

Weathering Resistance of Poplar Wood Coated by Organosilane Water Soluble Nanomaterials

Otpornost prema vremenskim utjecajima topolovine premazane vodenom otopinom organosilana u nanovelčinama

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ABSTRACT • The potential use of organosilane nanomaterials (nano-zycosil and nano-zycofil) for improving the weathering resistance of poplar wood was evaluated in comparison to common clear coatings (nitrocellulose and polyester lacquer). A 250 μm coating layer was applied by an automatic film applicator at the speed of 150 mm/s. The coated specimens were exposed to a 1000 W xenon arc light source at 65 % relative humidity and temperature of 20 °C inside a weather-o-meter for 1440 hrs. Among coating materials, nano-zycosil showed the best performance to improve the weathering resistance. Compared to the lacquer-coated samples, the roughness of nanoparticle-coated ones was less affected by weathering. Contact angle measurements indicated that nano-zycosil coating had a pronounced decreasing effect on the surface wettability. The combined analyses of SEM and EDX demonstrated that the nanoscale silane layer covered the whole wood surface homogeneously, whereas nitrocellulose and polyester coatings were deposited preferentially in the surface depressions.

Keywords: clear coatings, organosilane nanomaterials, weathering, wood

SAŽETAK • U radu je prikazano istraživanje mogućnosti uporabe organosilana u nanovelčinama (nano-zycosil i nano-zycofil) za povećanje otpornosti topolovine na vremenske utjecaje u usporedbi s uporabom klasičnih prozirnih premaznih materijala (nitroceluloznoga i poliesterskoga premaza). Sloj premaza od 250 μm nanesen je strojno, brzinom 150 mm/s. Premazani su uzorci 1440 sati bili izloženi izvoru svjetlosti ksenonske svjetiljke od 1000 W pri relativnoj vlazi zraka od 65 % i na temperaturi od 20 °C. Od premaznih materijala nano-zycosil se pokazao najboljim za povećanje otpornosti na vremenske utjecaje. U usporedbi s uzorcima obrađenim klasičnim premaznim materijalima, hrapavost nanopremaza bila je manje uvjetovana izlaganjem vremenskim utjecajima. Mjerenja kontaktnog kuta pokazala su da premaz nano-zycosilom znatno utječe na smanjenje kvašenja površine. Kombinirane analize SEM i EDX potvrdile su da sloj silana u nanovelčini ravnomjerno prekriva površinu drva, dok je sloj nitroceluloznoga i poliesterskog premaza u udubljenjima na površini deblji.

Ključne riječi: prozirni premazi, organosilani nanovelčine, izlaganje vremenskim utjecajima, drvo

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1 INTRODUCTION

1. UVOD

Wood surfaces exposed to outdoor conditions are rapidly degraded due to the combined action of weather factors, such as oxygen, ultraviolet light and relative humidity (RH). Several paints and varnishes are commonly used as coating materials to prevent the degradation. However, wood coated with common clear coatings is still susceptible to photo discoloration (Lung Chou *et al.*, 2008; Bulian *et al.*, 2017). The photo discoloration is unavoidable even if wood is coated with non-yellowing or durable clear coatings, such as aliphatic polyurethane ones (Chang and Chou, 1999). The coating performance depends on several factors, in particular the substrate, the coating system and the interactions between them (Bulian and Graystone, 2009). Nowadays, coating is an area of significant research in nanotechnology. Nanoparticle based coating systems can provide better weathering resistance (Clausen *et al.*, 2010; Nguyen-Tri *et al.*, 2018) and conservation than the conventional techniques because of very small pigment particles with very high specific surface. Some appropriate nanoparticles, such as silica (SiO₂), alumina (Al₂O₃) (Bussey *et al.*, 2018; Mori *et al.*, 1998; Powell *et al.*, 1997) and TiO₂ (Nanetti, 2006; Li *et al.*, 2005) have been previously used to improve the wood weathering resistance. For example, Veronovski *et al.* (2013) found that the surface treatment of wood with nano-TiO₂ (rutile) incorporated in water-based acrylic coatings improves the weathering resistance. Organosilane nanomaterials have been mainly developed for waterproofing of wood because they can block the pores by agglomeration and prevent the penetration of water into wood (Tshabalala and Gangstad, 2003; Godnjavec *et al.*, 2012). For concrete, Nano-zycosil with size of up to 6 nm can enhance the waterproofing property of the surfaces by filling the microcracks and nanopores (Taghiyari, 2013). Gholamiyan *et al.*, (2012, 2016) also reported that the organosilane nanomaterials (zycosil and zycofil) can be used as

water vapor diffusion retarders for wood. The present study was, therefore, conducted to evaluate if organosilane nanoparticles can significantly contribute to improvement of weathering resistance of wood.

