

# Sadržaj

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# Accelerated Weathering and Decay Resistance of Heat-Treated Wood Reinforced Polypropylene Composites

Ubrzano izlaganje polipropilenskih kompozita ojačanih pregrijanim drvom vremenskim utjecajima i otpornost na propadanje

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**ABSTRACT** • The aim of this study was to determine the accelerated weathering and decay resistance of the heat-treated wood reinforced polypropylene composites (HT-WPC). Polypropylene (PP) was used as a matrix and the heat-treated wood treated at 180 °C and 220 °C as reinforcement filler. The effect of three filler type, such as 40, 60 and 100 mesh, on the outdoor performance of composites was also investigated. The composites were prepared with twin screw extruder, and the test samples were obtained with compression molding. Lightness index ( $L^*$ ), color changes ( $\Delta E^*$ ) and physical changes on the surface of the composites after the accelerated weathering, and decay resistance of the composites were investigated. According to the results, the effects of heat-treated wood on color changes were found to be more than its filler size, and while the filler loadings were increased from 5 % to 20 %, it was determined to increase the color changes of the composites. In scanning electron microscopy (SEM) images, crack formation and deterioration on the surface of the composites were determined. In FTIR spectra, no difference was determined between the composites, and all peaks were similar to each one. The addition of heat-treated wood improved the antifungal efficiency of the composite, and the mass losses decreased with the increasing of heat treatment temperature. As a result, adding heat-treated wood to PP was found to improve the outdoor performance of the HT-WPCs.

**Keywords:** wood polymer composites, heat-treated wood, polypropylene (PP), outdoor performance, thermoplastics

**SAŽETAK** • Cilj ovog rada bio je utvrditi otpornost na propadanje polipropilenskih kompozita ojačanih pregrijanim drvom (HT-WPC) pri ubrzanom izlaganju vremenskim utjecajima. Polipropilen (PP) upotrijebljen je kao matrica, a drvo pregrijano na 180 i 220 °C kao armaturno punilo. Ispitivan je i učinak triju vrsta punila (mreža 40, 60 i 100) na svojstva kompozita u eksterijeru. Kompoziti su pripremljeni dvostrukim vijčanim ekstruderom,

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a ispitni su uzorci dobiveni kompresijskim lijevanjem. Ispitivana je svjetlina ( $L^*$ ), promjena boje ( $\Delta E^*$ ) i fizičke promjene na površini kompozita nakon ubrzanog izlaganja vremenskim utjecajima, kao i otpornost na propadanje kompozita. Prema dobivenim rezultatima, utvrđeno je da su učinci pregrijavanja drva na promjenu boje veći od učinka veličine punila. Nadalje, zamijećeno je povećanje promjene boje kompozita s povećanjem udjela punila od 5 na 20 %. Na snimkama dobivenim elektronskim mikroskopom (SEM) uočeno je stvaranje pukotina i propadanje površine kompozita. Međutim, na FTIR spektru nije ustanovljena razlika između kompozita i svi su vrhovi bili slični. Dodavanjem pregrijanog drva poboljšala se otpornost kompozita na gljive te se smanjio gubitak mase s povećanjem temperature pregrijavanja. Utvrđeno je da se dodavanjem pregrijanog drva u polipropilen (PP) poboljšavaju svojstva kompozita HT-WPC-a u eksterijeru.

