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# Influence of Pre-Impregnation Acetic Anhydride and Heating Time on Mechanical and Physical Properties of Wood-Plastic Composites

## Utjecaj predimpregnacije anhidridom octene kiseline i vremena zagrijavanja na mehanička i fizička svojstva drvno-plastičnih kompozita

### ORIGINAL SCIENTIFIC PAPER

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**ABSTRACT** • *This research aimed to assess the influence of different pre-impregnation times (PITs) (60, 180, and 300 min), heating or reaction times (H/RTs) (60, 90, and 120 min), and chemical modification of wood flour (WF) on the mechanical and physical properties of wood-plastic composites (WPCs). The study employed acetylated beech (*Fagus orientalis* L.) flour as the filler and polypropylene (PP) as the matrix phase producing of WPC samples through melt compounding and injection molding. The resulting composites underwent testing for their physical and mechanical properties. The findings revealed that WPCs derived from acetylated wood with PITs of 60 min and H/RTs of 60 min exhibited the highest mechanical properties, except for the bending modulus. Moreover, the lowest water absorption (WA) was observed in the PITs-H/RTs combination of 60-120 min, while the lowest thickness swelling (TS) occurred in the PITs-H/RTs combination of 300-60 min. The simultaneous utilization of pre-impregnation and reaction times demonstrated a synergistic effect on the physical and mechanical properties. Consequently, the chemical modification of wood flour and the application of suitable reaction times improved the interfacial adhesion, thereby enhancing the overall performance of the WPCs.*

**KEYWORDS:** WPCs; wood flour; acetylation; chemical modification; mechanical properties

**SAŽETAK** • *Cilj ovog istraživanja bio je procijeniti utjecaj različitih vremena predimpregnacije (PIT) (60, 180 i 300 min), vremena zagrijavanja (H/RT) (60, 90 i 120 min) i kemijske modifikacije drvnog brašna (WF) na mehanička i fizička svojstva drvno-plastičnih kompozita (WPC). U istraživanju je za proizvodnju uzoraka WPC-a taljenjem i injekcijskim prešanjem upotrijebljeno drveno brašno acetilirane bukovine (*Fagus orientalis* L.) kao punilo i polipropilen (PP) kao matrica. Zatim su ispitana fizička i mehanička svojstva dobivenih kompozita. Rezultati su*

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pokazali da drvno-plastični kompoziti dobiveni od acetiliranog drva s vremenom predimpregnacije i zagrijavanja od 60 min imaju najbolja mehanička svojstva, osim modula na savijanje. Nadalje, najmanje upijanje vode (WA) zabilježeno je za kombinaciju PIT-H/RT 60-120 min, dok je najmanje debljinsko bubrenje (TS) izmjereno za kombinaciju PIT-H/RT 300-60 min. Istodobno je primjena predimpregnacije i zagrijavanja pokazala sinergijski utjecaj na fizička i mehanička svojstva. Posljedično su kemijska modifikacija drvnog brašna i odabir odgovarajućih vremena zagrijavanja poboljšali adheziju između faza, a time i svojstva drvno-plastičnih kompozita.

**KLJUČNE RIJEČI:** WPC; drvno brašno; acetilacija; kemijska modifikacija; mehanička svojstva

## 1 INTRODUCTION

### 1. UVOD

Chemical modification is considered an active approach as the transformation occurs in the macromolecules of the wood cell wall, utilizing one or more chemicals that react to form a material integrated into the wood cell wall structure (Rowell, 2005). This alteration of the cell wall macromolecules in solid wood leads to a reduction in dimensional stability and moisture content, while enhancing resistance to biological degradation (Hill, 2006; Lyon *et al.*, 2008; Ermeydan, 2014; Thybring and Fredriksson, 2021; Zhou and Liu, 2022). A prominent challenge arises when hydrophilic lignocellulosic fibers are used as reinforcements in a hydrophobic synthetic matrix, as the lack of compatibility between the two materials results in disparate behavior and poor adhesion (Özmen *et al.*, 2013a).

The properties and performance of wood and wood-based composites were improved by chemical modification, which is defined as chemical reaction between some reactive site of a lignocellulose material and a simple single chemical reagent, with or without catalyst, to form a covalent bond between them. Acetylation is a reaction that leads to a change in the chemical and physical properties of the wood substrate and results in the substitution of hydrophilic hydroxyl groups with hydrophobic acetyl groups (Rowell, 2006a, 2006b; Rowell, 2007; Bodîrlău *et al.*, 2012).

The poor compatibility between the reinforcing material and the matrix contributes to a decline in the mechanical properties of the composite (Özmen *et al.*, 2013b; Kallakas *et al.*, 2015). To address this issue and enhance the interfacial adhesion between the polymer matrix and wood flour (WF), various chemical modifications such as alkaline treatment, acetylation, epoxidation, benzylation, silane treatment, or other WF modifications have been employed (Rowell, 2006a; Farsi, 2010; Gwon *et al.*, 2010; Tesinova, 2011; Teacă *et al.*, 2014; Kallakas *et al.*, 2015; de Prá Andrade and Poletto, 2021).

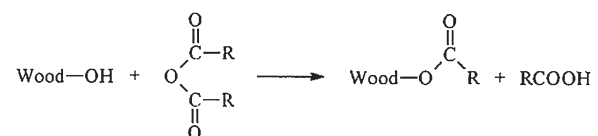
Chemical modification of *Abies alba* L. softwood samples was conducted by succinic anhydride (SA) and maleic anhydride (MA) reagents in the presence of xylene as solvent (reaction time 1 h and 90 °C), where MA exhibited lower reactivity towards wood than SA, presumably due to chemical structure (Teacă *et al.*, 2014).

The conventional acetylation process involves the impregnation of dried wood with liquid-phase acetic anhydride, followed by the application of external heat (Hung *et al.*, 2016). Acetylation of wood is a widely used method for modifying wood with acetic anhydride, which leads to the esterification of accessible hydroxyl groups in the cell wall with acetyl groups (Figure 1).

Larsson-brelid *et al.* (2006) conducted a study on WPCs using acetic anhydride-modified solid wood. Previous research has demonstrated that WPC materials reinforced with acetylated wood fibers exhibit improved mechanical properties, reduced moisture content, and enhanced resistance to brown-rot decay compared to unmodified WPC materials (Abdul Khalil *et al.*, 2002; Segerholm *et al.*, 2005).

The properties of acetylated wood are influenced by the specific method of acetylation. Factors such as reaction temperature, reaction time, type of catalysts, and their quantities significantly affect the extent of fiber degradation during the treatment process (Rowell, 1983). The weight percent gain (WPG) increases as the reaction temperature and time increase. For instance, in the chemical modification of pine wood using various chemicals, such as liquid acetic anhydride, the WPG of acetylated pine wood increases with higher temperature values (from 100 °C to 160 °C) and longer reaction times (from 60 min to 300 min) (Rowell, 1983).

