal and chemical modifications of timber have become relatively common (Hansmann et al., 2005; Despot et al., 2008; Esteves & Pereira, 2009; Sefc et al., 2009; Hom et al., 2020; Treu et al., 2020) across the globe due to their ease of processing and satisfactory commercial and laboratory performance. However, extensive energy consumption and the utilisation of non-environmentally-friendly chemicals are the two most common drawbacks associated with these methods. In the last few decades treating and modifying wood with microwaves (MW) has become more popular (Kol & Çayır, 2021) in the niche research domain of wood modification with a variety of use cases (Oloyede & Groombridge, 2000; Torgovnikov & Vonden, 2010; Balboni et al., 2018; Samani et al., 2019; Weng et al., 2021). For timber drying, for example, it has been shown to have a lower level of energy consumption (Brodie, 2010; Sethy et al., 2016) in comparison to the conventional methods, with an increased drying rate of timber and subsequently reduced the drying time (Awoyemi, 2004; Gamben et al., 2005). Several studies demonstrated strongly increased liquid permeability and preservative uptake in refractory and moderately refractory timber species after several levels of MW modification and pre-treatment (Liu et al., 2005; Treu & Gjolsjo, 2008; Torgovnikov & Vinden, 2009; Ramezanpour et al., 2015; Xu et al. 2015; Hermoso & Vega, 2016; Samani et al., 2019; Ganguly et al., 2021a). This was shown to originate primarily in the rupture of weak anatomical elements leading to the generation of micropores and microcracks (Mekhtiev & Torgovnikov, 2004; Terziev et al., 2020; Weng et al., 2020; Ganguly et al., 2021b) as well as an increment in

pore diameters along with destruction of pit membranes and damage to cell walls. However, this change in the wood microstructure upon exposure to higher intensities of MW often compromises the strength of timber (He et al., 2014), although there have been cases where the reduction is not statistically significant (Kol & Çayır, 2021). During the MW treatment of wooden materials, the build-up of internal gas pressure from vapourized water and volatile organic compounds needs to be considered, as it can increase permeability through microcracks in the cell walls (Hong-Hai et al., 2005) or negatively impact the workpiece's mechanical properties through material cracking (Ouertani et al., 2015) and other issues related to inhomogeneous rises in temperature (Bartoli et al., 2019). As such, the optimization of the treatment parameters is of utmost importance when using MW for wood modification.

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

mies, 2014; Hämmäläinen & Kärki, 2013). Therefore, this study focuses on gliding arc jet PT.

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

2 MATERIALS AND METHODS

2 MATERIALI IN METODEDE

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

The treatment of wood with microwaves (MW) was carried out using a MW oven (Model: M020MW, Gorenje, Velenje, Slovenia) with a frequency of 2.45 GHz and a maximum output of 700 W at a power consumption of 1500 W. One energy level of 1260 MJ/m³ was applied to the wood samples for all the treatments involving MW in the study. The duration of MW irradiation was 36 s. The parameters of MW treatments were defined based on MW power and volume of the samples as proposed by (Kol & Çayir, 2021) and our preliminary

experiments. After MW treatment, the samples were cooled in a desiccator at room temperature and subjected to further analysis.