**Ključne riječi:** drvno-plastični kompoziti, pregrijano drvo, polipropilen (PP), svojstva u eksterijeru, termoplasti

## 1 INTRODUCTION

### 1. UVOD

Wood polymer composites (WPCs) have been of interest due to major advantages such as cost, durability, high mechanical properties, etc. WPC has two main constituents: wood and polyolefins. The addition of wood can provide an increase in the mechanical properties of the WPC as compared with neat polymer (Kallakas *et al.*, 2015). However, wood is a biological material, which is degraded by biological degradation factors such as fungi, insect, bacteria, etc. It also has a hygroscopic structure, and it can absorb a lot of water in a wet medium or desorb by evaporating the water outside the cell in a dry medium. The behavior turns the wood into, dimensionally, an unstable structure. Therefore, in many outdoor applications, the durability and dimensional stability of wood is an important issue for the WPC performance. Biological durability of wood is known as a significant drawback of polymer composites reinforced with wood in outdoor conditions (Luo *et al.*, 2012). Pilarski and Matuana (2005) observed that the composites absorbed more moisture in outdoor uses, and some deterioration of the interfacial adhesion between the wood flour and the polymer matrix was seen. Naumann *et al.* (2012) reported the formation of micro- and macro-cracks in the surface of the wood polymer composite due to outdoor use. The results showed that the cracks cause the enhancement of moisture sorption and microbial attacks due to the weathering under UV radiation, water spray. Many studies in the literature have reported that the deteriorations occurred on the surface of the composites due to higher moisture content after the natural and artificial weathering (Kallakas *et al.*, 2015; Stark and Gardner, 2008; Butylina and Karki, 2014; Yilgor *et al.*, 2014; Lopez-Naranjo *et al.*, 2016). To eliminate these drawbacks, many studies related to the heat treatment in terms of different properties, such as the physical and mechanical properties of wood materials, were conducted (Aytekin *et al.*, 2009; Gunduz *et al.*, 2009; Kaygin *et al.*, 2009; Kaygin *et al.*, 2014). The heat treatment also improves the water sorption and fungal resistance of wood (Gunduz *et al.*, 2009; Gunduz *et al.*, 2010; Žlahtič-Zupanc *et al.*, 2018). When wood is thermally modified at a temperature of 180 °C and above, its thermal properties can also be improved because some components of wood are decomposed or at least have gone through various changes in its structure after the heat-treatment (Segerholm *et al.*, 2012; Kaboorani,

2009; Pelaez-Samaniego *et al.*, 2013; Karakus *et al.*, 2017).

The objective of this paper was to investigate the accelerated weathering performance and decay resistance of the heat-treated wood reinforced polypropylene composites in various filler sizes and loadings and to determine the feasibility of heat-treated wood in polymer composites for outdoor applications as an alternative to untreated wood.

## 2 MATERIALS AND METHODS

### 2. MATERIJALI I METODE

#### 2.1 Materials

##### 2.1. Materijali

Polypropylene matrix used in the study was obtained by Petkim Inc. (Turkey, Izmir). It has a density of 0.90 g/cm<sup>3</sup>, melt flow index of 24 g/min and melting temperature ( $T_m$ ) of 180 °C. Beech (*Fagus orientalis* L.) wood used as filler was kindly supplied by the local timber mill. (Bartın, Turkey). The heat-treated wood was prepared at 180 °C (HT1) and 220 °C (HT2) for 4 hours in a heating cabin with  $\pm 1$  °C sensitivity. Both the heat-treated and untreated wood were ground to small diameters of 40, 60 and 100 mesh to determine the effects of filler size and amount on the outdoor performance of the composites.

#### 2.2 Preparation of composites

##### 2.2. Priprema kompozita

Polypropylene was separately mixed with both the untreated (UT) and heat-treated wood (HT). The compounding was provided with a twin-screw extruder (Gulnar Extruder, Turkey) at a temperature of 180 °C and rotor speed of 65 min<sup>-1</sup>. The polymer-wood blends were granulated after water bath, they were dried at (105 $\pm$ 2) °C for 24 h, and all blends were compression molded at the processing temperature of 180 °C and the injection pressure of 25 kg/cm<sup>2</sup>. The formulations of the composites are shown in Table 1.