This study aimed to investigate the mechanical and physical properties of polypropylene composites (WPCs) reinforced with acetylated and unmodified beech WF. Three different pre-impregnation times (PITs) and three different heating or reaction times (H/RTs) were evaluated during the acetylation process using acetic anhydride. The obtained results were compared with those of unmodified WPCs.



**Figure 1** Anhydride modification scheme, where R = CH<sub>3</sub> (acetic anhydride) (Papadopoulos *et al.*, 2019)

**Slika 1.** Shema modifikacije anhidrida, pri čemu je R = CH<sub>3</sub> (anhidrid octene kiseline) (Papadopoulos *et al.*, 2019.)

## 2 MATERIALS AND METHODS

### 2. MATERIJALI I METODE

#### 2.1 Materials

##### 2.1.1. Materijali

Beech wood (*Fagus orientalis* L.) was sourced from the Kelardasht forest in northern Iran. The wood was initially cut into small pieces and then further reduced in size using a hammer mill. The resulting WF had a particle size ranging from 0.25 mm to 0.42 mm. Before impregnation and reaction processes, the wood samples were subjected to oven drying at (103±2) °C for 24 hours. Subsequently, the samples were allowed to cool to room temperature in a desiccator containing phosphorous pentoxide before their weight was determined. The moisture content of WF before reaction process was 0 %. Oven-dried WF was stored in sealed plastic bags.

Polypropylene (PP) with a melt flow index (MI) of 10 g/10 min and a density of 0.95 g/cm<sup>3</sup> was supplied by the Tabriz Petrochemical Company in Iran.

The coupling agent used in this study was maleated polypropylene (MAPP), obtained from Eastman Chemical Products, Inc.; as Epolene G-3003TM polymer with 8 % acid anhydride and a molecular weight of 103,500 (Nourbakhsh and Ashori, 2008).

Acetic anhydride (AA), a reagent required for the acetylation process, was purchased from Merck Company in Germany. Its molecular weight, purity, density, and melting point were 102.1 g/mol, 96 %, 1.08 g/cm<sup>3</sup>, and -73.1 °C, respectively (Kown and Nadir, 2015).

#### 2.2 Acetylation of beech wood flour

##### 2.2.1. Acetilacija drvnog brašna bukovine

A total of 500 g of oven-dried beech wood flour was placed in 2-L Erlenmeyer flasks along with acetic anhydride (96 %) for pre-impregnation. Catalysts were not used during the pre-impregnation process, which took place at room temperature for 60, 180, or 300 minutes as pre-impregnation times (*PITs*). The acetic

anhydride treatment of the WF was conducted through a reaction at (103±2) °C for 60, 90, or 120 minutes as heating or reaction times (*H/RTs*). These reaction times were determined based on previous studies by Serin (2005), Dizman (2005), and Cavdar *et al.* (2014), respectively. After the reaction, the WF was thoroughly washed with semi-warm deionized water to remove any residual chemicals and by-products. The acetylated beech WF was then dried in an oven at (103±2) °C for 24 hours. Subsequently, it was placed in a desiccator containing phosphorous pentoxide until it reached room temperature, and the weight percentage gain (*WPG*) was determined using Eq. 1:

$$WPG (\%) = [(W2 - W1)/W1] * 100 \quad (1)$$

In the equation, *W1* represents the weight of the sample before treatment, and *W2* represents the weight of the sample after treatment.

#### 2.3 Production of WPCs

##### 2.3.1. Proizvodnja WPC-a

The composition of acetic anhydride-treated WF/PP composites and the corresponding weight percentage gain (*WPG*) are presented in Table 1.

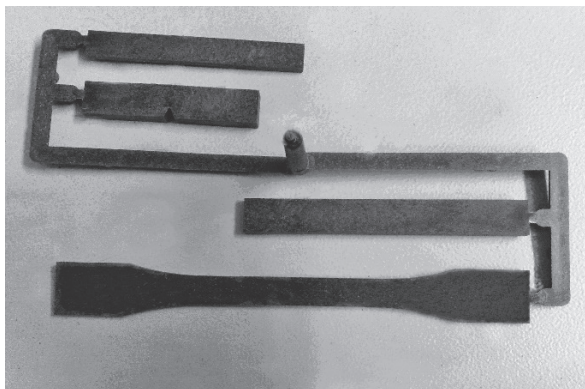
Before the sample preparation, both unmodified and modified beech wood flours were subjected to drying in an oven at a temperature of (103±2) °C for 24 hours. The mixing of the components was conducted using a Hake internal mixer (HBI System 90, USA) at a temperature of 180 °C and a rotation speed of 60 rpm.

The process involved feeding the PP into the mixing chamber, followed by the addition of the coupling agent (PP-g-MA) once the PP had melted. After five minutes, the WF was introduced, and the total mixing time was 11 minutes. The resulting mixture was then ground using a pilot scale grinder (Wieser, WGLS 200/200 Model, Germany). The obtained granules were subsequently dried at a temperature of 70 °C for 24 hours. Test samples were prepared by injection

**Table 1** Procedure of chemical modification of wood flour  
**Tablica 1.** Postupak kemijske modifikacije drvnog brašna

| WPC or Treatment code<br><i>WPC ili oznaka tretmana</i> | WF, wt % | PP, wt % | MAPP, wt % | <i>PITs</i> , min | <i>H/RTs</i> , min |
|---|----------|----------|------------|-------------------|--------------------|
| <i>PITs</i> 60- <i>H/RTs</i> 60                         | 60       | 40       | 2          | 60                | 60                 |
| <i>PITs</i> 60- <i>H/RTs</i> 90                         | 60       | 40       | 2          | 60                | 90                 |
| <i>PITs</i> 60- <i>H/RTs</i> 120                        | 60       | 40       | 2          | 60                | 120                |
| <i>PITs</i> 180- <i>H/RTs</i> 60                        | 60       | 40       | 2          | 180               | 60                 |
| <i>PITs</i> 180- <i>H/RTs</i> 90                        | 60       | 40       | 2          | 180               | 90                 |
| <i>PITs</i> 180- <i>H/RTs</i> 120                       | 60       | 40       | 2          | 180               | 120                |
| <i>PITs</i> 300- <i>H/RTs</i> 60                        | 60       | 40       | 2          | 300               | 60                 |
| <i>PITs</i> 300- <i>H/RTs</i> 90                        | 60       | 40       | 2          | 300               | 90                 |
| <i>PITs</i> 300- <i>H/RTs</i> 120                       | 60       | 40       | 2          | 300               | 120                |
| Control   | 60       | 40       | 2          | -                 | -                  |

*PITs* – Pre-impregnation times, *H/RTs* – Heating or reaction times, WF – Wood flour, PP – Polypropylene, MAPP – Maleic anhydride polypropylene / *PITs* – vrijeme predimpregnacije, *H/RTs* – vrijeme zagrijavanja ili reakcije, WF – drveno brašno, PP – polipropilen, MAPP – anhidrid maleinske kiseline – polipropilen



**Figure 2** Mechanical testing samples  
**Slika 2.** Uzorci za mehanička ispitivanja

molding according to the ASTM D 3641 (2015) standard using an injection molding machine at a temperature of 185 °C and a pressure of 10 MPa (Iman machine, Iran). Finally, the specimens were stored in controlled conditions with a relative humidity of 50 % and a temperature of 23 °C for at least 40 hours prior to testing. A sample product ready for mechanical testing is shown in Figure 2. Three replicated samples were tested for each property at each treatment level.