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

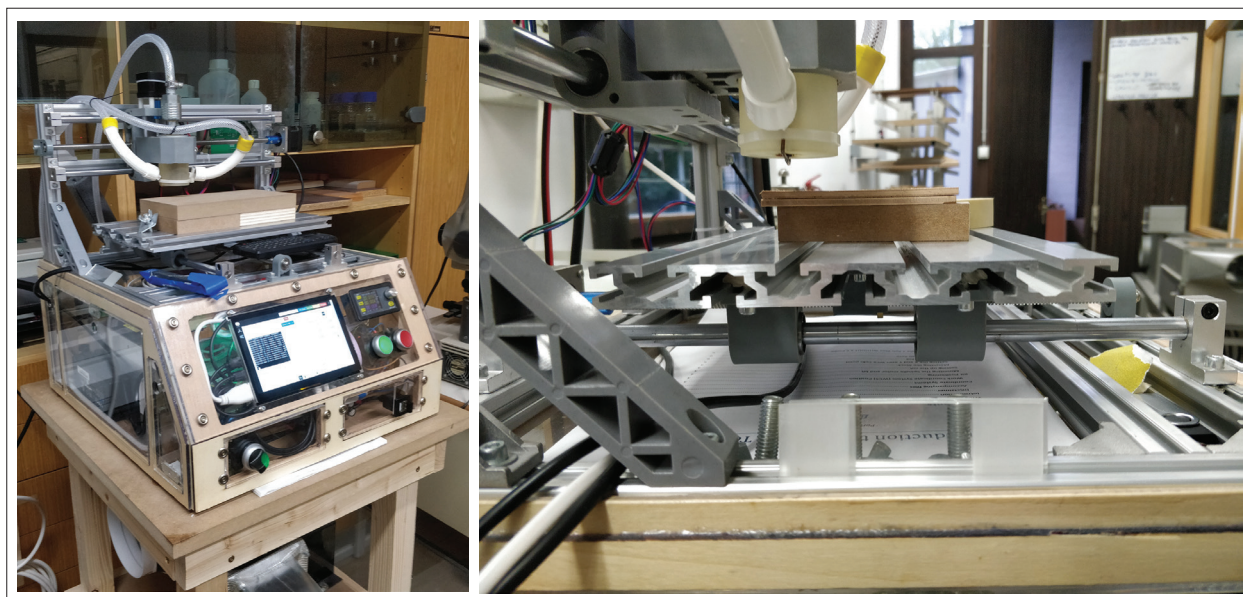


Figure 1. Gliding arc jet device on a CNC positioning stage.

Slika 1. Naprava z drsnim obločnim plazemskim curkom in računalniško krmiljeno pozicionirno mizico.

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

$$y = A_1 \cdot \exp(-x/t_1) + y_0 \quad (1)$$

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

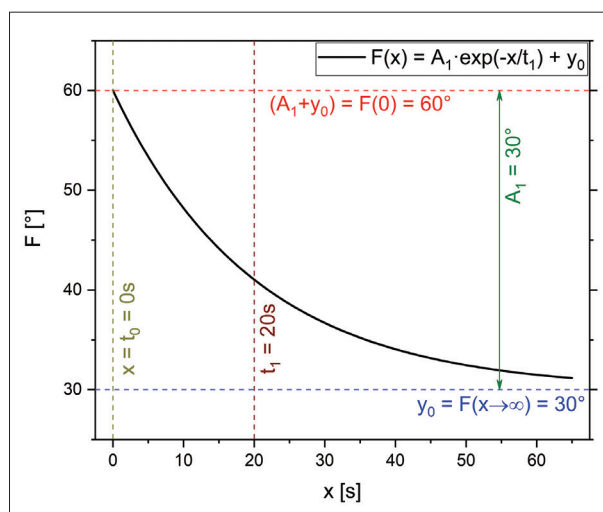


Figure 2. Example curve with exponential decay according to equation 1.

Slika 2. Primer eksponentno pojemajoče krivulje po enačbi 1.

droplet's behaviour and this is referred to as the final WCA. Parameter A_1 , shown as vertical green arrow line, represents the difference between the contact angle at $t_0 = 0s$ and the value for $t \rightarrow \infty$. The intuitive value discussed in the manuscript, however, is the WCA directly upon the first contact of the droplet with the specimen's surface ($t_0 = 0s$), which is given by $A_1 + \gamma_0$, shown as dashed red horizontal line, and this is referred to as the initial WCA. The third parameter t_1 , shown as a dashed brown vertical line, represents the time, after which the value of the function has fallen from $F(0s) = (A_1 + \gamma_0)$ down to $F(t_1) = (A_1/e + \gamma_0)$, i.e. by approx. 63.2% of A_1 . The smaller parameter t_1 , the faster the droplet spreads across the surface, i.e. the higher the wetting rate. Parameter t_1 is thus referred to as the time constant of wetting.