#### 2.3 Characterization of composites

##### 2.3. Karakterizacija kompozita

#### 2.3.1 Accelerated weathering performance (QUV)

##### 2.3.1. Ubrzano izlaganje vremenskim utjecajima (QUV)

The accelerated weathering tests were performed by cycles of UV-light irradiation during 500 hours in a QUV test cycle chamber according to ASTM G154.

**Table 1** Formulations of composites

**Tablica 1.** Formulacije kompozita

| Samples<br><i>Uzorci</i> | Filler type<br><i>Vrsta punila</i> | Filler size, mesh<br><i>Veličina punila, mesh</i> | Filler loadings, %<br><i>Udio punila, %</i> | Polymer, %<br><i>Polimer, %</i> |
|--------------------------|------------------------------------|---|---|---------------------------------|
| PP                       |                                    |   |   | 100                             |
| UT40_5                   | Untreated (UT)                     | 40  | 5   | 95                              |
| HT1-40_5                 | Treated at 180°C (HT1)             |   |   |                                 |
| HT2-40_5                 | Treated at 220°C (HT2)             |   |   |                                 |
| UT40_20                  | Untreated (UT)                     | 40  | 20  | 80                              |
| HT1-40_20                | Treated at 180°C (HT1)             |   |   |                                 |
| HT2-40_20                | Treated at 220°C (HT2)             |   |   |                                 |
| UT60_5                   | Untreated (UT)                     | 60  | 5   | 95                              |
| HT1-60_5                 | Treated at 180°C (HT1)             |   |   |                                 |
| HT2-60_5                 | Treated at 220°C (HT2)             |   |   |                                 |
| UT60_20                  | Untreated (UT)                     | 60  | 20  | 80                              |
| HT1-60_20                | Treated at 180°C (HT1)             |   |   |                                 |
| HT2-60_20                | Treated at 220°C (HT2)             |   |   |                                 |
| UT100_5                  | Untreated (UT)                     | 100   | 5   | 95                              |
| HT1-100_5                | Treated at 180°C (HT1)             |   |   |                                 |
| HT2-100_5                | Treated at 220°C (HT2)             |   |   |                                 |
| UT100_20                 | Untreated (UT)                     | 100   | 20  | 80                              |
| HT1-100_20               | Treated at 180°C (HT1)             |   |   |                                 |
| HT2-100_20               | Treated at 220°C (HT2)             |   |   |                                 |

The average irradiance level was 0.85 W/m<sup>2</sup> at 340 nm and the temperature in the chamber was approximately 50 °C. Three replicates of each composite were used. After the QUV test, color changes were determined and surface characterization with SEM of all the composites was conducted.

### 2.3.2 Color measurement

#### 2.3.2. Mjerenje boje

Color changes were determined with a Konica Minolta spectrophotometer (Osaka, Japan) by measuring the *L*, *a* and *b* values on the specimens. For each specimen, four-color measurement was made and the color analysis was carried out according to ISO 7724-2 standard. The changes in color coordinates ( $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$ ) were determined with the difference between the initial and final values. The total color changes ( $\Delta E^*$ ) were calculated according to the following equations:

$$(\Delta E^*) = [(\Delta a^*)^2 + (\Delta b^*)^2 + (\Delta L^*)^2]^{1/2} \quad (1)$$

### 2.3.3 Determination of antifungal efficiency

#### 2.3.3. Određivanje otpornosti na gljive

Decay test was carried out on the composites during 12 weeks in a climate chamber with a temperature of 23 °C and relative humidity of 70 % according to the principles of EN 113. Test specimens were prepared in dimensions of 30 mm × 12 mm × 2 mm. White rot fungi were used in the test because hardwood was used for the production of WPCs. The decay test for white rot, *T. versicolor* (*L.: Fr.) Pilat* was based on the specimens on 48 % malt extract agar in Petri dishes. At the end of the exposure time, the mycelia layers on the surfaces of the composites were removed and weighed. The mass loss was determined based on the dry mass before and after the decay test. Six replicates were used for each composite in the decay test.

