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Impregnability of Paulownia and Populus Wood with Copper Based Preservatives

Učinci impregnacije drva paulovnije i topole zaštitnim sredstvima na bazi bakra

Original scientific paper • Izvorni znanstveni rad
Received – prispjelo: 11. 1. 2017.
Accepted – prihvaćeno: 30. 8. 2017.
*UDK: 630*841.3; 630*.841.4; 674.031.623.234; 674.031.951.62*
doi:10.5552/drind.2017.1701

ABSTRACT • *The importance of fast growing wood species such as Paulownia and Populus wood is increasing. Unfortunately, these wood species do not have durable wood, so they have to be protected for use in outdoor applications. One of the most important groups of wood preservatives for heavy duty applications is the copper based one. Paulownia and Populus wood samples were thus treated with copper-ethanolamine (Cu/MEA) and acid copper chromate (ACC) according to various treatment processes: Bethell, Rüping and dipping. The dipping treatment was performed with various submersion times, ranging from 1 min to 10 days. The uptake of preservative solution, penetration and colour of the specimens were then determined. The results clearly indicate that the permeability of Paulownia is not as good as that of Populus wood. The permeability of Paulownia can be improved with the proper selection of impregnation procedure and wood preservatives. Cu/MEA was found to be more suitable for treatment of Paulownia wood than classical ACC.*

Keywords: *impregnation, dipping, Paulownia, Populus, penetration, uptake of preservative solution*

SAŽETAK • *Važnost brzorastućih vrsta drveća kao što su paulovnija (Paulownia) i topola (Populus) svakim danom postaje sve veća. Nažalost, drvo tih vrsta nije baš trajno, stoga se prije uporabe u vanjskim uvjetima mora zaštititi. Jedna od najvažnijih skupina sredstava za zaštitu drva za uporabu u nepovoljnim uvjetima jesu ona na bazi bakra. Uzorci drva paulovnije i topole tretirani su bakar-etanolaminom (Cu/MEA) i acetatnim bakrenim kromatom (ACC) različitim postupcima obrade: Bethellovim, Rüpingovim i potapanjem. Postupak je proveden uz različita vremena uranjanja, u rasponu od 1 min do 10 dana. Tijekom eksperimenta određivana je količina upijene otopine zaštitnog sredstva, dubina njezine penetracija i boja uzoraka. Rezultati jasno pokazuju da permeabilnost paulovnije nije baš dobra kao permeabilnost drva topole. Permeabilnost paulovnije može se poboljšati pravilnim izborom postupka impregnacije i zaštitnog sredstva. Istraživanje je potvrdilo da je Cu/MEA prikladniji za zaštitu drva paulovnije od klasičnoga ACC-a.*

Ključne riječi: *impregnacija, potapanje, paulovnija, topola, penetracija, upijanje otopine zaštitnog sredstva*

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1 INTRODUCTION
1. UVOD

Wood is one of the most commonly used raw materials in a wide variety of applications. However, when wood is used in outdoor applications, it is exposed to a variety of degradation factors (Brischke and Rapp, 2008). Among them, fungi are the most important in the majority of exposure conditions (Despot, 1998). Some very durable (durability class 1) wood species are available on the global market, such as teak, makoré, padouk, opepe and doussié (EN 350) (CEN, 2016). However, the majority of wood species and, consequently, the majority of wood on the market is not durable, so it has to be protected for use in outdoor applications (Brischke *et al*., 2013).

However, wood, as a regenerating and thus renewable resource, is not available indefinitely in terms of volumes and regional availability. In recent years, increasing competition for wood, intensified by rising prices for fossil fuels, has been observed. Shortages of wood are expected due to increased consumption of wood by developing countries, climate changes, etc. (Schwarzbauer and Stern, 2010). Industry will, therefore, have to focus on fast-growing species, such as *Paulownia* and *Populus*, as raw material. *Paulownia* is a fast-growing tree and can be used in a variety of applications, such as furniture, construction, aircraft and packaging (Flynn and Holder, 2001). It is commercialized as a wood species, with a good ratio between density and mechanical properties. However, its low permeability due to tylosis formation (Ghorbani *et al*., 2012) causes problems in penetration of wood preservatives on the one hand but, on the other hand, improves water resistance, which has been identified as the second most important factor contributing to the performance of wood in above ground applications (Žlahtič *et al*., 2015).