3 RESULTS AND DISCUSSION

3 REZULTATI IN RAZPRAVA

Figure 3 shows the measurement data and corresponding fits of exponential decay functions (eq. 1) on different spots of two MW-treated spruce specimens. The time-dependent progression of the measured WCAs varied strongly between individual points on all specimens regarding the initial contact angles directly after droplet deposition and the slope of the curve (i.e. time-dependent wetting), as

well as the final WCA. Moreover, all measurements show deviations from the continuous curvature beyond the statistical variations of individual values.

Figure 4 shows a statistical analysis of the final WCA as given by parameter γ_0 in eq. 1. The results are displayed such that the small square marks indicate the statistical mean value averaged over all measurements and specimens of the corresponding sample type, the coloured boxes enclose the 25%-75% medians, and the metering lines indicate 1.5 IQR (interquartile range), whereas outliers are marked by black diamonds.

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

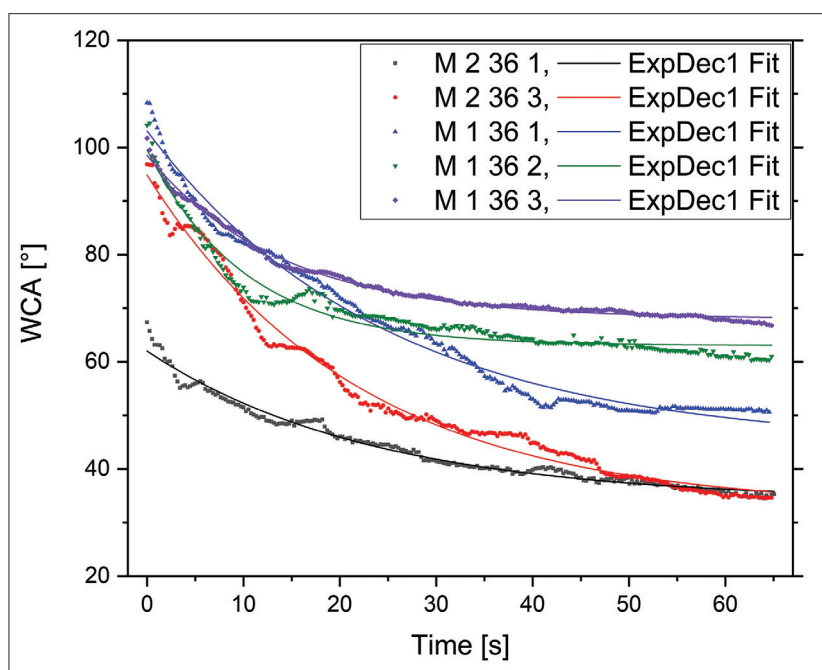


Figure 3. Example WCA measurement and exponential curve fit for MW-treated specimens.

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

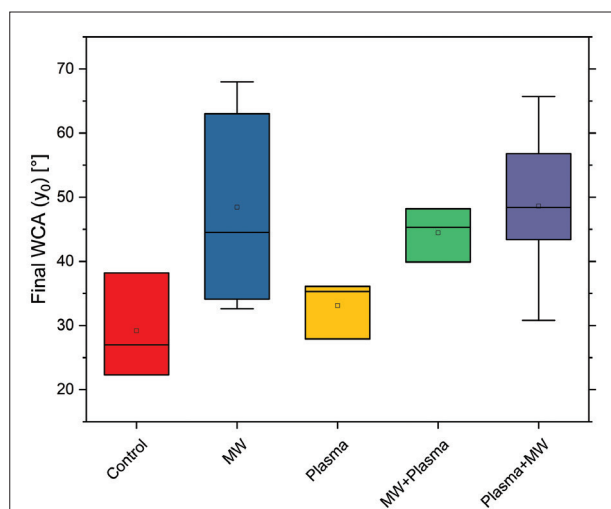


Figure 4. Statistical analysis of final WCA on differently treated specimens.