## 2.4 Measurements

### 2.4. Mjerenja

#### 2.4.1 Mechanical properties

##### 2.4.1. Mehanička svojstva

Flexural and tensile tests were carried out in accordance with ASTM D 790 (2007) and D 638 (2007) standards, respectively, using an Instron machine (Model 1186, England). The tests were performed at a crosshead speed of 5 mm/min. For the Izod impact test, a Zwick impact tester (Model SIT 20 D, Santam Co., Iran) was employed. To prepare the samples, a notch was made at the center of one longitudinal side, in accordance with the specifications provided in ASTM D 256 (2007).

#### 2.4.2 Physical properties

##### 2.4.2. Fizička svojstva

Water absorption (*WA*) measurements were conducted following the guidelines outlined in ASTM D 570-98 (2010) standard. To determine the water absorption of the acetylated wood-plastic composite (WPC) specimens, three specimens of each WPC type were first dried in an oven at (103±2) °C for 24 hours. The dried specimens were weighed with a precision of 0.001 g and then immersed in distilled water at room temperature for a period of 24 hours. After the immersion time elapsed, the specimens were carefully removed from the water and any excess moisture on the surface was removed using blotting paper. The weight of the specimens was then measured again after 24 hours of immersion in water at room temperature. The

mean percentages of water absorption (*WA*) and thickness swelling (*TS*) over time were calculated for each composite (both modified and unmodified) and for each of the 10 formulations using Eqs. 2 and 3 as follows:

$$WA(t) \% = [(W_t - W_0) / W_0] \times 100 \quad (2)$$

$$TS(t) \% = [(T_t - T_0) / T_0] \times 100 \quad (3)$$

Where *WA* (*t*) represents the water absorption at time *t*, *W*<sub>0</sub> is the initial weight of the specimens, *W*<sub>*t*</sub> is the weight of the specimens at time *t*, *TS* (*t*) denotes the thickness swelling at time *t*, *T*<sub>0</sub> is the initial thickness of the specimens, and *T*<sub>*t*</sub> is the thickness of the specimens at time *t*.

## 2.5 Statistical analysis

### 2.5. Statistička analiza

The statistical analysis was performed using the SPSS software (Version 24) through the implementation of a general linear model (Univariate). To understand the combined effect of *PITs* (min) and *H/RTs* (min), a two-way ANOVA (analysis of variance) technique was used in the experiments. To assess the statistical significance at a significance level of *p*<0.05, the Duncan multiple range test was employed.

## 3 RESULTS AND DISCUSSION

### 3. REZULTATI I RASPRAVA

#### 3.1 Weight percent gain

##### 3.1. Povećanje mase

The result of the reaction between beech wood flour hydroxyl groups and acetic anhydride (AA), in other words the weight percentage gain of the acetic anhydride chemically modified WF is presented in Figure 3.

The individual effects of pre-impregnation times and heating/reaction times on the weight percent gain (*WPG*) of PP composites filled with AA-modified WF are summarized in Table 2.

Statistically, results showed that the interaction between the variables *PITs* and *H/RTs* was negative and had a significant effect on the weight percent gain of samples.

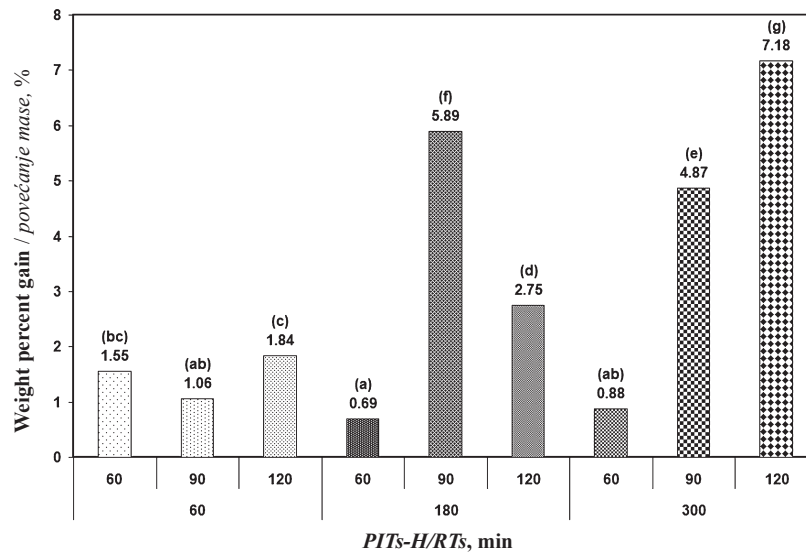
According to a statistical analysis, the individual effect of variables *PITs* and *H/RTs* showed significant effects on the *WPG* measured. The interaction between *PITs* and *H/RTs* was negative and indicated a significant effect on the *WPG* factor within the range of 95 % confidence for the experimental WPC samples made from AA-treated wood flour investigated.

The highest (7.18 %) and lowest (0.69 %) *WPG* levels were achieved for 300-120 min and 180-60 min of *PITs*-*H/RTs*, respectively (Figure 3).