The selection of the wood preservative is one of the key steps to determine the use of treated wood (Willeitner, 2001; Melcher and Müller, 2016). The only active ingredients suitable for heavy duty applications worldwide are the copper based ones (Humar *et al.*, 2001; Humar and Lesar, 2013). The majority of the alternatives have been removed from the market or banned since December 2003 after the implementation of the Biocidal Products Directive (BPD 98/8/EC 1998) and Regulation (EU) No. 528/2012 (2012). At the moment, there are three leading groups of copper based wood preservatives on the world market: classical copper-chromium based preservatives, copper amine based solutions and micronized copper. Three different types of Alkaline copper quat (ACQ) formulations for the treatment of wood have been developed over time (Type B, C and D). Type D is manufactured using a monoethanolamine copper formulation (Cu-MEA) (AWPA, 2014). Waterborne copper-monoethanolamine (Mea) has completely replaced chromated copper arsenate (CCA) in the EU, although copper-chromium based solutions are still the key preservatives in the rest of the world. In addition, in the US, copper-amine solutions have been replaced by preservative solutions based on micronized copper, particularly for residential applications. Unfortunately, micronized copper is not suitable for refractory wood species. The particles in the suspension are too big to penetrate through the pit voids. In some countries, acid copper chromate (ACC) is used as an alternative. ACC is a leach-resistant preservative recommended for use above ground and for non-structural items in ground contact and it has been used in Europe and the United States since the 1920s (Preston, 2000; Humar *et al.*, 2006; Civardi *et al.*, 2015).

The aim of the present study was to elucidate whether two fast growing species, *Populus* and *Paulownia*, are suitable for impregnation with the currently most important copper based wood preservatives (acid copper chromate and copper-monoethanolamine). The prime objectives of the present study were hence to determine the influence of wood preservatives, retention and treatment methods on the permeability of *Paulownia fortunie* and *Populus deltoids*.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

Three trees of both *Paulownia fortunei* and *Populous deltoids* were harvested from Gorgan Province, located in north-eastern Iran extending from 36° 46' to 36° 46'40'' N and 54° 22' to 54° 22'50'' E with elevation ranging between 270 to 350 m above sea level. For this study, specimens of *Paulownia fortunie* and *Populus deltoids* of the following dimensions were prepared: 15 mm (T) × 15 mm (R) × 50 mm (L). All specimens were free from defects (knots, checks, rots or blue stain). They were prepared from the adult part of the tree. The experiments were performed with 4 parallel specimens. In total, 88 specimens were used.

The following wood preservative solutions were used for impregnation: Cu/MEA consisted of $CuCO_3$ (Merck) and C_2H_7NO), while ACC was a mixture of $CuSO_4 \times 5H_2O$ (Merck) and $K_2Cr_2O_7$ (Merck). The copper concentration was the same in both solutions (c_{Cu} = 0.5 %). The samples were air dried in laboratory conditions (23 °C; 60 % RH) for four weeks. The first week in the closed chambers, then in half opened, and the last week in completely opened chambers.

Before the treatment process, samples were divided into series. One series of samples was sealed with an epoxy resin (Epikote 828, USA) so that penetration and retention values would specify radial/or tangential pathways, while series of samples were taken without end sealing to give an indication of the absorption/penetration of the preservative from all sides.

In order to fully elucidate the impregnability of the investigated materials, various treatment procedures were applied: Bethell (full cell), Rüping (empty cell) and dipping treatment (various dipping times). These treatment procedures were applied to both preservative solutions used. The specimens were dried and the dry weight was recorded before impregnation. Specimens were weighed before and after impregnation. The uptakes of preservative solutions were determined gravimetrically from the mass difference and the volume of the specimens.

Prior to treatment, specimens were placed in glass jars and submerged in the preservative. Glass bars were used to prevent their floating. During the modified Bethell process, wood specimens were placed in a pilot plant tank and subjected to a vacuum of (-0.8 bar) for 15 min, followed by pressure at 8 bar for 60 min. In the Rüping treatment, all procedures were performed with similar parameters. However, the initial pressure of 2 bar for 15 min was followed by a pressure of 8 bar for 60 min. During the dipping treatment, specimens were dipped into Cu monoethanolamine and ACC solutions for 1, 10, 1440, 2880, 4320, 8640, 11520 and 14400 minutes to establish the influence of different dipping times. Impregnation was performed at room temperature. The specimens were then wiped lightly to remove the preservative solution from the surface and immediately weighed to the nearest 0.01 g.