2 MATERIALS AND METHODS

2. MATERIJALI I METODE

2.1 Wood sampling and coating materials

2.1. Uzorkovanje drva i premazni materijali

Flat-sawn boards of poplar wood (*Populus nigra*) with dimensions of 50 by 100 by 200 mm (T, R and L directions, respectively) at moisture content of 12 % were selected for the study. Sealer and nitrocellulose lacquer and polyester lacquer were used as clear coatings. They were purchased from Dorsa Chemistry (Brilliant) Co. The technical properties of the lacquers are summarized in Table 1. Organosilane water soluble nanomaterials (nano-zycosil and nano-zycofil) were purchased from Zydex Company. The most important properties of the nanomaterials are presented in Table 2.

2.2 Coating methods

2.2. Metode nanošenja premaznog materijala

The wood surfaces were sanded with 150-grade sandpaper before coating. Six different coating systems were applied as shown in Table 3. A 250 μm wet pre-cure coating layer was applied by an automatic film applicator at the speed of 150 mm/s. The nanomaterial-coated samples were oven dried at temperature of 103 ± 2 °C for 24 h, and the others were dried in a conditioned room (T = 25 °C and RH=65 %) for about 20 minutes. The lacquers were diluted by a lacquer thinner (1:2) before application.

2.3 Weathering test

2.3. Izlaganje vremenskim utjecajima

The coated specimens were exposed to a 1000 W xenon arc light source at 65 % RH and chamber temperature of 30 °C in a weather-o-meter for 1440 hrs.

Table 1 Technical properties of nitrocellulose lacquer and polyester lacquer

Tablica 1. Svojstva nitroceluloznoga i poliesterskoga premaznog materijala

Coating type / Vrsta premaznog materijala	Viscosity at 25 °C / Viskoznost pri 25 °C	Percent solids / Udio suhe tvari	Density at 25 °C / Gustoća pri 25 °C	Color (Gardner) / Boja (Gardner)	Flash point, °C / Točka zapaljenja, °C
Nitrocellulose lacquer / nitrocelulozni premazni materijal	16-38 Pa·s	21±1	0.94±0.01	3 max	≤20
Polyester lacquer / poliesterski premazni materijal	10-25 Pa·s	30±1	0.94±0.01	3 max	≤20

Table 2 The most important information on used nanomaterials

Tablica 2. Najvažniji podatci o primijenjenim nanomaterijalima

Property / Svojstvo	Zycosil	Zycofil
Size / veličina	10-30 nm	90-300 nm
Color / boja	Pale yellow / blijedožuta	Yellow / žuta
Density / gustoća	1.7 (g/cm ³) (25 °C)	2.5 (g/cm ³) (25 °C)
Flash point / točka zapaljenja	More than / iznad 100 °C	More than / iznad 100 °C
Auto-ignition temperature / temperatura samozapaljenja	More than / iznad 200 °C	More than / iznad 150 °C
Viscosity / viskoznost	0.5-1 Pa·s (25 °C)	0.2-0.7 Pa·s (25 °C)

Table 3 Guide for coating systems

Tablica 3. Oznake sustava premaznih materijala

Coating type / Vrsta premaznog materijala	Coating materials Premazni materijal
Control	None
CZ	nano-zycosil
CZF	nano-zycofil
CZZF	nano-zycosil+ nano-zycofil
CPS	Clear polyester lacquer
CSC	sealer + nitrocellulose lacquer
CSC+CPS	sealer + nitrocellulose lacquer + polyester lacquer