### 2.3.4 Scanning Electron Microscopy (SEM)

#### 2.3.4. Skenirajuća elektronska mikroskopija (SEM)

The morphological analysis of the composites was conducted with environmental scanning electron microscopy (ESEM) (Tescan MAIA3 XMU-SEM), with an accelerating voltage of 5 kV. For enhanced conductivity, the surface of all samples was sputter-coated with a blend of gold-palladium using a Denton sputter coater.

### 2.3.5 Fourier Transfer Infrared Spectroscopy (FTIR)

#### 2.3.5. Fourierova transformacijska infracrvena spektroskopija (FTIR)

The FTIR-ATR analysis was carried out with a Shimadzu IRAAffinity-1 spectrometer equipped with a single reflection ATR pike MIRacle sampling accessory. Four accumulated spectra with a resolution of 4 cm<sup>-1</sup> were obtained for wavenumbers from 800 cm<sup>-1</sup> to 4000 cm<sup>-1</sup> with 32 scans for each sample.

## 3 RESULTS AND DISCUSSION

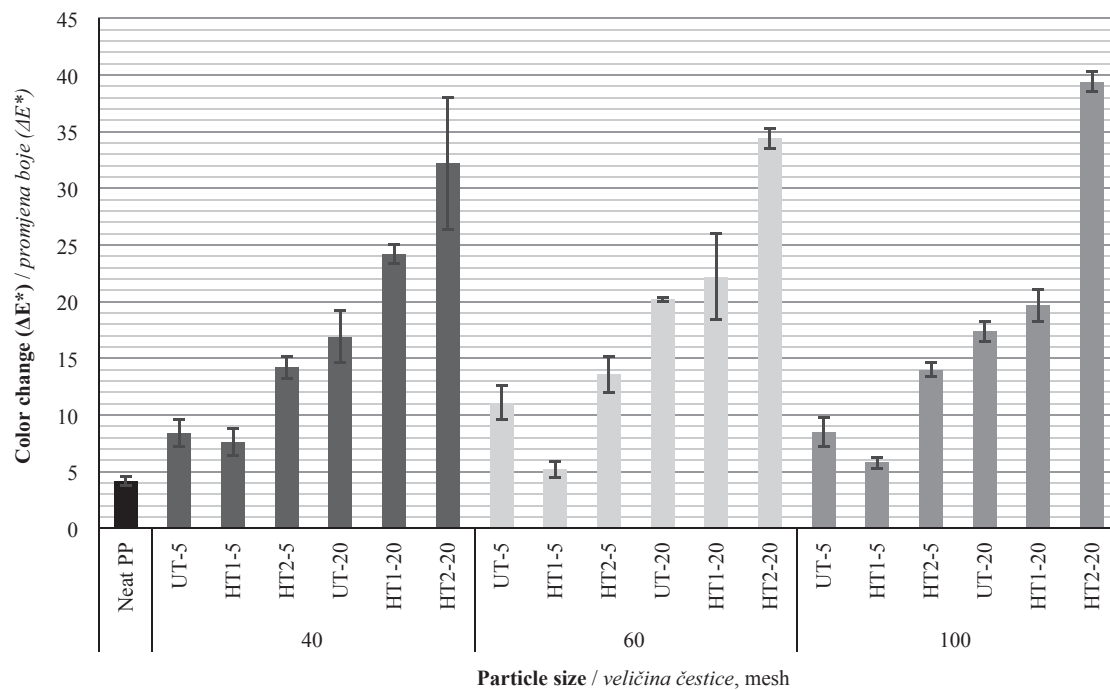
### 3. REZULTATI I RASPRAVA

#### 3.1 Accelerated weathering performance

##### 3.1. Ubrzano izlaganje vremenskim utjecajima

The color and physical changes on the surface of the composites after the accelerated weathering were examined. Figure 1 and Figure 2 show the color changes ( $\Delta E^*$ ) as a percentage of all the composites.