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

tively. Although this presents a reduction of the WCA by PT again after MW, the WCA does increase in all cases compared to the untreated control specimens. This indicates a stronger effect of drying and evaporation of VOCs towards larger WCAs than the impact of PT through the chemical production of additional polar groups on the structural constituents of the wood material. This increase of WCA on green wood after PT is contrary to most published literature (c.f. Žigon et al., 2018) and might not only be related to a drying of the outermost surface layers through enhanced evaporation of moisture induced by the plasma, but it might well relate further to a change in plasma-chemical reactions due to the water vapour above the wood surface. From various literature sources, it is well known that the plasma chemistry that occurs here changes with varying humidity levels in non-thermal air plasmas. In particular, the amount of oxygen radicals and consequently the production rate of ozone is reduced with the increasing presence of water molecules in the atmosphere, mainly via two effects: on the one hand, oxygen radical production and hydroxyl radical generation are competitive processes, both originating from dissociation after resonant energy transfer from metastable excited nitrogen molecules. On the other hand, water molecules act as scavengers for oxygen radicals to form hydroxyl radicals. Both mechanisms lead to

the reduced production of ozone and the correspondingly increased production of hydroxyl radicals, which further contribute to the formation of nitric oxide (Herron & Green, 2001; Prysiazhnyi et al., 2012). Moreover, these effects increase with the gas temperature (Sakiyama et al., 2012) and are thus more pronounced, for example, in gliding arc plasmas than in DBDs. These effects need to be further verified, e.g. by future measurements with Optical Emission Spectroscopy and X-ray Photoelectron Spectroscopy, in order to provide insights into both the plasma discharge and their effect on the yielded chemical changes on the surface. From the final WCA results, it is also noteworthy that the PT decreases statistical variations compared to the control specimens in terms of standard deviation, 1.5 IQR, and the ranges from first to third quartile medians. In contrast to that, standard deviation and 1.5 IQR are increased significantly by MW treatment, both on the untreated control and the previously plasma-treated specimens.

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

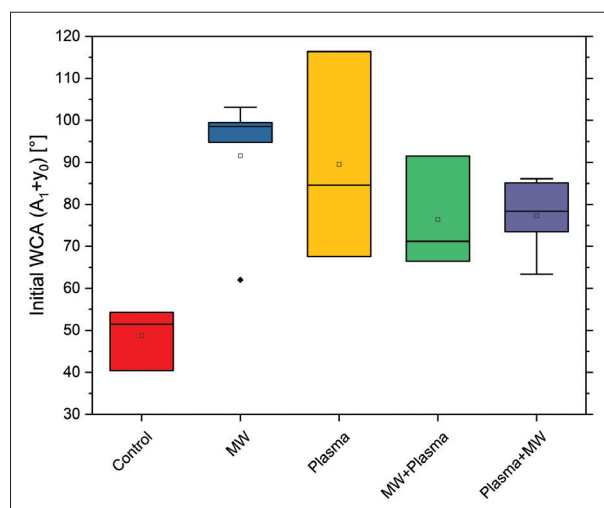


Figure 5. Statistical analysis of initial WCA on differently treated specimens.

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

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

Figure 6 shows a statistical analysis of the time constant t_1 as defined in eq. 1, representing the change in WCA over time as the droplet spreads across the sample surface, i.e. the wetting rate, whereas the concurrent penetration does not impact the measured WCA. The MW-only treated specimens exhibit an average wetting time of 19 s, equal to the untreated control specimens. The PT-only specimens show a lower wetting time of 15 s, whereas the combined treatments yield increased wetting times of 20 s and 23 s for PT following MW and MW after PT, respectively. However, all these values fall into each other's standard deviation and 1.5 IQR, and thus are not statistically sig-

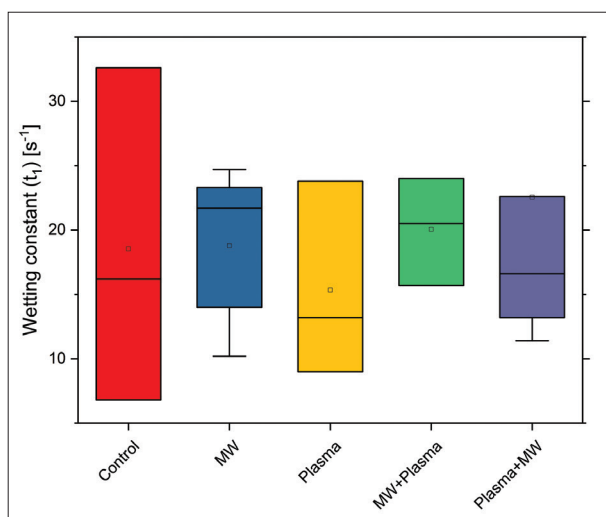


Figure 6. Statistical analysis of the time constant for wetting on differently treated specimens.