2.4 Color measurement

2.4. Mjerenje boje

Spectrophotometry was applied to measure brightness (L^*), redness (b^*) and yellowness (a^*) variables of specimens in the CIE-LAB system before and after exposure to the accelerated weathering. Ten boards were tested for each type of coating system. The spectrophotometry was performed using a miniscan EZ spectrophotometer with the aperture diameter of 25 mm and with a standard illuminant D65 and a 10° standard observer. The parameters of ΔE^* (color difference), C^* (color saturation) and h^* were calculated using the following equations:

$$C^* = \sqrt{(a^*)^2 + (b^*)^2} \quad (1)$$

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

$$h^* = \text{Arctan}(b^*/a^*) \quad (3)$$

2.5 Measuring the surface roughness

2.5. Mjerenje hrapavosti površine

A Mitutoyo SJ-201P instrument was employed for measuring the surface roughness. Three roughness parameters characterized by ISO 4287 standard (1997), namely average roughness (R_a), mean peak-to-valley height (R_z), and maximum peak-to-valley height (R_y) were considered to evaluate the surface characteristics of coatings before and after weathering.

2.6 Determination of wettability

2.6. Ispitivanje kvašenja

The wetting behavior of coated samples conditioned at 65 % RH and 20 °C was characterized by static contact angle (CA) measurement and deionized water as test liquid. The CA values were determined by sessile drop method using a KSV Cam-101 Scientific Instrument (Helsinki, Gottingen University, Germany). The measurements were carried out for 1 and 10 seconds after deposition of the water drop on the surface. The average CA value was obtained with five drops for each sample.

2.7. SEM, EDX and ATR-FTIR studies

2.7. SEM, EDX i ATR-FTIR istraživanje

The surface morphology of coated samples was characterized by a scanning electron microscopy (SEM). The surface chemical composition of speci-

mens was examined by energy dispersive X-ray analysis (EDX). Fourier transform infrared spectrophotometer (ATR-FTIR; Bruker model Confocheck) was also applied to identify the formed chemical bonds.

3 RESULTS AND DISCUSSION

3. REZULTATI I RASPRAVA

3.1 Color changes

3.1. Promjene boje

Table 4 shows L^* , a^* and b^* values and their changes on various coated wood samples due to weathering. Before weathering, L^* for uncoated specimens was greater than that for all coated ones. The color parameters (L^* , a^* and b^*) of nano-zycofil (CZF)- and nano-zycosil (CZ)-coated specimens were found to be different. The color change of weathering for CZ was less than that for CZF. One of the main reasons for color change of clear coatings is the discoloration of wood substrate, affected by its individual chemical components, such as extractives (Fengel and Wegener, 1984). After coating, b^* and a^* decreased for all coatings, except CZ. Sigh *et al.* (2008) and Lung Chou *et al.* (2008) also reported a similar result for yellowing of clear coatings. After weathering, the least change in lightness index was observed for CZ and CZZF coatings. The lightness of CZ and CSC+CPS coatings dropped from 75.6 to 74.3 and from 78.1 to 67.5, respectively. After weathering, all coatings except CZ and CZZF were darker than the control sample as shown by the decreased L^* values (see Table 4). This L^* reduction is due to destruction of chemical bonds and photochemical degradation of cross-linking reactions at the surface coatings (Vlad Cristea *et al.*, 2011). After weathering, all coatings are more reddish and yellowish (indicated by increased b^* and a^* values, respectively, in Table 4) than the control sample (Tsuchikawa *et al.*, 2003 and Pastore *et al.*, 2004). The least change in a^* and b^* parameters was observed for CZZF. After coating, CZ showed the least color change (ΔE). According to the measured color data, it can be noted that all coatings are more red, yellow and saturated (c^*) than the uncoated wood both before and after weathering (Table 5). The color saturation after weathering ranged from 48.7 for sealer and nitrocellulose lacquer-coating to 35.6 for uncoated wood. Similar results were also found for hue angle (h^*).

3.2 Surface roughness

3.2. Hrapavost površine

The surface roughness values (R_a , R_z and R_q) for each coating are depicted in Figure 1. After coating, the CSC+CPS and polyester (CPS) coatings were smoother than the other ones, probably due to rigid film formation on the wood surface (Gholamiyan *et al.*, 2012). After weathering, the surface roughness increased for all coatings expect CZ. Increase in the surface roughness of wood after weathering is believed to be due to the erosion of primary cell wall caused by lignin degradation (Bulcke *et al.*, 2007; Meincken and Evans, 2009). It is claimed that the surface roughness of nano-

Table 4 Guide for coating systems**Tablica 4.** Oznake sustava premaznih materijala