As shown in Figure 1 and 2, it was determined that the filler size of wood had no important effect on the color changes after QUV exposure. The maximum color changes were found for the composites with wood treated at 220 °C in all mesh sizes. It can be said that the status caused due to the heat-treated wood tends to grease rapidly in the weathering environment

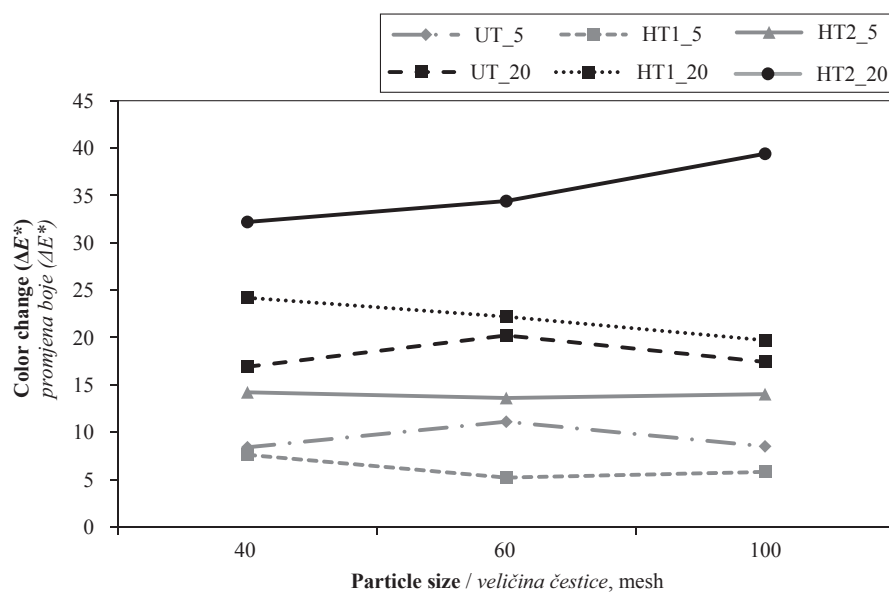


**Figure 1** Color changes ( $\Delta E^*$ ) of all composites  
**Slika 1.** Promjena boje ( $\Delta E^*$ ) svih kompozita