**Table 2** Individual effect of pre-impregnation times and heating/reaction times on weight percent gain of PP composites filled with AA-modified WF

**Tablica 2.** Individualni utjecaj vremena predimpregnacije i vremena zagrijavanja/reakcije na povećanje mase PP kompozita ispunjenih AA modificiranim WF-om

|  | Pre-impregnation times / Vremena predimpregnacije      |         |         |
|--|--|---------|---------|
| Property / Svojstvo                        | 60 min   | 180 min | 300 min |
| Weight percent gain, % / povećanje mase, % | 1.48   | 3.11    | 4.31    |
|  | Heating/reaction times / Vremena zagrijavanja/reakcije |         |         |
| Property / Svojstvo                        | 60 min   | 90 min  | 120 min |
| Weight percent gain, % / povećanje mase, % | 1.04   | 3.98    | 3.92    |



**Figure 3** Interaction effect of pre-impregnation (PITs) and heating or reaction times (H/RTs) on weight percent gain of PP composites filled with AA-modified WF; Groups with the same letters in each row indicated no statistical difference ( $p < 0.05$ ) between samples according to Duncan’s multiple range test

**Slika 3.** Utjecaj interakcije predimpregnacije (PITs) i vremena zagrijavanja ili reakcije (H/RTs) na povećanje mase PP kompozita ispunjenih AA modificiranim WF-om; prema Duncanovu testu, grupe s istim slovima u svakom stupcu nisu pokazale statistički značajnu razliku ( $p < 0,05$ ) među uzorcima

### 3.2 Mechanical properties

#### 3.2.1 Mehanička svojstva

The mechanical properties of PP composites filled with acetic anhydride WF and Duncan’s multiple range test results are shown in Figures 4, 5, 6, 7, and 8.

The individual effects of pre-impregnation times and heating/reaction times on the mechanical proper-

ties of PP composites filled with AA-modified WF are summarized in Tables 3 and 4, respectively.

#### 3.2.1 Flexural strength

##### 3.2.1.1 Čvrstoća na savijanje

Statistically, it was shown that the interaction between the variables PITs and H/RTs was negative and exhibited a significant effect on the flexural strength

**Table 3** Individual effect of pre-impregnation times on mechanical properties of PP composites filled with AA-modified WF

**Tablica 3.** Individualni utjecaj vremena predimpregnacije na mehanička svojstva PP kompozita ispunjenih AA modificiranim WF-om

| Mechanical properties<br>Mehanička svojstva                           | Pre-impregnation times / Vremena predimpregnacije |                           |                          |
|---|---|---------------------------|--------------------------|
|   | 60 min  | 180 min                   | 300 min                  |
| Flexural strength, MPa / čvrstoća na savijanje, MPa                   | 55.68 (2.4) <sup>b</sup>                          | 52.02 (1.3) <sup>a</sup>  | 52.46 (3.4) <sup>a</sup> |
| Flexural modulus, MPa / modul savijanja, MPa                          | 4648 (2262) <sup>a</sup>                          | 5996 (417) <sup>a</sup>   | 4958 (2239) <sup>a</sup> |
| Tensile strength, MPa / vlažna čvrstoća, MPa                          | 35.73 (4.1) <sup>b</sup>                          | 29.87 (2.2) <sup>a</sup>  | 29.66 (4.2) <sup>a</sup> |
| Tensile modulus, MPa / modul elastičnosti pri vlažnom naprežanju, MPa | 5561 (2387) <sup>b</sup>                          | 4373 (1348) <sup>ab</sup> | 3285 (1400) <sup>a</sup> |
| Impact strength, J/m / čvrstoća na udarac, J/m                        | 0.42 (0.1) <sup>a</sup>                           | 0.41 (0.1) <sup>a</sup>   | 0.38 (0.0) <sup>a</sup>  |

Groups with the same letters in each row indicated no statistical difference ( $p < 0.05$ ) between samples according to Duncan’s multiple range test. The values in parentheses are standard deviations.

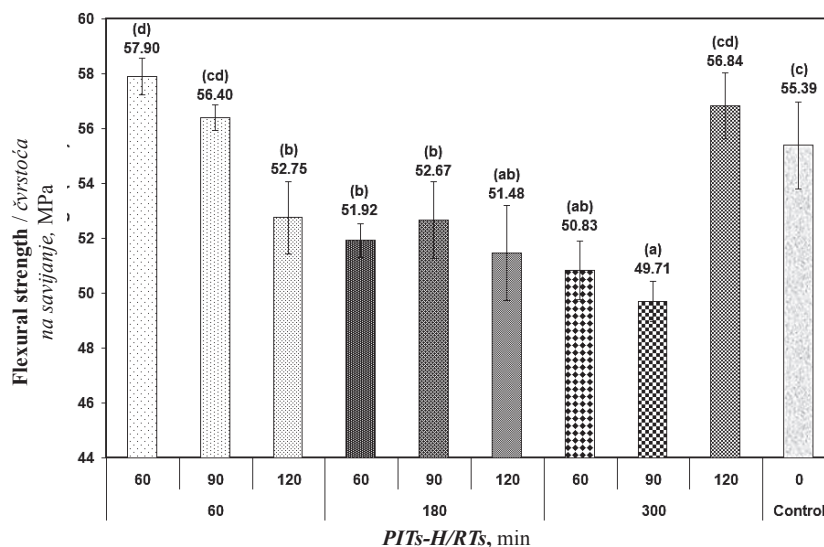
Prema Duncanovu testu, grupe s istim slovima u svakom retku nisu pokazale statistički značajnu razliku ( $p < 0,05$ ) među uzorcima. Vrijednosti u zagradama standardna su odstupanja.

**Table 4** Individual effect of heating/reaction times on mechanical properties of PP composites filled with AA-modified WF  
**Tablica 4.** Individualni utjecaj vremena zagrijavanja/reakcije na mehanička svojstva PP kompozita ispunjenih AA modificiranim WF-om

| Mechanical properties<br><i>Mehanička svojstva</i>                            | Heating/reaction times / <i>Vremena zagrijavanja/reakcije</i> |                           |                          |
|---|---|---------------------------|--------------------------|
|   | 60 min  | 90 min                    | 120 min                  |
| Flexural strength, MPa / <i>čvrstoća na savijanje</i> , MPa                   | 53.55 (3.4) <sup>a</sup>                                      | 52.92 (3.0) <sup>a</sup>  | 53.69 (2.7) <sup>a</sup> |
| Flexural modulus, MPa / <i>modul savijanja</i> , MPa                          | 4007 (2376) <sup>a</sup>                                      | 5334 (1657) <sup>ab</sup> | 6261 (381) <sup>b</sup>  |
| Tensile strength, MPa / <i>vlačna čvrstoća</i> , MPa                          | 33.26 (4.5) <sup>a</sup>                                      | 30.03 (3.1) <sup>a</sup>  | 31.97 (5.4) <sup>a</sup> |
| Tensile modulus, MPa / <i>modul elastičnosti pri vlačnom naprezanju</i> , MPa | 5351 (1907) <sup>b</sup>                                      | 3655 (2491) <sup>a</sup>  | 4214 (930) <sup>ab</sup> |
| Impact strength, J/m / <i>čvrstoća na udarac</i> , J/m                        | 0.40 (0.1) <sup>a</sup>                                       | 0.42 (0.0) <sup>a</sup>   | 0.39 (0.0) <sup>a</sup>  |

Groups with the same letters in each row indicated no statistical difference ( $p < 0.05$ ) between samples according to Duncan's multiple range test. The values in parentheses are standard deviations.