Coating type / Vrsta premaznog materijala		Control	CZ	CZF	CZZF	CPS	CSC	CSC+CPS
<i>L*</i>	Before / prije	80.5 (1.5)	75.6 (1.8)	79 (1.3)	77.6 (2.2)	76.1 (3.9)	74.7 (2.5)	78.1 (1.0)
	After / nakon	73.2 (2.3)	74.2 (1.6)	70.7 (2.8)	73.3 (0.6)	69.3 (2.1)	65.4 (1.2)	67.5 (0.4)
	Mean changes / prosječna promjena	-7.2	-1.3	-8.2	-4.3	-6.7	-9.2	-10.5
<i>a*</i>	Before / prije	3.5 (0.3)	2.8 (0.8)	3.8 (0.9)	4 (0.7)	4.8 (0.5)	5.1 (0.8)	3.6 (0.6)
	After / nakon	9.5 (0.1)	9.8 (0.5)	11.4 (0.4)	8.7 (0.7)	11.7 (0.6)	13.8 (0.6)	13.1 (0.5)
	Mean changes / prosječna promjena	5.9	7	7.6	4.7	6.9	8.6	9.4
<i>b*</i>	Before / prije	19.1 (0.3)	20.8 (0.6)	20.4 (1.8)	22.5 (0.2)	25.6 (1.3)	25.7 (1.6)	23.2 (2.1)
	After / nakon	34.2 (0.7)	38.8 (2.6)	38.4 (0.3)	36.6 (0.8)	42.1 (2.6)	46.7 (1)	44.9 (1.0)
	Mean changes / prosječna promjena	15.1	17.9	17.9	14	16.42	20.9	21.7

Values in parentheses represent the standard deviation / Vrijednosti u zagradama standardne su devijacije.

Table 5 Changes of ΔE , c^* and h^* before and after weathering**Tablica 5.** Promjene ΔE , c^* and h^* prije i nakon izlaganja vremenskim utjecajima

Coating type / Vrsta premaznog materijala		Control	CZ	CZF	CZZF	CPS	CSC	CSC+CPS
ΔE^*	Before / prije	...	2.1 (0.6)	2.2 (0.2)	4.5 (0.4)	8 (0.5)	9 (0.1)	4.7 (0.1)
	After / nakon	...	4.6 (0.5)	5.2 (0.8)	2.4 (0.8)	9 (0.7)	15.3 (0.9)	12.5 (0.8)
	Mean changes / prosječna promjena		2.4	3.1	-2	0.9	6.2	7.8
c^*	Before / prije	19.4 (1.5)	21 (1.6)	20.8 (2.5)	22.9 (1.6)	26.1 (2)	26.3 (1.8)	23.5 (1.6)
	After / nakon	35.5 (2.1)	40.0 (2.9)	40.1 (3.2)	37.6 (2.1)	43.7 (2.5)	48.7 (3)	46.8 (3.6)
	Mean changes / prosječna promjena	16.1	18.9	19.2	14.7	17.5	22.4	23.2
h^*	Before / prije	1.3 (0.3)	1.4 (0.1)	1.3 (0.1)	1.3 (0.2)	1.3 (0.2)	1. (0.13)	1.4 (0.2)
	After / nakon	1.2 (0.1)	1.3 (0.1)	1.2 (0.2)	1.3 (0.1)	1.2 (0.1)	1.2 (0.2)	1.2 (0.1)
	Mean changes / prosječna promjena	-0.09	-0.1	-0.1	-0.06	-0.09	-0.09	-0.1

Values in parentheses represent the standard deviation / Vrijednosti u zagradama standardne su devijacije.

coatings is not significantly affected by weathering because of small size and high surface to volume ratio of nanoparticles (Li *et al.*, 2001). Thus, the smaller size of nano-zycosil particles (10-30 nm) compared to nano-zycofil (90-300 nm) ones may play an important role in the surface roughness.

3.3 Wettability

3.3. Kvašenje

The results showed that the wettability of wood decreased due to coating by all used coating systems (Figure 2), and the contact angle was increased significantly with the applied coatings. However, weathering had a decreasing effect on the contact angle for all coatings. The contact angle on the control sample dropped from 43° to 38° after 1440 h weathering and that of CZ from 87° to 67°. Among coatings, CZ and CSC+CPS exhibited the highest contact angle after the weathering exposure. In agreement with our results, waterproofing of organosilane nanomaterials has been reported, previously (Tshabalala and Gangstad, 2003; Godnjavec *et al.*, 2012).