Slika 6. Statistična analiza časovne konstante omočenja na različno obdelanih vzorcih.

nificant. It does appear, though, that all MW-treated specimens show the lowest ranges between first and third quartile medians, whereas PT further reduces the 1.5 IQR. This might be an indication that the high degree of variation could be correlated with the moisture content at the wood surface.

4 CONCLUSIONS

4 SKLEPI

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

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

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

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

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

The specific utilization of electric energy per specimen volume amounted to 2700 MJ/m³ for MW, 5645 MJ/m³ for PT and 8345 MJ/m³ for combined MW+PT and PT+MW treatments. It should be noted that the use of PT as a surface treatment

strongly increases the energy efficiency for larger specimen volumes, thus being more favourable for industrial applications than small-scale laboratory experiments seem to indicate. However, the results clearly show that PT is not energy-efficient in terms of drying wood material, but its energy utilization and CO₂ equivalent should be evaluated in comparison to the surface finishing techniques and the corresponding materials replaced by PT.

SUMMARY

5 POVZETEK

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

Začetni stični koti vode neposredno po odlaganju kapljice na površino lesa ($A_1 + y_0$) so bili največji v primerih, ko smo les posamično obdelali bodisi z mikrovalovi ali plazmo. Kombinacija obdelave z mikrovalovi in plazmo je povzročila nižji začetni stični kot, ki je bil primerljiv ne glede na vrstni red obdelave. Statistična variabilnost začetnega stičnega kota je bila zmanjšana na vzorcih, obdelanih le z mikrovalovi, obdelava s plazmo pa je le-to močno povečala. To je verjetno posledica dejstva, da smo les obdelali z mikrovalovi v surovem, t.j. svežem stanju, kar je s tokom vlage na površino lesa omogočilo tudi migracijo organskih snovi, zlasti sladkorjev in ekstraktivov, s tem pa povzročilo homo-

genizacijo površine. V nasprotju s tem plazemska obdelava deluje heterogeno z večjo intenzivnostjo na območjih z večjo gostoto ali višjo električno prevodnostjo, kot je kasni les v primerjavi z ranim lesom, tako da plazma prispeva k povečanju kemične nehomogenosti površin.

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

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

Specifična poraba električne energije na prostornino vzorca znaša 2700 MJ/m³ za mikrovalovno obdelavo, 5645 MJ/m³ za plazemsko obdelavo in 8345 MJ/m³ za kombinirano obdelavo z mikrovalovi in plazmo. Treba je opozoriti, da plazma kot površinska obdelava močno poveča energijsko učinkovitost za večje količine vzorcev, kar je ugodno za industrijsko uporabo, kot nakazujejo majhni laboratorijski poskusi. Poleg tega rezultati jasno kažejo, da plazemska obdelava ni energetsko učinkovita za sušenje lesnega materiala, vendar je treba izrabo energije in ekvivalent CO₂ ovrednotiti v primerjavi s tehnikami površinske obdelave in ustreznimi materiali, ki jih plazma nadomesti.