Prema Duncanovu testu, grupe s istim slovima u svakom retku nisu pokazale statistički značajnu razliku ( $p < 0,05$ ) među uzorcima. Vrijednosti u zgradama standardna su odstupanja.



**Figure 4** Interaction effect of pre-impregnation (*PITs*) and heating or reaction times (*H/RTs*) on flexural strength of PP composites filled with AA-modified WF; Groups with the same letters in each column indicated no statistical difference ( $p < 0.05$ ) between samples according to Duncan's multiple range test

**Slika 4.** Utjecaj interakcije predimpregnacije (*PITs*) i vremena zagrijavanja ili reakcije (*H/RTs*) na čvrstoću na savijanje PP kompozita ispunjenih AA modificiranim WF-om; prema Duncanovu testu, grupe s istim slovima u svakom stupcu nisu pokazale statistički značajnu razliku ( $p < 0,05$ ) među uzorcima

(*FS*) of WPC samples. According to a statistical analysis, the individual effect of variable *PITs* showed a significant effect on the *FS* measured (Table 3), while the individual effect of variable *H/RTs* had no significant effect on the *FS* measured (Table 4). The interaction between *PITs* and *H/RTs* was negative and showed a significant effect on the *FS* factor within the range of 95% confidence for the experimental WPC samples made from AA-treated wood flour investigated (Figure 4).

The test results showed that the *PITs* and *H/RTs*, as reaction variables of chemical modification with AA, affected the mechanical and physical properties of the composite samples. Flexural strength (*FS*) values of samples increased with the decrease in pre-impregnation times considering the individual effect of *PITs*, while the decreases were not significant considering the individual effect of *H/RTs*. The *FS* values were determined to be between 55.39 MPa (control) and 57.9

MPa of the samples produced with WF modification at 60 minutes of *PITs* (Figure 4).

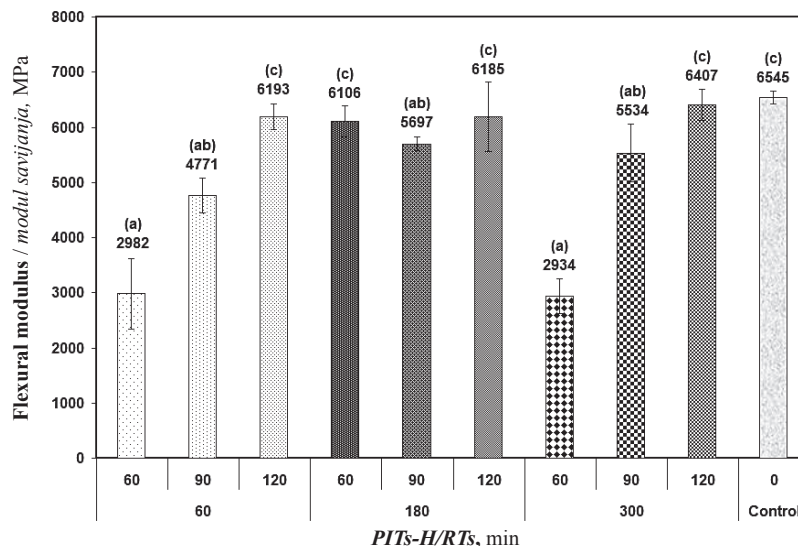
The greatest increase in *FS* was found in the samples modified with 60-60 min of *PITs-H/RTs* in the interaction effect of reaction variables (57.90 MPa) (Figure 4).

### 3.2.2 Flexural modulus

#### 3.2.2. Modul savijanja

Statistically, it was shown that the interaction between the variables *PITs* and *H/RTs* was positive and had no significant effect on the flexural modulus (*FM*) of WPC samples (Figure 5).

According to a statistical analysis, the individual effect of variable *H/RTs* showed significant effect on the *FM* measured (Table 4), while the individual effect of variable *PITs* had no significant effect on the *FM* measured (Table 3). The interaction between *PITs* and



**Figure 5** Interaction effect of pre-impregnation (*PITs*) and heating or reaction times (*H/RTs*) on flexural modulus of PP composites filled with AA-modified WF; Groups with the same letters in each column indicated no statistical difference ( $p < 0.05$ ) between samples according to Duncan's multiple range test

**Slika 5.** Utjecaj interakcije predimpregnacije (*PITs*) i vremena zagrijavanja ili reakcije (*H/RTs*) na modul savijanja PP kompozita ispunjenih AA modificiranim WF-om; grupe s istim slovima u svakom stupcu nisu pokazale statistički značajnu razliku ( $p < 0,05$ ) među uzorcima

*H/RTs* was positive and showed no significant effect on the *FM* factor within the range of 95 % confidence for the experimental WPC samples made from AA-treated wood flour investigated (Figure 5).

The use of different heating or reaction times (*H/RTs*) in the chemical modification process with AA led to an increase in the flexural modulus (*FM*). Among the various *H/RTs* tested, the highest increase in *FM* was observed with 120-minute *H/RTs* (6261 MPa) as individual effect; however, there was a significant difference in *FM* values between the 60-minute and 120-minute *H/RTs*. The greatest value in *FM* was found in the unmodified or control samples (6545 MPa) (Figure 5).

The lowest values in *FS* and *FM* were found in the samples modified with 300-90 min (49.71 MPa) and 300-60 min (2934 MPa) of *PITs-H/RTs* in the interaction effect of reaction variables, respectively (Figures 4 and 5). Compared to the control group, the WPCs produced in 60-60 min and 300-120 min of *PITs-H/RTs* provided about 4% higher *FS* and 2% lower *FM* values, respectively.

### 3.2.3 Tensile strength

#### 3.2.3. Vlačna čvrstoća

Statistically, it was shown that the interaction between the variables *PITs* and *H/RTs* was positive and had no significant effect on the tensile strength (*TS*) of WPC samples. According to a statistical analysis, the individual effect of variable *PITs* showed a significant effect on the *TS* measured (Table 3), while the individual effect of variable *H/RTs* had no significant effect on the *TS* measured (Table 4). The interaction between *PITs* and *H/RTs* was positive and showed no significant

effect on the *TS* factor within the range of 95 % confidence for the experimental WPC samples made from AA-treated wood flour investigated (Figure 6).

The tensile strength (*TS*) values varied depending on the reaction variables, ranging from 29.62 MPa to 38.39 MPa in the control samples and samples produced by modifying WF in 60 min of *PITs* and 60 min of *H/RTs*, respectively (Figure 6).