Impregnated specimens were air dried for four weeks. Thereafter, the specimens were dried in an oven at 103±3 for 24 h and weighed to the nearest 0.01 g. The penetration of preservatives in longitudinal and transverse directions was determined by spraying with Chrome Azurol S solution to indicate the presence of copper. The wood turned blue where the CCA and Cu/Mea had penetrated, whereas untreated zones were coloured red. The area of the treated zone and its percentage of the total cross section were calculated visually by measuring the penetration of preservative in each specimen.

The lightness of samples was measured using a TES-135 (Taiwan) based on the ASTM 2244 standard specification, where the lightness value ranged from 100 (white) to zero (dark). The measurement of lightness in $L^*a^*b^*$ coordinates was repeated at six locations on each sample. Colour measurements were performed, as the colour is the first indication of copper absorption. With copper based preservatives, the selective absorption of copper commonly appears. Hence, the surface uptakes more copper than assumed from the uptake of preservative solution (Humar and Lesar, 2009). CIELAB system characterized colours by three parameters: L^*, a^* and b^*. L axis represents the lightness whereas, a^* and b^* are the chromaticity coordinates. In the CIELAB coordinates, $+a^*$ stands for red, $-a^*$ for green, $+b^*$ for yellow, $-b^*$ for blue, and L^* varies from 100 (white) to zero (black). The total colour change was calculated using the following equation (1).

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

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

Experiments were performed on *Paulownia fortunie* and *Populus deltoids* specimens with a dry density of the respective specimens between 0.28 and 0.42 g/cm^{-3}. The density of *Paulownia* is considerably lower than that of *Populus*. This must be taken into account when interpreting the results. However, these densities are slightly lower than the ones reported for Spruce (300...430...640 kg/m^3) (Wagenführ, 1996).

The uptake of preservative solution is the first essential information that indicates the quality of the treatment. Additionally, this parameter is the only one that can be monitored during the process of impregnation. If the uptake of preservative solution is not sufficient, neither penetration nor retention, (retention is the uptake of active ingredients in wood after impregnation, usually prescribed by specifiers), can meet the specified requirements (Humar and Lesar, 2009).

Uptake of preservative solutions data are presented in Table 1. The uptake of preservatives increased with increasing immersion times. After one minute of dipping, wood samples retained approximately 10 kg/m^3 of wood preservatives, while after 14400 min, they retained 196 kg/m^3 (Cu/MEA) and 65 kg/m^3 (AAC) of wood preservatives in *Populus* wood samples. The uptake of preservative solutions in *Paulownia* wood was slightly lower. Literature data revealed that with spruce wood comparable uptakes can be achieved, namely after 1 min, samples uptake 12 kg/m^3 of Cu/MEA based preservatives. In addition, seven days of immersion resulted in an average uptake of 152 kg/m^3 (Humar and Lesar, 2009). However, these values are difficult to compare, as different methodologies were applied. In addition, respective study on spruce wood clearly indicates that the uptake of preservative solution is considerably influenced by concentration. The Cu/MEA solution of higher concentration penetrates better than the ones of lower concentration (Humar and Lesar, 2009). It is believed that the prime reason for lower penetration of wood preservatives to *Paulownia* wood is that there are more tyloses in *Paulownia* wood than in *Populus*. However, similar ratios between the uptake of AAC and Cu/MEA wood preservatives can also be determined with *Paulownia* wood. It can be assumed that the primary reason for better uptake of Cu/MEA is that the solvent in Cu/MEA, ethanolamine, is a chemical that increases wood swelling (Mantanis *et al.*, 1994). As the cellulose armature swells, the wood enlarges, which enables better penetration of copper/ethanolamine based wood preservative. This phenomenon was more evident with specimens that were dipped for a longer period, since the wood has more time to swell and uptake more wood preservative. This phenomenon has already been elucidated and discussed (Humar *et al.*, 2012).