Ganguly, S., Žigon, J., Srinivasa, K., Petrič, M., & Dahle, S.: The synergistic effect of microwave drying and plasma surface treatments on the wettability of green wood

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SUPPLEMENTAL INFORMATION AND RAW DATA

DODATNE INFORMACIJE IN PODATKI

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

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

REFERENCES

VIRI

- Avramidis, G., Wascher, R., Militz, H., & Viöl, W. (2016). Impact of air-plasma treatment at atmospheric pressure on wood and wood extractives. *International Wood Products Journal*, 7 (2), 76–79. DOI: <https://doi.org/10.1080/20426445.2016.1162429>
- Awoyemi, L. (2004). Effects of microwave modification on the kiln drying time of *Eucalyptus obliqua* wood. *Journal of Indian Academy of Wood Science*, 1, 83–88.
- Balboni, B. M., Ozarska, B., Garcia, J. N., & Torgovnikov, G. (2018). Microwave treatment of *Eucalyptus macrorhyncha* timber for reducing drying defects and its impact on physical and mechanical wood. *European Journal of Wood and Wood Products*, 76, 861–870. DOI: <https://doi.org/10.1007/s00107-017-1260-1>
- Bartoli, M., Frediani, M., Briens, C., Berruti, F., & Rosi, L. (2019). An overview of temperature issues in microwave-assisted pyrolysis. *Processes*, 7 (10), 658. DOI: <https://doi.org/10.3390/pr7100658>
- Boehme, C., & Hora, G. (1996). Water absorption and contact angle measurement of native European, North American and tropical wood species to predict gluing properties. *Holzforschung*, 50 (3), 269–276. DOI: <https://doi.org/10.1515/hfsg.1996.50.3.269>
- Brodie, G. (2010). Wood: Microwave modification of properties. *Encycl. Agric. Food Biol. Eng.*, 2010, 1878–1881.
- Dahle, S., Žigon, J., Petrič, M., & Kariž, M. (2020a). Plasma treatment of spruce wood changes its dielectric properties. *Les/Wood*, 69 (2), 83–95. DOI: <https://doi.org/10.26614/les-wood.2020.v69n02a03>
- Dahle, S., Žigon, J., Gospodarič, B., Zaplotnik, R., Merhar, M., & Petrič, M. (2020b). Open-source integrated plasma treatment and CNC machining system. Zenodo. DOI: <https://doi.org/10.5281/zenodo.4280597>
- Dahle, S., Ganguly, S., Žigon, J., Srinivasa, K., & Petrič, M. (2021). Raw and analyzed data for the manuscript “The synergistic effect of microwave drying and plasma surface treatments on the wettability of green wood”. (1.0.0) [Data set]. Zenodo. DOI: <https://doi.org/10.5281/zenodo.5835738>
- ISO standard EN 13183-1:2002 Moisture content of a piece of sawn timber—Part 1: Determination by oven dry method.
- Ganguly, S., Hom, S. K., Tripathi, S., Ghosh, S., Kanyal, R., & Samani, A. (2021a). Quantitative evaluation of microwave irradiation on short-rotation plantation wood species. *Maderas Ciencia y Tecnologia*, 23, 1–14.
- Ganguly, S., Balzano, A., Petrič, M., Kržišnik, D., Tripathi, S., Žigon, J., & Merela, M. (2021b). Effects of different energy intensities of microwave treatment on heartwood and sapwood microstructures in Norway spruce. *Forests*, 2021 (12), 598. DOI: <https://doi.org/10.3390/f12050598>
- Guanben, D., Siqun, W., & Zhiyong, C. (2005). Microwave drying of wood strands. *Drying Technology*, 23, 1–16.
- Haase, J. G., Leung, L. H., & Evans, P. D. (2019). Plasma pre-treatments to improve the weather resistance of polyurethane coatings on black spruce wood. *Coatings*, 9 (1), 8. DOI: <https://doi.org/10.3390/coatings9010008>
- Hämäläinen, K., & Kärki, T. (2013). Effects of atmospheric plasma treatment on the surface properties of wood-plastic composites. *Advanced Materials Research*, 718–720, 176–185. DOI: <https://doi.org/10.4028/www.scientific.net/AMR.718-720.176>
- He, S., Lin, L., Fu, F., Zhou, Y., & Fan, M. (2014). Microwave treatment for enhancing the liquid permeability of Chinese fir. *BioResources*, 9 (2), 1924–1938.
- Hermoso, E., & Vega, A. (2016). Effect of microwave treatment on the impregnability and mechanical properties of eucalyptus globulus wood. *Maderas Ciencia y Tecnologia*, 18, 55–64.
- Herron, J. T., & Green, D. S. (2001). Chemical kinetics database and predictive schemes for nonthermal humid air plasma chemistry. Part II. Neutral species reactions. *Plasma Chemistry and Plasma Processing*, 21, 459–481. DOI: <https://doi.org/10.1023/A:1011082611822>
- Hong-Hai, L., Qing-Wen, W., Lin, Y., Tao J., & Ying-Chun, C. (2005). Modification of larch wood by intensive microwave irradiation. *Journal of Forestry Research*, 16, 237–240. DOI: <https://doi.org/10.1007/BF02856823>
- Kalnins, M., Katzenberger, C., Schmieding, S., & Brooks, J. (1988). Contact angle measurement on wood using videotape technique. *Journal of Colloid and Interface Science*, 125 (1), 344–346. DOI: [https://doi.org/10.1016/0021-9797\(88\)90084-7](https://doi.org/10.1016/0021-9797(88)90084-7)
- Klarhöfer, L., Viöl, W., & Maus-Friedrichs, W. (2010). Electron spectroscopy on plasma treated lignin and cellulose. *Holzforschung*, 64, 331–336. DOI: <https://doi.org/10.1515/hf.2010.048>