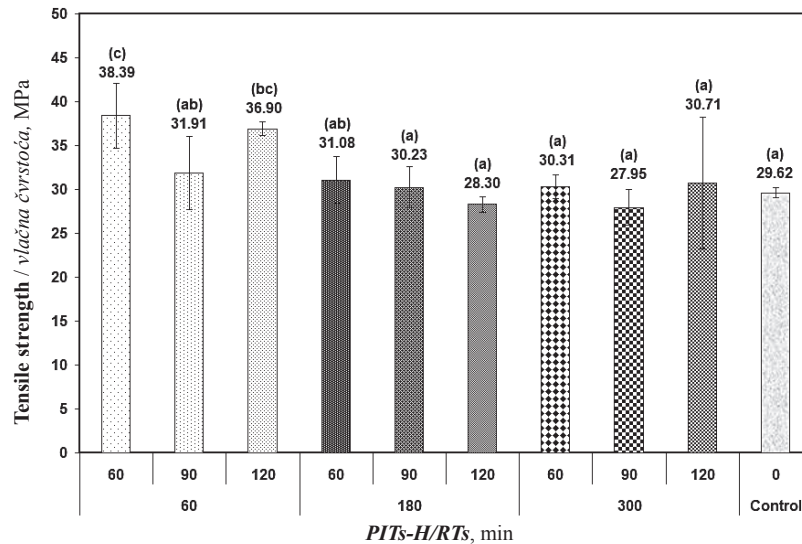
The highest *TS* value was obtained from samples produced through a 60-minute pre-impregnation time (*PIT*). Remarkable changes were observed in *TS* values between the control samples and the modified ones.

### 3.2.4 Tensile modulus

#### 3.2.4. Modul elastičnosti pri vlačnom naprezanju

Statistically, it was shown that the interaction between the variables *PITs* and *H/RTs* was negative and had a significant effect on the tensile modulus (*TM*) of WPC samples. According to a statistical analysis, the individual effect of variables *PITs* and *H/RTs* showed significant effects on the *TM* measured (Tables 3 and 4). The interaction between *PITs* and *H/RTs* was negative and showed a significant effect on the *TM* factor within the range of 95 % confidence for the experimental WPC samples made from AA-treated wood flour investigated (Figure 7).

In addition, the tensile modulus of the samples also exhibited an increase caused by variations in both reaction variables, namely *PITs* and *H/RTs*, during the acetylation of WF with AA. This increase in the *TM* can be attributed to the enhanced stiffness of WPCs (Figure 7). The increasing severity of pre-



**Figure 6** Interaction effect of pre-impregnation (*PITs*) and heating or reaction times (*H/RTs*) on tensile strength of PP composites filled with AA-modified WF; Groups with the same letters in each column indicated no statistical difference ( $p < 0.05$ ) between samples according to Duncan’s multiple range test

**Slika 6.** Utjecaj interakcije predimpregnacije (*PITs*) i vremena zagrijavanja ili reakcije (*H/RTs*) na vlačnu čvrstoću PP kompozita ispunjenih AA modificiranim WF-om; prema Duncanovu testu, grupe s istim slovima u svakom stupcu nisu pokazale statistički značajnu razliku ( $p < 0,05$ ) među uzorcima

impregnation times and heating/reaction times negatively affected the tensile modulus and other mechanical properties.

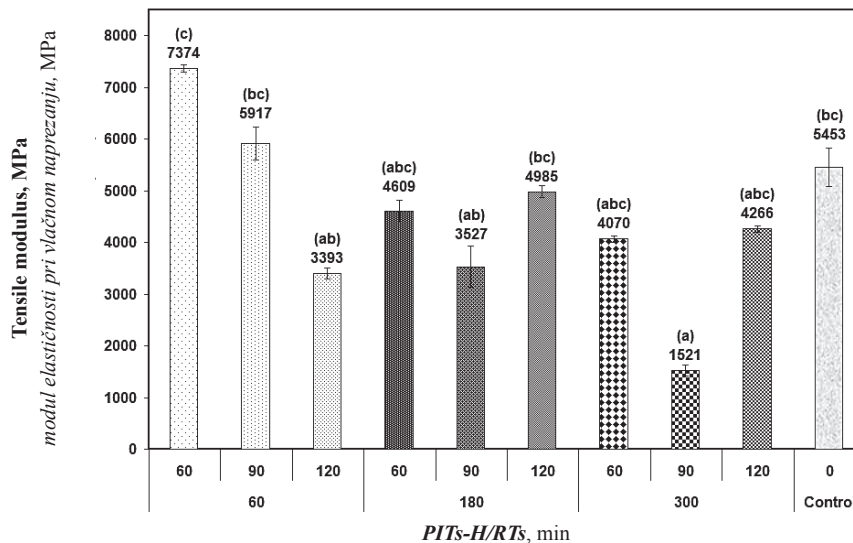
### 3.2.5 Impact strength

#### 3.2.5. Čvrstoća na udarac

Statistically, it was shown that the interaction between the variables *PITs* and *H/RTs* was positive and had no significant effect on the impact strength (*IS*) of WPC samples. According to a statistical analysis, the

individual effect of variables *PITs* and *H/RTs* showed no significant effects on the *IS* measured (Tables 3 and 4). The interaction between *PITs* and *H/RTs* was positive and showed no significant effect on the *IS* factor within the range of 95 % confidence for the experimental WPC samples made from AA-treated wood flour investigated (Figure 8).

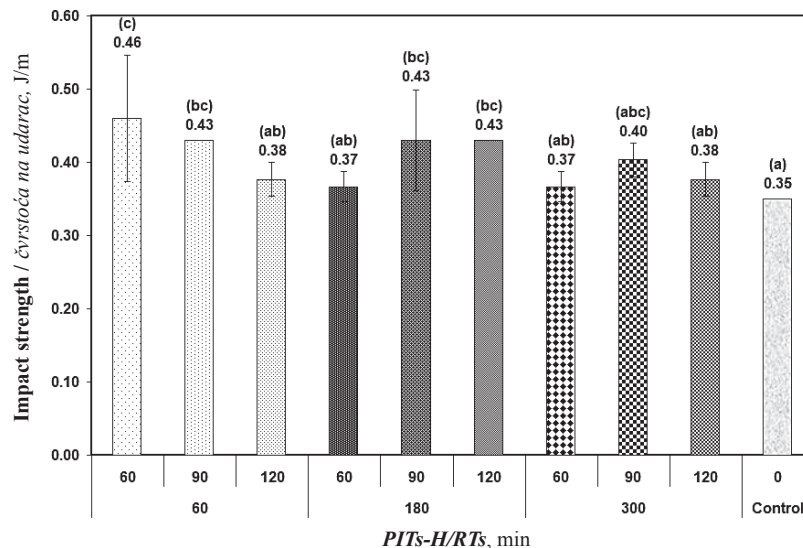
The impact strength (*IS*) values of samples increased with the decrease in pre-impregnation times in the individual effect of *PITs*, while the values did not