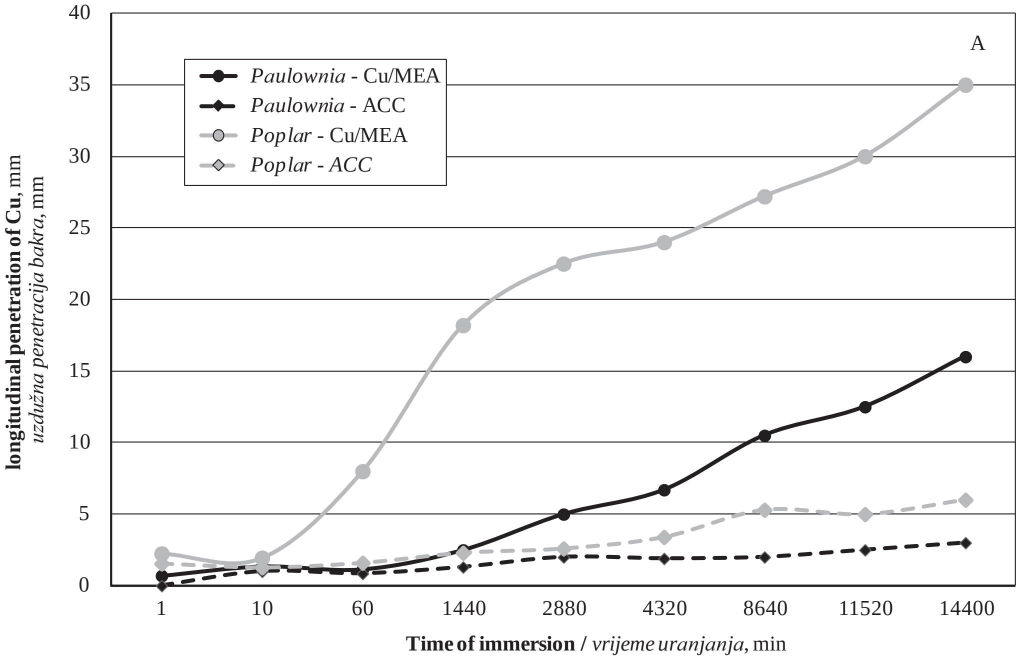
The highest uptake of wood preservatives was determined with specimens treated according to the Bethell full cell process. This is understandable and was expected (Humar and Lesar, 2009), since the full cell process is an impregnation procedure that fills the lumina of the cells with wood preservatives. Since this process is fairly severe and the wood specimens were fairly small, almost no difference was observed between the wood preservatives applied and the wood species used. On the other hand, these data indicate that even refractory wood of *Paulownia* can be successfully impregnated if the proper procedure is applied. The specifiers usually prescribe the amount of the active ingredients that have to penetrate wood. Hence, it is up to the impregnation facility how this requirement will be achieved. In Europe, wood preservation is based on several standards. The essential standard is EN 351-1 (CEN, 2007). This standard prescribes the penetration classes and the treated zone. There are six penetration classes ranging from NP1 (there are no requirements) to NP6 (full sapwood penetration and 6 mm penetration to exposed heartwood). Spruce wood is usually required to meet penetration class NP3 (penetration of 6 mm), while penetration class NP5 (full sapwood penetration) is required for Scots pine wood. Contrary to penetration, end users and specifiers usually prescribe retention. Retention requirement is to uptake the formulation/active ingredients expressed in kg per m3 of wood in the treated zone. This information is usually based on the extensive field testing based on the standard EN 599-1 (CEN, 2009) that prescribes which tests needs to be performed for the use in the respective use class. The list provided by the Nordic Wood Preservation Council (2008, 2015) is the most frequently used reference by end users who specify the orders of impregnated wood. If wood is not treated correctly, premature failures appear (Humar and Thaler, 2017), which leads to bad reputation of wood preservation in general.