- Král, P., Ráhel, J., Stupavská, M., Šrajter, J., Klímek, P., Mishra, P. K., & Wimmer, R. (2015). XPS depth profile of plasma-activated surface of beech wood (*Fagus sylvatica*) and its impact on polyvinyl acetate tensile shear bond strength. *Wood Science and Technology*, 49, 319-330. DOI: <https://doi.org/10.1007/s00226-014-0691-7>
- Krapež Tomec, D., Žigon, J., Kitek Kuzman, M., Dahle, S., Šernek, M., & Kariž, M. (2021). Effect of plasma treatment of 3D printed parts on bonding to wood with PVAc adhesive. *Polymers*, 13 (8), 1211. DOI: <https://doi.org/10.3390/polym13081211>
- Liu, H. H., Wang, Q. W., Yang, L., Jiang, T., & Cai, Y. C. (2005). Modification of larch wood by intensive microwave irradiation. *Journal of Forestry Research*, 16 (3), 237-240. DOI: <https://doi.org/10.1007/BF02856823>
- Mekhtiev, M. A., & Torgovnikov, G. (2004). Method of check analysis of microwave-modified wood. *Wood Science and Technology*, 38 (7), 507-519. DOI: <https://doi.org/10.1007/s00226-004-0259-z>
- Melamies, I. A. (2014). Feeling the rush of speed—New impregnation process for race skis. *JOT International Surface Technology*, 3.2014, 32-35.
- Oloyede, A., & Groombridge, P. (2000). The Influence of microwave heating on the mechanical properties of wood. *Journal of Materials Processing Technology*, 100, 67–73.
- Ouertani, S., Hassini, L., Azzouz, S., Torres, S. S., Belghith, A., & Koubaa, A. (2015). Modeling of combined microwave and convective drying of wood: prediction of mechanical behavior via internal gas pressure. *Drying Technology*, 33, 1234-1242. DOI: <https://doi.org/10.1080/07373937.2015.1022828>
- Papp, E. A., & Csiha, C. (2017). Contact angle as function of surface roughness of different wood species. *Surfaces and Interfaces*, 8, 54-59. DOI: <https://doi.org/10.1016/j.surfin.2017.04.009>
- Prysiaznyh, V., Zaporozhen, V., Kersten, H., & Černák, M. (2012). Influence of humidity on atmospheric pressure air plasma treatment of aluminium surfaces. *Applied Surface Science*, 258 (14), 5467-5471. DOI: <https://doi.org/10.1016/j.apsusc.2012.02.040>
- Rehn, P., Wolkenhauer, A., Bente, M., Förster, S., & Viöl, W. (2003). Wood surface modification in dielectric barrier discharges at atmospheric pressure. *Surface and Coatings Technology*, 174–175, 515-518. DOI: [https://doi.org/10.1016/S0257-8972\(03\)00372-4](https://doi.org/10.1016/S0257-8972(03)00372-4)
- Riedl, B., Angel, C., Prigent, J., Blanchet, P., & Stafford, L. (2014). Effect of wood surface modification by atmospheric-pressure plasma on waterborne coating adhesion. *Bioresources*, 9 (3), 4908-4923.
- Sakiyama, Y., Graves, D. B., Chang, H.-W., Shimizu, T., & Morfill, G. E. (2012). Plasma chemistry model of surface microdischarge in humid air and dynamics of reactive neutral species. *Journal of Physics D: Applied Physics*, 45 (42), 425201. DOI: <https://doi.org/10.1088/0022-3727/45/42/425201>