**Figure 7** Interaction effect of pre-impregnation (*PITs*) and heating or reaction times (*H/RTs*) on tensile modulus of PP composites filled with AA-modified WF; Groups with the same letters in each column indicated no statistical difference ( $p < 0.05$ ) between samples according to Duncan’s multiple range test

**Slika 7.** Utjecaj interakcije predimpregnacije (*PITs*) i vremena zagrijavanja ili reakcije (*H/RTs*) na modul elastičnosti pri vlačnom naprezanju PP kompozita ispunjenih AA modificiranim WF-om; prema Duncanovu testu, grupe s istim slovima u svakom stupcu nisu pokazale statistički značajnu razliku ( $p < 0,05$ ) među uzorcima





**Figure 8** Interaction effect of pre-impregnation (*PITs*) and heating or reaction times (*H/RTs*) on impact strength of PP composites filled with AA-modified WF; Groups with the same letters in each column indicated no statistical difference ( $p < 0.05$ ) between samples according to Duncan's multiple range test

**Slika 8.** Utjecaj interakcije predimpregnacije (*PITs*) i vremena zagrijavanja ili reakcije (*H/RTs*) na čvrstoću na udarac PP kompozita ispunjenih AA modificiranim WF-om; prema Duncanovu testu, grupe s istim slovima u svakom stupcu nisu pokazale statistički značajnu razliku ( $p < 0,05$ ) među uzorcima

show significant changes for the individual effect of *H/RTs*. The *IS* values were found to be between 0.35 J/m (control) and 0.46 J/m from the samples produced by modifying WF in 60 min of *PITs* and 60 min of *H/RTs* (Figure 8).

Figure 4 clearly shows that the acetylation of beech WF with acetic anhydride had a positive effect on flexural strength, while the variation in the pre-impregnation and heating or reaction times had no positive effect on flexural modulus in comparison with unmodified or control samples (Figure 5).

The greatest increase in *TS* was found for samples modified with 60-60 min of *PITs-H/RTs* in the interaction effect of reaction variables (38.39 MPa) (Figure 6), and the greatest value in *TM* was also found in samples modified with 60-60 min of *PITs-H/RTs* in the interaction effect of reaction variables (7374 MPa) (Figure 7). The lowest values in *TS* and *TM* were found in samples modified with 300-90 min (27.95 MPa) and (1521 MPa) of *PITs-H/RTs* in the interaction effect of reaction variables, respectively.

Compared to the control group, composites produced with 60-60 min of *PITs-H/RTs* WF provided about 22 % and 26 % higher *TS* and *TM* values, respectively (Figures 6 and 7).

The greatest increase in the *IS* was found in samples modified with 60-60 min of *PITs-H/RTs* in the interaction effect of reaction variables (0.46 J/m), while the lowest values in the *IS* were found in the unmodified samples (0.35 J/m), 180-60 (0.37 J/m), and 300-60 min (0.37 J/m) of *PITs-H/RTs* in the interaction effect of reaction variables, respectively (Figure 8). Compared to the control group, the WPCs produced in 60-60 min of *PITs-H/RTs* WF provided about 23 % higher impact strength values.

### 3.3 Physical properties

#### 3.3. Fizička svojstva

The individual effects of pre-impregnation times and heating/reaction times on the physical properties of PP composites filled with AA-modified WF are summarized in Tables 5 and 6, respectively.

**Table 5** Individual effect of pre-impregnation times on physical properties of PP composites filled with AA-modified WF  
**Tablica 5.** Individualni utjecaj vremena predimpregnacije na fizička svojstva PP kompozita ispunjenih AA modificiranim WF-om

| Physical properties<br><i>Fizička svojstva</i>                      | Pre-impregnation times / <i>Vremena predimpregnacije</i> |                         |                         |
|---|--|-------------------------|-------------------------|
|   | 60 min   | 180 min                 | 300 min                 |
| Water absorption (24 h), % / <i>upijanje vode (24 h), %</i>         | 1.86 (1.5) <sup>b</sup>                                  | 1.06 (0.2) <sup>a</sup> | 1.66 (0.5) <sup>b</sup> |
| Thickness swelling (24 h), % / <i>debljinsko bubrenje (24 h), %</i> | 0.69 (4.3) <sup>a</sup>                                  | 0.19 (4.4) <sup>a</sup> | 0.65 (4.1) <sup>a</sup> |

Groups with the same letters in each row indicated no statistical difference ( $p < 0.05$ ) between samples according to Duncan's multiple range test. The values in parentheses are standard deviations.

Prema Duncanovu testu, grupe s istim slovima u svakom retku nisu pokazale statistički značajnu razliku ( $p < 0,05$ ) među uzorcima. Vrijednosti u zagradama standardna su odstupanja.

**Table 6** Individual effect of heating/reaction times on physical properties of PP composites filled with AA-modified WF

**Tablica 6.** Individualni utjecaj vremena zagrijavanja/reakcije na fizička svojstva PP kompozita ispunjenih AA modificiranim WF-om

| Physical properties<br><i>Fizička svojstva</i>                      | Heating/Reaction times / <i>Vremena zagrijavanja/reakcije</i> |                         |                         |
|---|---|-------------------------|-------------------------|
|   | 60 min  | 90 min                  | 120 min                 |
| Water absorption (24 h), % / <i>upijanje vode (24 h), %</i>         | 2.29 (1.3) <sup>b</sup>                                       | 1.20 (0.3) <sup>a</sup> | 1.08 (0.2) <sup>a</sup> |
| Thickness swelling (24 h), % / <i>debljinsko bubrenje (24 h), %</i> | -0.59 (5.7) <sup>a</sup>                                      | 1.19 (1.1) <sup>a</sup> | 0.94 (4.4) <sup>a</sup> |

Groups with the same letters in each row indicated no statistical difference ( $p < 0.05$ ) between samples according to Duncan's multiple range test. The values in parentheses are standard deviations.

Prema Duncanovu testu, grupe s istim slovima u svakom retku nisu pokazale statistički značajnu razliku ( $p < 0,05$ ) među uzorcima. Vrijednosti u zagradama standardna su odstupanja.

The water absorption (*WA*) values of samples decreased with the increase in pre-impregnation times and heating or reaction times for the individual effects of *PITs* and *H/RTs* (Tables 5 and 6), where the values showed significant differences for both reaction variables. The same happened for thickness swelling (*TSw*), where the values showed significant differences in the individual effect of both reaction variables, but without special order. The greatest decrease in water absorption (*WA*) of 24 h was found for samples modified with 60-120 min of *PITs-H/RTs* (0.94 %) in the interaction effect of reaction variables (Table 7).