3.4 SEM and EDX analysis

3.4. SEM i EDX analiza

Scanning electron microscopy (SEM) images of control and nano-zycosil coated films are shown in Figure 3. As can be seen in Figure 4, most particles are in nanometric dimensions (<100 nm). SEM images confirm that silane nanoparticles can effectively cover the wood surface after coating, which is similar to the results reported by a number of previous researchers (Li *et al.*, 2010; Da Silva *et al.*, 2012; Mahltig *et al.*, 2010). A significant silane peak in the EDX spectra of organosilane coated specimens confirms the presence of nano-zycosil or nano-zycofil particles (Figure 5).

3.5 Surface chemistry

3.5. Kemijska svojstva površine

The ATR-FTIR spectra of nano-zycosil coated and uncoated specimens are compared in Figure 6. The absorption bands at 1110-1090 cm^{-1} , 810-800 cm^{-1} and 480-470 cm^{-1} are attributed to the Si-O-Si asymmetric stretching, symmetric stretching and bending vibration, respectively. The spectrum of coated specimen

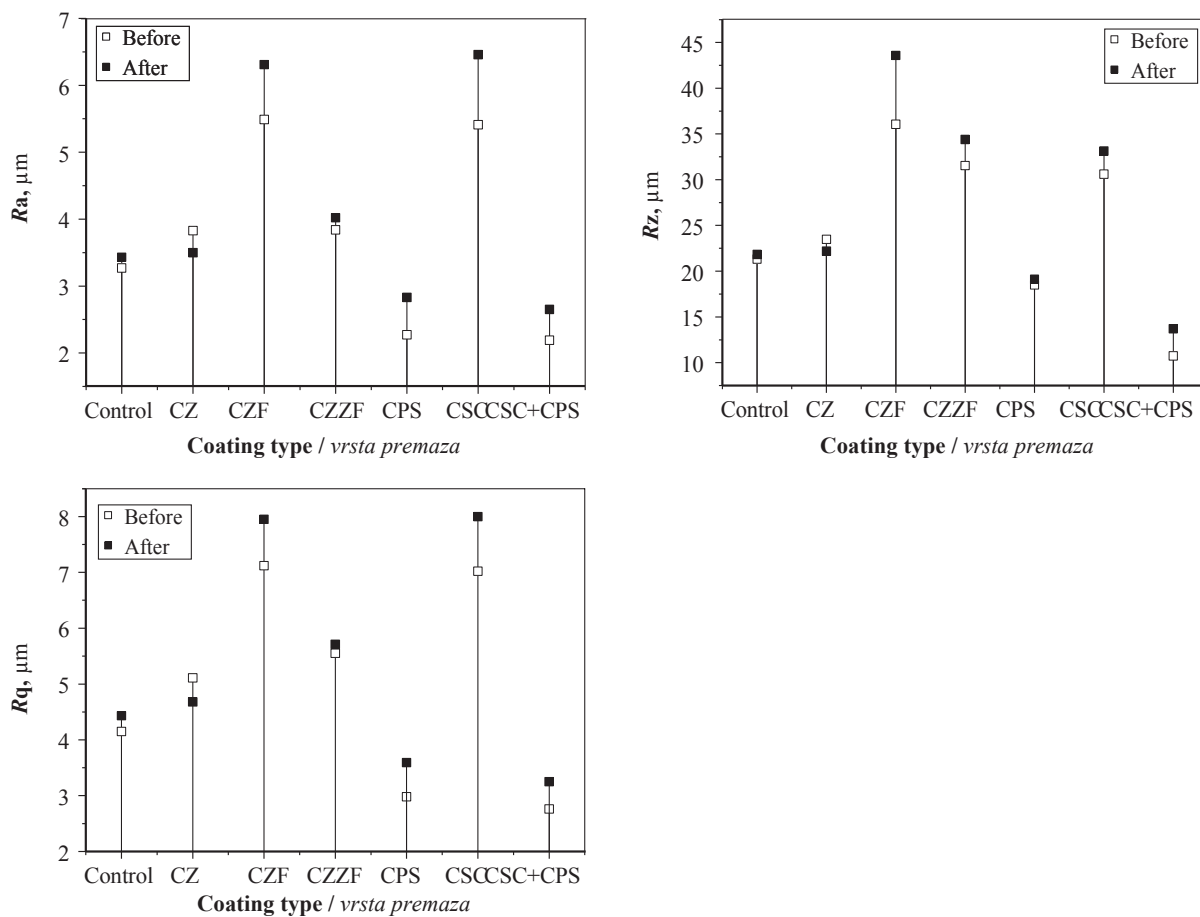


Figure 1 Surface roughness parameters for coated and uncoated specimens before and after weathering
Slika 1. Parametri hrapavosti površine premazanih i nepremazanih uzoraka prije i nakon izlaganja vremenskim utjecajima