- Samani, A., Ganguly, S., Kanyal, R., & Tripathi, S. (2019). Effect of microwave pre-treatment on preservative retention and treatability of *Melia composita* wood. *Journal of Forest Science*, 65, 391–396.
- Sethy, A. K., Torgovnikov, G., Vinden, P., & Przewloka, S. (2016). Moisture conditioning of wood using a continuous microwave dryer. *Drying Technology*, 34, 318–323.
- Šernek, M. (2002). Influence of drying temperature on properties of wood surfaces. *Zbornik gozdarstva in lesarstva*, 67, 173-191. <http://eprints.gozdis.si/id/eprint/315>
- Terziev, N., Daniel, G., Torgovnikov, G., & Vinden, P. (2020). Effect of microwave treatment on the wood structure of Norway spruce and radiata pine. *Bioresources*, 15 (3), 5616-5626. DOI: <https://doi.org/10.15376/biores.15.3.5616-5626>
- Torgovnikov, G., & Vinden, P. (2009). High-intensity microwave wood modification for increasing permeability. *Forest Products Journal*, 59, 84–92.
- Torgovnikov, G., & Vinden, P. (2010). Microwave wood modification technology and its applications. *Forest Products Journal*, 60, 173–182.
- Treu, A., Rieche, H., & Militz, H. (2008). Spruce and pine heartwood treatment by means of microwave radiation. In: *Proceedings of the 39th Annual Meeting the International Research Group on Wood Protection*, 2008.
- Wascher, R., Avramidis, G., & Viöl, W. (2021). Plywood made from plasma-treated veneers: investigation of performance differences between plasma-pretreated and untreated beech veneers at comparable melamine resin load. *Forests*, 12 (10), 1423. DOI: <https://doi.org/10.3390/f12101423>
- Weng, X., Zhou, Y., Fu, Z., Gao, X., Zhou, F., & Jiang, J. (2021). Effects of microwave pretreatment on drying of 50 mm-thickness Chinese fir lumber. *Journal of Wood Science*, 67, 1–9.
- Weng, X., Zhou, Y., Fu, Z., Gao, X., Zhou, F., & Fu, F. (2020). Effects of microwave treatment on microstructure of Chinese fir. *Forests*, 11, 772. DOI: <https://doi.org/10.3390/f11070772>
- Žigon, J., Petrič, M., & Dahle, S. (2018). Dielectric barrier discharge (DBD) plasma pretreatment of lignocellulosic materials in air at atmospheric pressure for their improved wettability: A literature review. *Holzforchung*, 72 (11), 979-991. DOI: <https://doi.org/10.1515/hf-2017-0207>
- Žigon, J., Pavlič, M., Kibleur, P., van den Bulcke, J., Petrič, M., van Acker, J., & Dahle, S. (2020a). Treatment of wood with atmospheric plasma discharge: study of the treatment process, dynamic wettability and interactions with a waterborne coating. *Holzforchung*, 75 (7), 603-613. DOI: <https://doi.org/10.1515/hf-2020-0182>
- Žigon, J., Kovač, J., Zaplotnik, R., Saražin, J., Šernek, M., Petrič, M., & Dahle, S. (2020b). Enhancement of strength of adhesive bond between wood and metal using atmospheric plasma treatment. *Cellulose*, 27, 6411-6424. DOI: <https://doi.org/10.1007/s10570-020-03212-8>