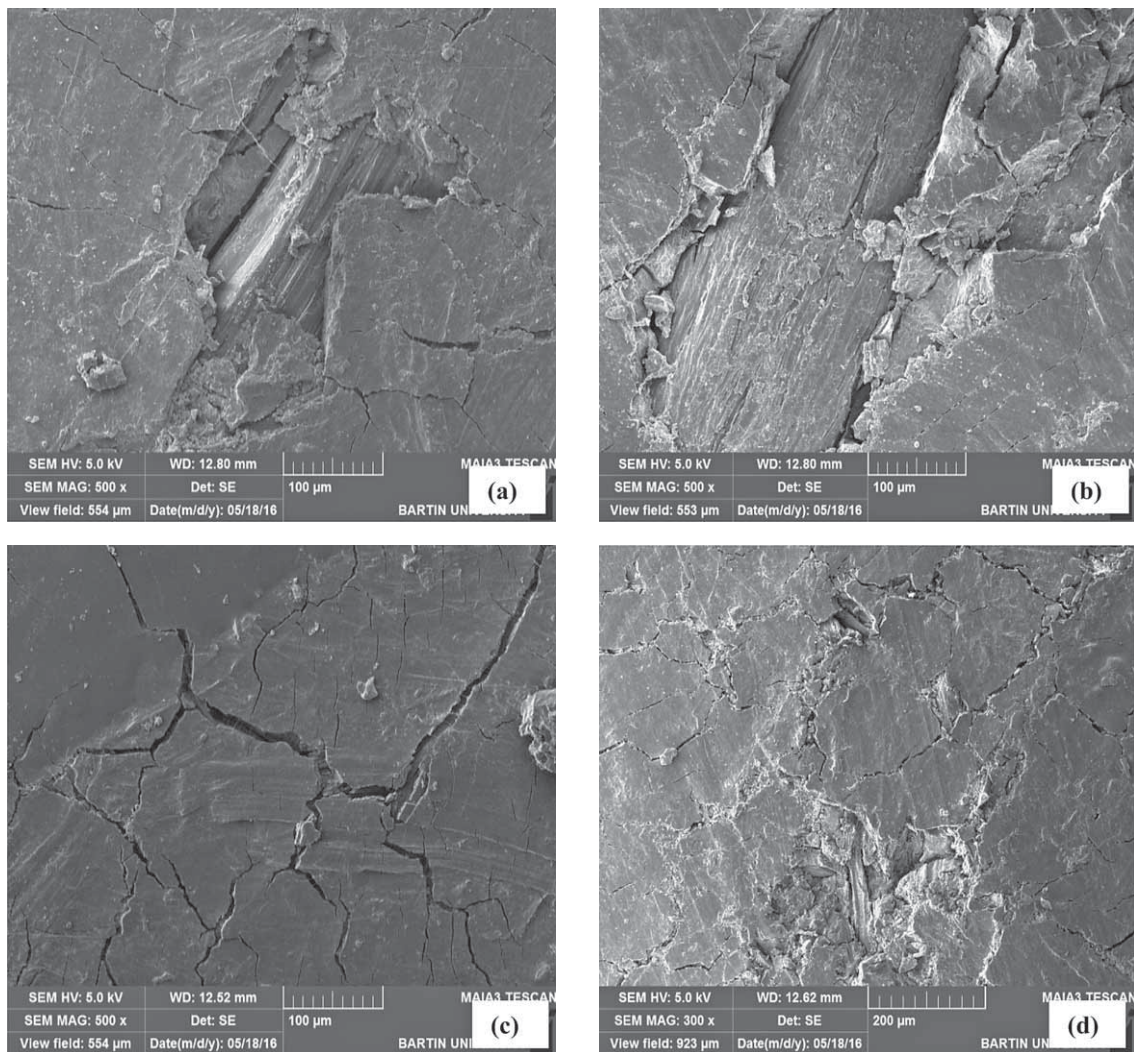
(Baysal *et al.*, 2014). The color changes in the composites with 20 % wood filler were found to be higher than the composites with 5 % wood filler. Hence, it can be said that the higher the addition of wood, the higher are the color changes. In a study, it was determined that the addition of the heat-treated wood to polymer composite had negative effects on the color change, and heat-treatment was found to be ineffective in improving UV resistance of wood (Xing *et al.*, 2015). After QUV test, whitening on the surface of the polypropylene composites with heat-treated wood was observed, and when heat-treatment temperature was raised from 180 °C to 220 °C, the whitening on the surface of polymer composites was found to increase. As shown by SEM im-

ages in Figure 3, various small cracks and deep slits were also observed on the surfaces of the composites with both untreated and heat-treated wood as given in Figure 3c and d, and the pieces of wood in the plastic matrix were observed to come to the surface of the composites (Figure 3a and b). Similar results were obtained after the accelerated or natural weathering in other studies (Turku and Karki, 2016; Selden *et al.*, 2004; Lee *et al.*, 2012).