Penetration is another important parameter that indicates the quality of the impregnation process. The specifiers usually prescribe penetration related to specific use class and wood species, as sometimes it is difficult to achieve target penetration to refractory wood species. For use class 3.2 and use class 4, full sapwood penetration is required for pine wood, and 8 mm for spruce wood. Due to the refractory nature of spruce wood, this wood is not recommended for applications in in-ground applications, even if treated (Humar *et al.*, 2017). In contrast to the uptake of preservative solution, this parameter cannot be controlled during the impregnation process. However, good uptake of preservative solution does not ensure a sufficient service life if the active ingredients remain on the surface layer. This is known as the chromatographic effect. As can be seen in Table 1, the time of immersion and impregnation process has a considerable effect on copper penetration (Figure 1). Penetration in the longitudinal direction is five to ten times better than in the radial direction. For example, with *Paulownia* Bethell treated with Cu/MEA, the copper penetrated 40 mm in the longitudinal, 5 mm in the tangential and 6 mm in the radial direction. Penetration during the Rüping process was lower, as was expected. However, both data indicate that *Paulownia* is a refractory wood species. During commercial impregnation, penetration classes with penetration more than 10 mm in radial and tangential directions are usually required. This indicates that after pressure impregnation, the so called shell treatment has been achieved, whereby only the outer layers are protected and the interior is left untreated. However, penetration can be improved with prolongation of the impregnation time, increased pressure or incising.

An alternative treatment suitable for less exposed wood applications is dipping. This process is predominantly suitable for impregnation plants with small capacities, since this technology does not require as much investment as a proper impregnation plant (Humar and Lesar, 2009). As can be seen in Figure 1, penetration of copper increased with immersion time in all directions. The highest increase was recorded after the first day of immersion in all directions. After ten days of dipping, the specimens sometimes reached the same values as recorded after the full cell process (Figure 1D). For example, radial penetration of Cu/MEA into *Paulownia* reached 9 mm, which is better than reported after Bethell or Rüping treatments. It is interesting that penetration in the longitudinal direction was better with *Populus* than with *Paulownia* wood, probably due to the presence of tyloses in *Paulownia* wood. However, in tangential and radial directions, better penetration

Table 1 Influence of wood species and impregnation method on the uptake of preservative solutions into unsealed samples. Standard deviations are presented in parentheses
Tablica 1. Utjecaj vrste drva i metode impregnacije na upijanje zaštitnog sredstva (standardna odstupanja prikazana su u zagradama)

Wood species *Vrsta drva*		***Populus*** *Topola*		***Paulowni*** *Paulovnija*	
Preservative solution *Otopina zaštitnog sredstva*		Cu/MEA	ACC	Cu/MEA	ACC
Treatment method *Metoda tretiranja*	**Time of immersion** *Vrijeme potapanja*, min	**Uptake of preservative solution, kg/m³** *Upijanje otopine zaštitnog sredstva*, kg/m³			
Dipping *potapanje*	1	10.5 (04)	9.0 (01)	8.0 (02)	4.2 (01)
	10	10.6 (07)	10.5 (02)	12.1 (09)	7.0 (06)
	60	24.9 (03)	13.9 (08)	14.3 (09)	9.7 (05)
	1440	75.0 (05)	26.2 (05)	43.1 (01)	18.7 (04)
	2880	86.1 (10)	29.4 (05)	57.5 (03)	21.5 (08)
	4320	118.9 (07)	37.2 (11)	67.7 (01)	25.5 (22)
	8640	150.0 (16)	46.4 (09)	90.7 (07)	35.5 (03)
	11520	155.8 (22)	52.7 (13)	99.0 (05)	37.0 (08)
	14400	196.2 (23)	64.7 (04)	117.0 (12)	40.0 (15)
Rueping process /*Ruepingov postupak*		288.0 (03)	275.5 (25)	155.0 (06)	175.0 (43)
Bethell process / *Bethellov postupak*		365.0 (13)	329.0 (42)	330.0 (57)	320.0 (44)