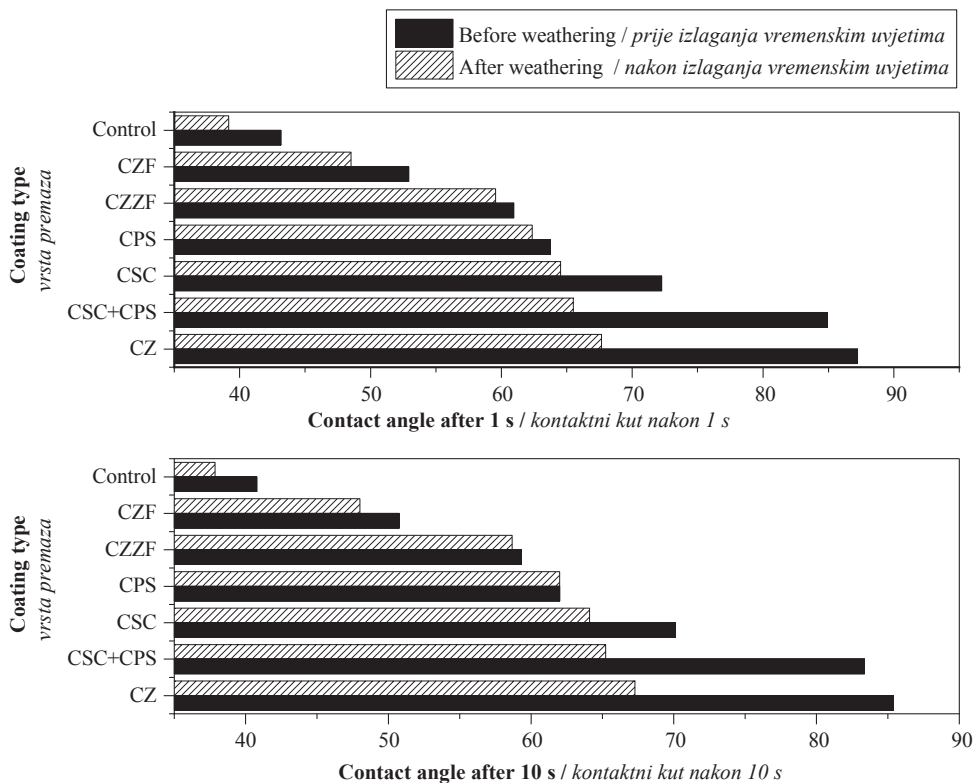


Figure 2 Contact angles for coated and uncoated specimens before and after weathering
Slika 2. Kontaktni kut premazanih i nepremazanih uzoraka prije i nakon izlaganja vremenskim utjecajima