Table 2 shows the decay resistance of the composites with untreated and heat-treated wood. The weight losses of the composites with both heat-treated and untreated wood were determined to be higher than those of the neat polypropylene. The heat-treated wood



**Figure 2** Color changes of composites according to filler size  
**Slika 2.** Promjena boje kompozita s obzirom na veličinu punila



**Figure 3** Physical changes on the surface of composites: (a, b) small pieces of wood, (c, d) surface deterioration and cracks  
**Slika 3.** Fizičke promjene na površini kompozita: (a, b) mali komadići drva, (c, d) propadanje površine i pukotine

**Table 2** Mass loss of neat polypropylene and composites exposed to decay test

**Tablica 2.** Gubitak mase čistog polipropilena i kompozita izloženih testu propadanja

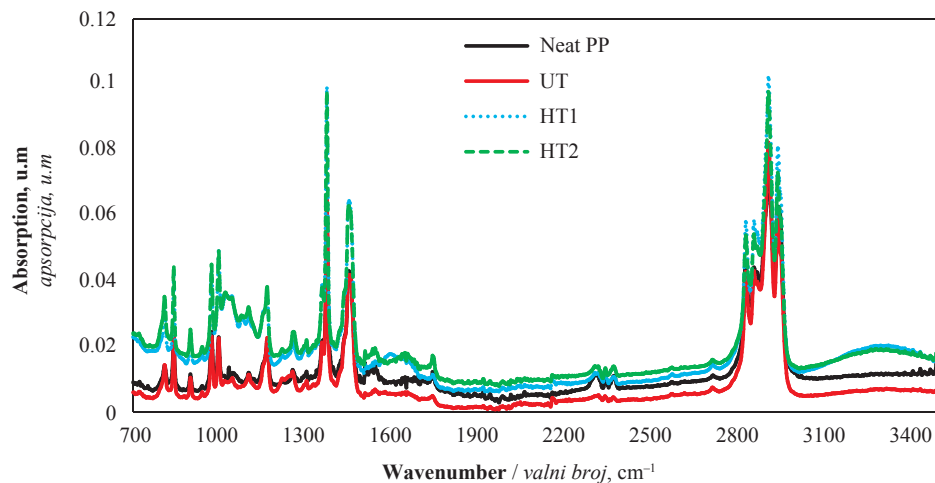
| Samples<br><i>Uzorci</i> | Filler loading, %<br><i>Udio punila, %</i> | Mesh size<br><i>Veličina</i> | Mass loss, %<br><i>Gubitak mase, %</i> |             |
|--------------------------|--|------------------------------|--|-------------|
| Neat PP                  |  |                              | 0.2 (±0.02)                            |             |
| UT                       | 5  | 40                           | 0.9 (±0.10)                            |             |
| HT1                      |  |                              | 0.9 (±0.14)                            |             |
| HT2                      |  |                              | 0.8 (±0.12)                            |             |
| UT                       |  |                              | 1.4 (±0.14)                            |             |
| HT1                      |  | 60                           | 1.0 (±0.12)                            |             |
| HT2                      |  |                              | 0.9 (±0.13)                            |             |
| UT                       |  |                              | 1.0 (±0.14)                            |             |
| HT1                      |  |                              | 0.9 (±0.15)                            |             |
| HT2                      |  | 0.9 (±0.14)                  |  |             |
| UT                       |  | 20                           | 40                                     | 1.2 (±0.15) |
| HT1                      |  |                              |  | 1.1 (±0.12) |
| HT2                      |  |                              |  | 0.8 (±0.11) |
| UT                       | 1.6 (±0.16)                                |                              |  |             |
| HT1                      | 60   |                              | 1.5 (±0.15)                            |             |
| HT2                      |  |                              | 1.1 (±0.12)                            |             |
| UT                       |  |                              | 1.2 (±0.14)                            |             |
| HT1                      |  |                              | 1.2 (±0.14)                            |             |
| HT2                      | 1.1 (±0.16)                                |                              |  |             |

was found to improve the decay resistance of the composites. However, decay resistance of all the composite was determined to be lower than that of the neat polypropylene. According to Table 2, the decay resistance of the composites with heat-treated wood was higher as compared with the composites with untreated wood. The results showed that the decay resistance of the composites decreased when the filler size decreased from 40 to 60, while the decay resistance of the composites generally increased with 100 mesh wood filler. As a result, it can be said that both heat treatment of wood and filler loading and size have an effect on the decay resistance of the composites.