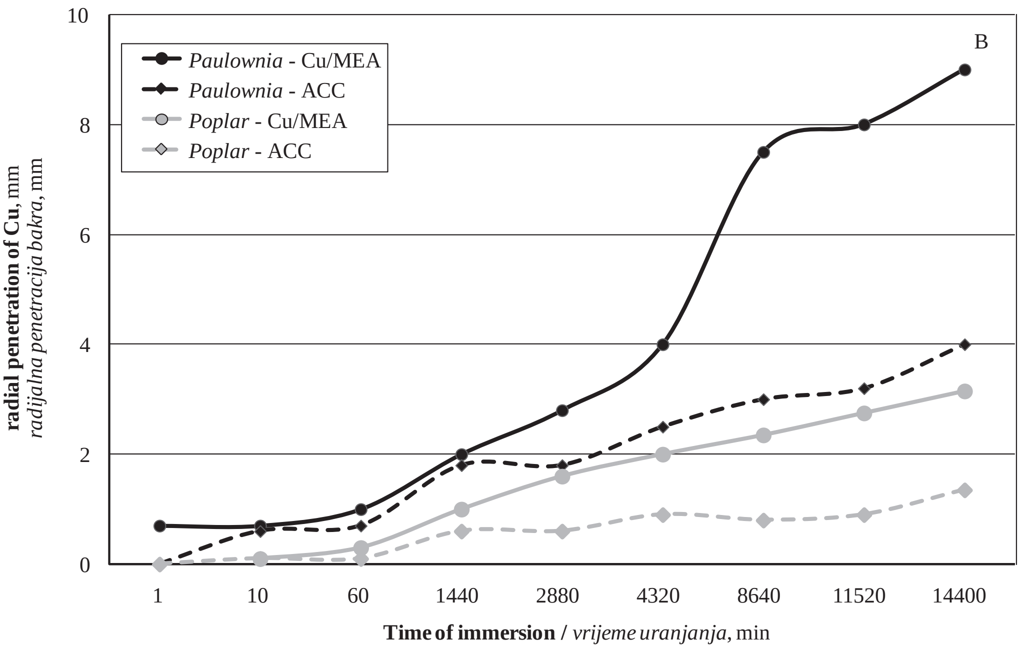
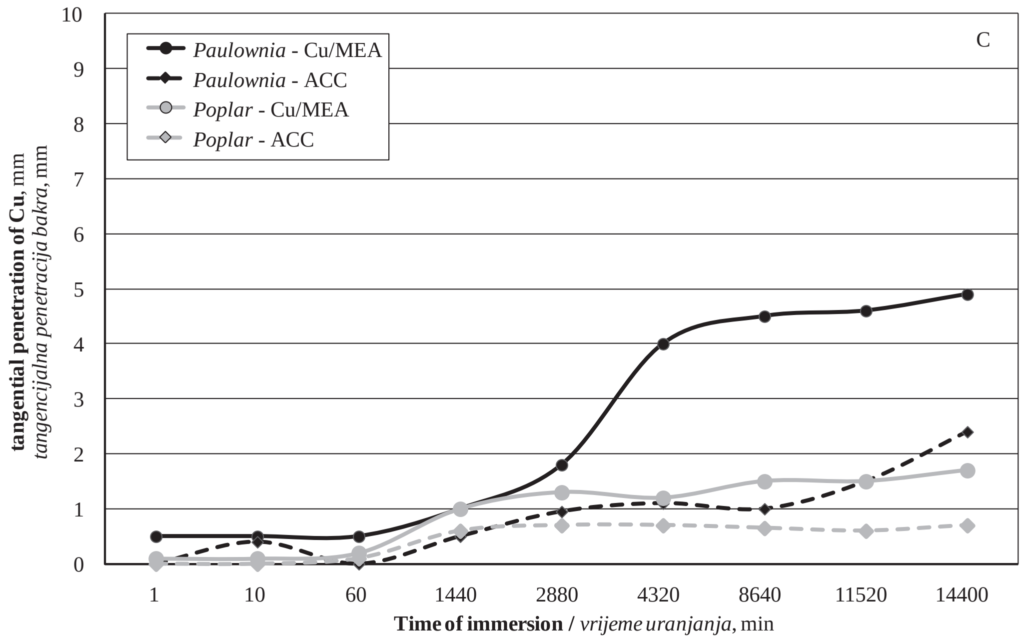


Figure 1 Influence of preservative solution and immersion time on penetration of preservative solutions in longitudinal (A), radial (B) and tangential (C) directions. Penetration after Bethell and Rueping process is presented in plot D.
Slika 1 Utjecaj otopine zaštitnog sredstva i vremena potapanja uzoraka na penetraciju otopine zaštitnog sredstva u uzdužnome (A), radijalnome (B) i tangencijalnome (C) smjeru; penetracija nakon Bethellova i Ruepingova postupka tretiranja prikazana je grafom D

was achieved with *Paulownia* than with *Populus* wood. This phenomenon was noted with both preservative solutions applied. In addition, it was noted that Cu/MEA penetrated much better than ACC, presumably due to the already explained phenomenon of wood swelling caused by ethanolamine.