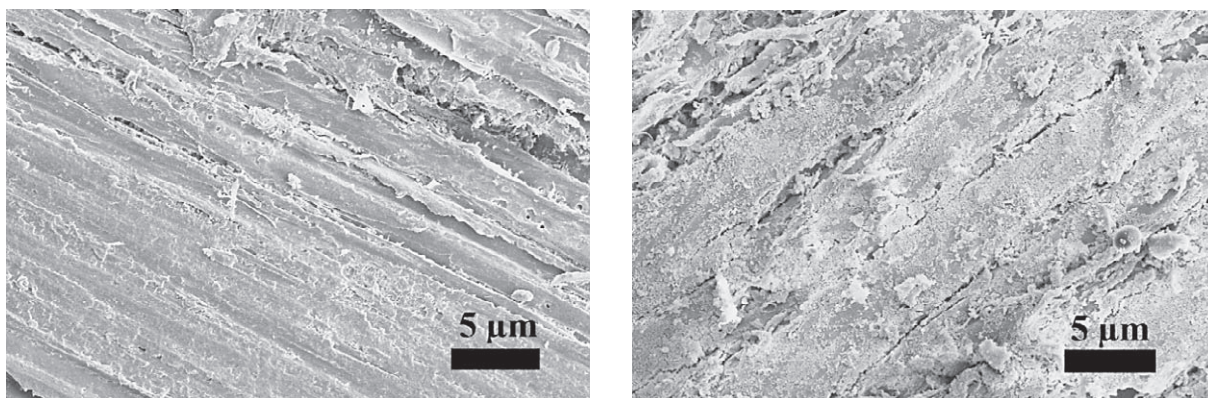


Figure 3 SEM image of control (left) and nano-zycosil (right) coating
Slika 3. SEM fotografija kontrolnog premaza (lijevo) i premaza *nano-zycosilom* (desno)

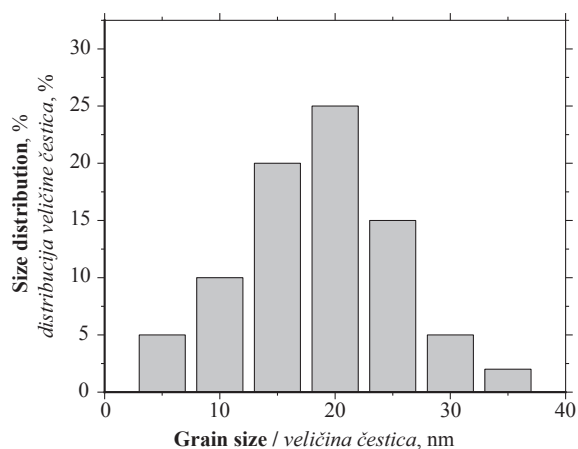


Figure 4 Particles size distribution of nano-zycosil
Slika 4. Raspodjela veličine čestica *nano-zycosila*

shows bands resulting from Si-CH₃ bonds (1269 cm⁻¹) (Tshabalala and Gangstad, 2003). The bands at broad absorption band of 3445-3425 cm⁻¹ and 1640-1630 cm⁻¹ are due to -OH groups of silane coatings (Rao *et al.*, 2010). In addition, the weak bands centered around 2880 cm⁻¹ due to the C-H stretch and 1460 cm⁻¹ due to the C-H bending vibrations of the wood components were replaced by sharper and much stronger bands at 2921 cm⁻¹ due to the C-H bending vibrations of the long hydrocarbon chain bound to the wood by the nano-zycosil coating (Tshabalala and Gangstad, 2003 and Wang *et al.*, 2011).

4 CONCLUSIONS 4. ZAKLJUČAK

SEM images and EDX spectra showed that the wood surfaces can be effectively coated by the organosilane water soluble nanomaterials. SEM images revealed a significant surface coverage by the nanoparticles, and the EDX analysis also confirmed the presence of silane in the organosilane coated specimens. Among coating materials, nano-zycosil (CZ) exhibited the best performance for improving the weathering resistance of poplar wood. Different color parameters were observed between nano-zycosil and nano-zycofil coat-