The weight loss was determined to range from 0.9 % to 1.6 %, and the maximum and minimum mass losses were found for the composites with 60 mesh untreated wood for 20 % loadings, and with 40 mesh heat-treated wood for 20 % loadings.

### 3.2 FTIR Analysis 3.2. FTIR analiza

The FTIR analysis was conducted to determine the chemical structure of neat PP, composites with untreated wood, wood heat-treated at 180 °C and 220 °C. The FTIR-ATR main peaks of neat PP, PP composites



**Figure 4** Spectra of neat polypropylene and composites with untreated and heat-treated wood  
**Slika 4.** Spektri čistog polipropilena i kompozita s nepregrijanim i pregrijanim drvom

**Table 3** Spectra of neat polypropylene, and composites with untreated and heat-treated wood (Li *et al.*, 2015; Ha *et al.*, 2016)

**Tablica 3.** Spektri čistog polipropilena i kompozita s nepregrijanim i pregrijanim drvom (Li *et al.*, 2015.; Ha *et al.*, 2016.)

| Wavenumber, cm <sup>-1</sup><br>Valni broj, cm <sup>-1</sup> | Functional groups<br>Funkcionalne skupine        |
|--|--|
| 3300   | Hydroxyl groups of cellulose (-OH)               |
| 2950, 2839   | C-H stretch vibration of PP                      |
| 1601   | C=C stretch vibration of hemicellulose           |
| 1457   | -CH <sub>2</sub> bending vibration of PP         |
| 1376   | -CH <sub>3</sub> bending vibration of PP         |
| 1167   | Symmetric -CH <sub>3</sub> deformation vibration |
| 1043   | C-O-C and C-O groups of cellulose and lignin     |
| 998, 973   | -CH <sub>3</sub> wagging vibration of PP         |

with untreated wood, wood heat-treated at 180 °C and 220 °C are given in Figure 4.

According to Figure 4, the main spectra of neat polypropylene were ranged from 973 cm<sup>-1</sup> to 2950 cm<sup>-1</sup>. 1457 cm<sup>-1</sup> and 1376 cm<sup>-1</sup> peaks show -CH<sub>2</sub> and -CH<sub>3</sub> bending vibration of polypropylene (Sagativo *et al.*, 2017). 1167 cm<sup>-1</sup>, 998 cm<sup>-1</sup>, and 973 cm<sup>-1</sup> peaks belong to -CH<sub>3</sub> deformation vibration and -CH<sub>3</sub> wagging vibration, respectively. 3300 cm<sup>-1</sup> peak means -OH groups of cellulose and lignin in the composites with untreated wood (UT) and heat-treated wood (HT1 and HT2). 1601 cm<sup>-1</sup> and 1043 cm<sup>-1</sup> peaks show C=C stretch of hemicellulose and C-O-C and C-O stretch of carbohydrates in cellulose chains (Li *et al.*, 2015; Ha *et al.*, 2016) (Table 3). As a result, the structural properties of all the composites were observed to be similar.

#### 4 CONCLUSIONS 4. ZAKLJUČAK

In the study, the accelerated weathering performance and decay resistance of the heat-treated wood

reinforced polypropylene composites were investigated. SEM images of the specimens showed the deterioration and some cracks on the surface of the composites. According to the color change results, the effect of heat treatment on the color changes was higher than that of filler size, and while the filler loadings increased from 5 % to 20 %, it was determined to increase the color changes of the composites. FTIR spectra showed that the composites had similar chemical structure. The decay test showed that the mass loss decreased with the increase of temperature in the heat-treatment of wood. The heat-treatment of wood showed to improve the antifungal efficiency of the composites. Finally, it can be said that heat-treated wood has more potential for related applications than the composite with untreated wood. However, the heat-treated wood tends to gray out rapidly in the weathering environment, which could be a drawback for common outdoor use.

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