However, it should be noted that, during long term dipping, selective absorption of copper can occur on wood constituents (Humar and Lesar, 2009). This is reflected in the fact that the outer layer contains more copper than indicated by the uptake of preservative solution. Additionally, users should also be aware that the strength of the solution decreases with the number of impregnation cycles. This phenomenon is also reflected in the colour (Figure 2) (Humar and Lesar, 2009).

Total colour changes increased with increasing dipping time and correlate with the retention. The most prominent change appeared during the first minute of immersion. This indicates that the reaction between active ingredients and wood is fairly fast. However, total changes were more prominent with Cu/MEA treated wood than with ACC. This can be linked to the uptake of wood preservatives, since it has already been reported that Cu/MEA penetrates wood better. Colour is one



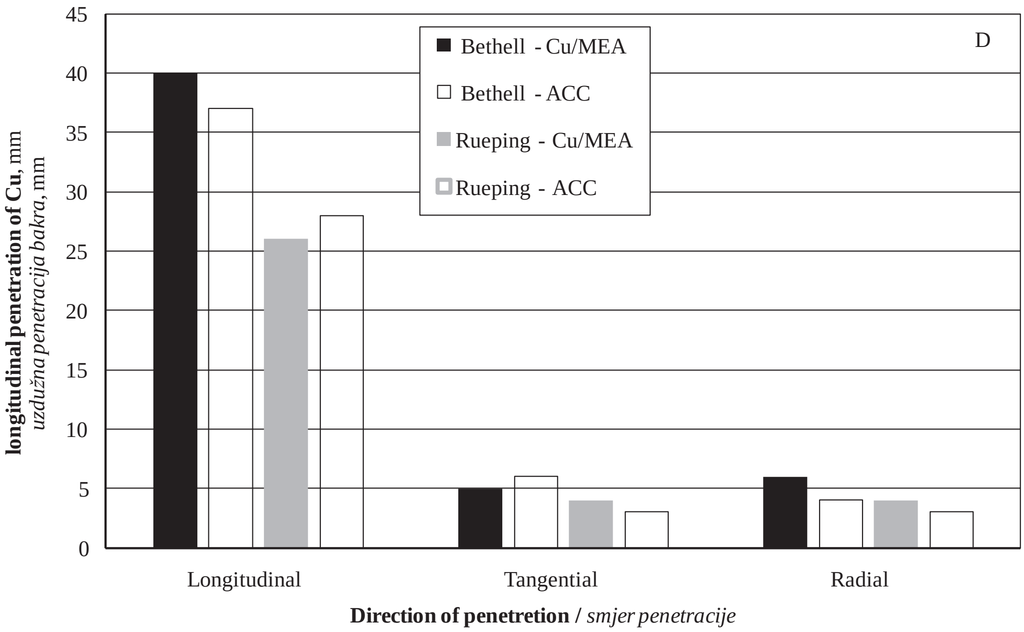


Figure 1. Continued
Slika 1. Nastavak

of the issues related to user expectations. The darker tones of Cu/MEA treated wood were considered negative in the initial period of introduction of these wood preservatives. Nowadays, this colour is generally expected.

4 CONCLUSIONS
4. ZAKLJUČAK

The treatability of *Paulownia* wood is not as good as that reported for *Populus* wood. However, if the proper procedure is applied, *Paulownia* wood can be expected to meet the specified requirements. However, Cu/MEA based wood preservatives were found more suitable for *Paulownia* wood than classical ACC, due to better penetration. *Paulownia* was found to be particularly suitable for long term dipping treatments, in which it performed better than *Populus*. Colour changes of *Paulownia* after treatment were comparable or even less prominent than those reported for *Populus* wood.

Acknowledgment – Zahvala

The authors would like to acknowledge the partial support of the University of Gorgan under project number 4-336-94 and the Slovenian Research Agency ARRS in the framework of the programme P4-0015.