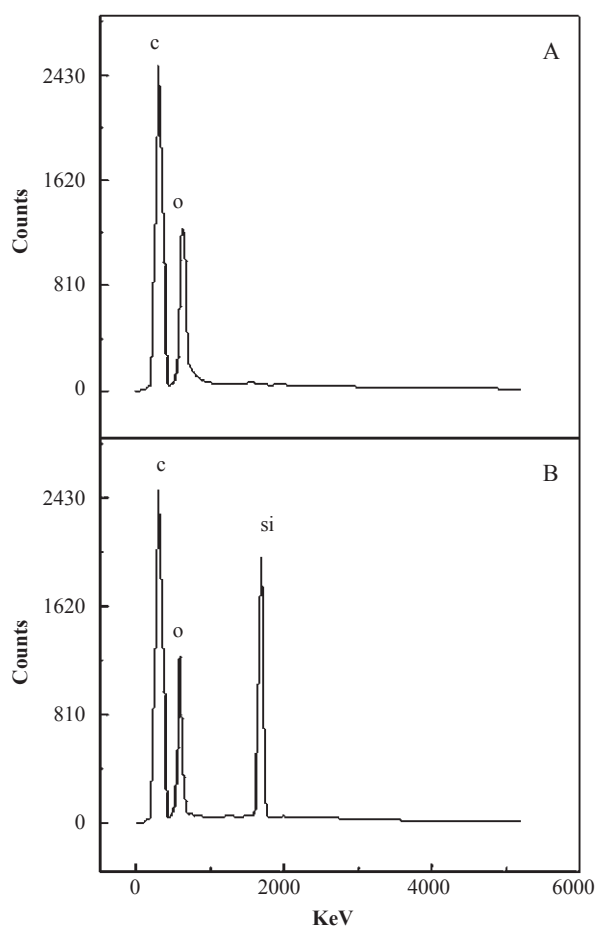


Figure 5 EDX spectra of (A) control and (B) nano-zycosil coated surfaces
Slika 5. EDX spektar (A) kontrolne površine i (B) površine premazane *nano-zycosilom*

ings. After weathering, all coatings except CZ and CZZF were darker than the uncoated wood. Compared to the clear coatings, the roughness of nano-zycosil and nano-zycofil was less affected by weathering, and the former showed a better performance. This can be due to the very small size and high surface to volume ratio of nanoparticles. Further researches on the nanofilm formation using sol-gel method are recommended for improving the weathering resistance of wood surfaces.

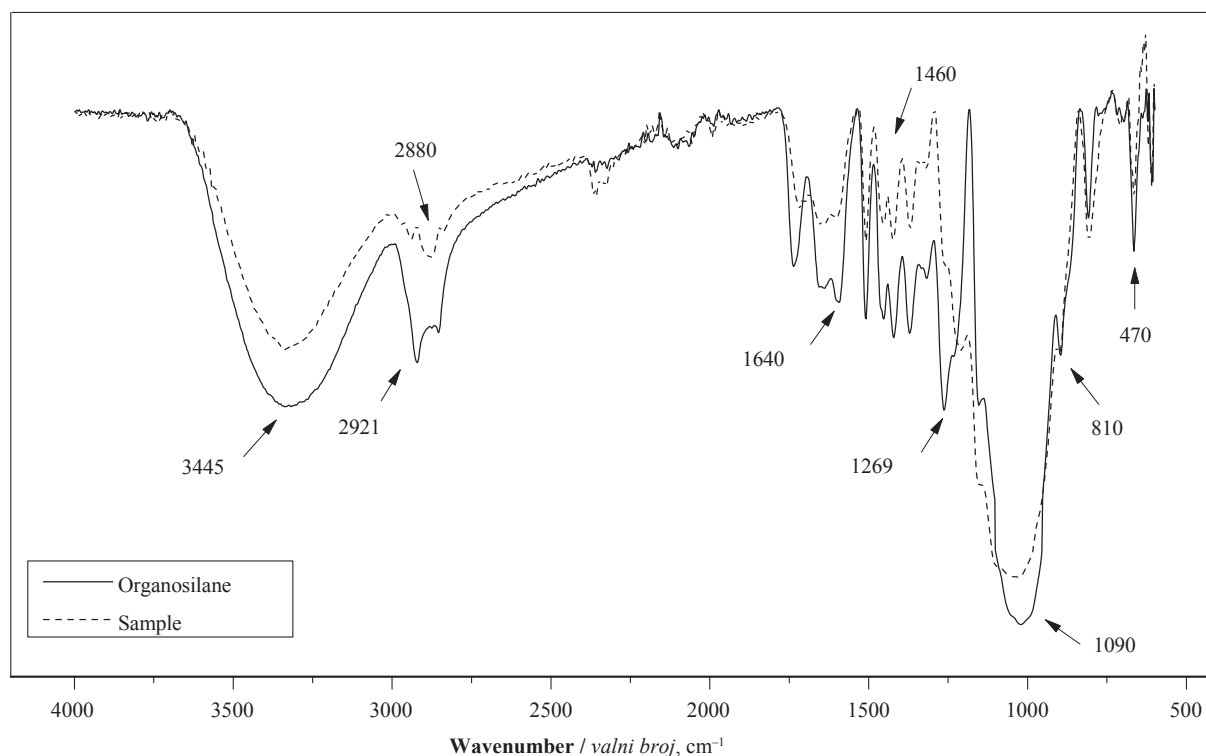


Figure 6 ATR-FTIR spectra for control and organosilane coated surfaces
Slika 6. ATR-FTIR spektar kontrolne površine i površine obrađene organosilanom

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