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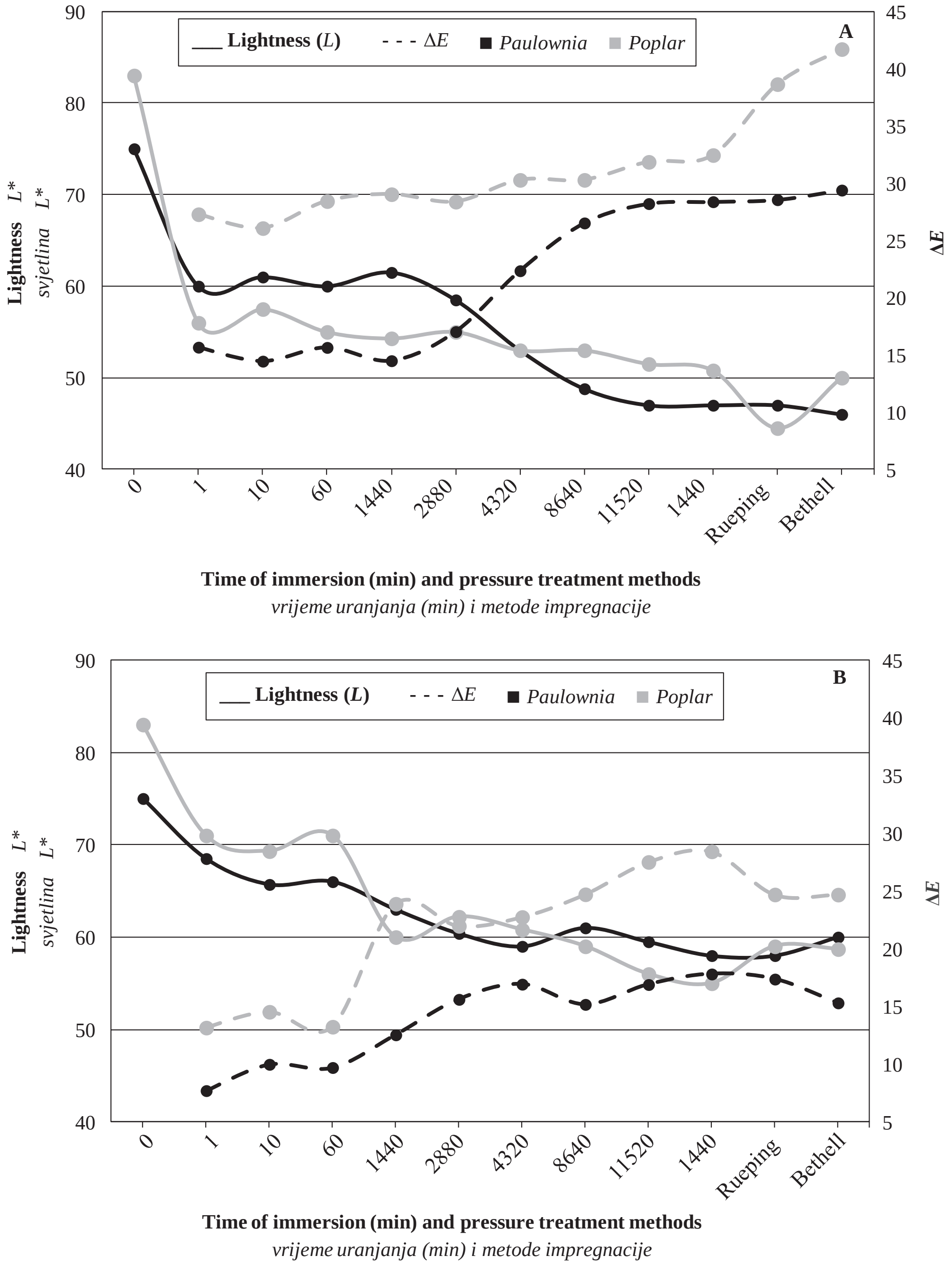

Figure 2 Influence of wood species and impregnation method on lightness (L^*) and total colour change (ΔE) of treated wood. Plot A shows the changes after impregnation with Cu/MEA, while plot B reflects changes of ACC treated wood.
Slika 2 Utjecaj vrste drva i metode impregnacije na svjetlinu (L^*) i ukupnu promjenu boje (ΔE) tretiranog drva; graf A predočuje promjene nakon impregnacije Cu/MEA-om, a graf B prikazuje promjene drva tretiranoga ACC-om

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