In contrast, the greatest increase value in *WA* of 24 h was found for samples modified with 60-60 min of *PITs-H/RTs* in the interaction effect of reaction variables (3.45 %). Compared to the control group, composites produced in 60-120 min and 60-60 min of *PITs-H/RTs* provided about 25 % lower and 63% higher *WA* values, respectively (Table 7).

The results showed that a significant reduction in *TSw* and *WA* properties of the WPCs was produced by using acetylated wood. This phenomenon may be explained by the fact that the amount of hydroxyl groups was substituted by acetyl moieties following the acetylation treatment, which leads to the decrease in the hydrophilicity of wood particles (Hung *et al.*, 2016). The greatest decrease in thickness swelling (*TSw*) of 24 h was found in samples modified with 180-60 min of *PITs-H/RTs* in the interaction effect of reaction variables (-2.58 %). In contrast, the greatest increase value in *WA* of 24 h was found in the samples modified with 60-60 min of *PITs-H/RTs* in the interaction effect of reaction variables (2.43 %). Compared to the control group, composites produced in 180-60 min and 60-60

min of *PITs-H/RTs* provided about 197 % lower and 8 % higher *TSw* values, respectively (Table 7).

The acetylation process significantly impacted the mechanical properties of wood-polymer composites (WPCs), including flexural strength (*FS*), tensile strength (*TS*), tensile modulus (*TM*), and impact strength (*IS*), with variations observed based on weight percentage gain (*WPG*) and pre-impregnation and heating or reaction times. Lower levels of *WPG* and shorter reaction times resulted in improved properties, indicating enhanced interfacial adhesion and polymer-WF interaction. However, higher levels of *WPG* and longer reaction times exhibited poor interfacial adhesion. Previous studies have highlighted the better bonding achieved between acetylated WF and the polymer matrix, although this improvement did not necessarily translate into improved mechanical properties of the composites.

This study introduced the use of acetic anhydride (AA) in different pre-impregnation and heating or reaction times, leading to enhancements in flexural strength, tensile strength, tensile modulus, and impact strength of WPCs. Similar results were found in previous studies (Abdul Khalil *et al.*, 2002; Segerholm *et al.*, 2007; Kwon and Ayrilmis, 2015). Acetylated wood particles contribute to the sufficient interfacial adhesion with the polymer matrix, which leads to a more efficient transfer of stress along the wood particle/polymer interface as compared to the control wood/polymer (Hung *et al.*, 2016). However, no significant change was observed in the flexural modulus. This can be attributed to the altered failure mode, shifting from fiber pullout to fiber breakage, caused by improved bonding between the polymer and WF. Acetylated WF

**Table 7** Individual effect of pre-impregnation and heating or reaction times on physical properties of PP composites filled with AA-modified WF

**Tablica 7.** Individualni utjecaj vremena predimpregnacije i zagrijavanja ili reakcije na fizička svojstva PP kompozita ispunjenih AA modificiranim WF-om

| Physical properties<br><i>Fizička svojstva</i> | Treatment code ( <i>PITs/HRTs</i> ) / <i>Oznaka tretmana (PIT/HRT)</i> , min |                            |                             |                             |                            |                            |                             |                             |                            |                            |
|--|--|----------------------------|-----------------------------|-----------------------------|----------------------------|----------------------------|-----------------------------|-----------------------------|----------------------------|----------------------------|
|  | 60- 60   | 60- 90                     | 60-120                      | 180-60                      | 180-90                     | 180-120                    | 300-60                      | 300-90                      | 300-120                    | Control                    |
| <i>WA</i> (24 h), %                            | 3.45 <sup>c</sup><br>(0.4)   | 1.19 <sup>a</sup><br>(0.1) | 0.94 <sup>a</sup><br>(0.1)  | 1.12 <sup>a</sup><br>(0.1)  | 0.95 <sup>a</sup><br>(0.4) | 1.11 <sup>a</sup><br>(0.1) | 2.32 <sup>b</sup><br>(0.0)  | 1.46 <sup>ab</sup><br>(0.2) | 1.20 <sup>a</sup><br>(0.1) | 1.26 <sup>a</sup><br>(0.3) |
| <i>TSw</i> (24 h), %                           | 2.43 <sup>a</sup><br>(0.0)   | 0.00 <sup>a</sup><br>(0.0) | -0.35 <sup>a</sup><br>(0.8) | -2.58 <sup>a</sup><br>(0.8) | 1.85 <sup>a</sup><br>(0.8) | 1.30 <sup>a</sup><br>(0.3) | -1.63 <sup>a</sup><br>(0.7) | 1.73 <sup>a</sup><br>(0.1)  | 1.87 <sup>a</sup><br>(0.8) | 2.65 <sup>a</sup><br>(0.6) |

*WA* – Water absorption; *TSw* – Thickness swelling. Groups with the same letters in each row indicated no statistical difference ( $p < 0.05$ ) between samples according to Duncan's multiple range test. The values in parentheses are standard deviations.

*WA* – upijanje vode; *TSw* – debljinsko bubrenje. Prema Duncanovu testu, grupe s istim slovima u svakom retku nisu pokazale statistički značajnu razliku ( $p < 0,05$ ) među uzorcima. Vrijednosti u zagradama standardna su odstupanja.

exhibited higher flexural strength compared to unmodified WF, with a slight reduction in flexural modulus potentially attributed to the weight gain caused by acetylation. The density is one of the significant parameters affecting the mechanical properties.

The compatibility between WF and the polymer matrix improved due to reduced polarity after acetylation, resulting in increased mechanical properties. However, excessive acetylation levels could hinder effective stress transmission from the matrix to the fibers, leading to reduced composite properties. The flexural strength, tensile strength, and moduli, as well as impact strength, were significantly increased in samples treated with AA, even with lower WPG compared to other treatments. However, a linear correlation between pre-impregnation and reaction times and mechanical properties was not observed. Inadequate washing to remove unreacted chemicals and by-products after modification may have contributed to the reduction and fluctuation in properties. The application of a Soxhlet cleaning procedure after acetylation was found to have a positive effect on the mechanical properties of WPCs compared to simple water washing.

The success of chemical modification in WF for WPCs depends on various factors, such as the method used, reaction time and temperature, and removal of unreacted chemicals. Substituting hydroxyl groups in WF with acetyl groups increased the hydrophobicity of the WF surface, improving compatibility with the polymer matrix. Additionally, the upward trend observed in flexural strength, tensile strength, and moduli, and impact strength with increasing WPG may be attributed to reduced moisture sorption and increased brittleness of the samples. The use of anhydrides and higher reaction temperatures influenced the cross-linking of lignin in WF during the formation of cellulose-reinforced thermoplastic composites.

As a result of the chemical reaction between wood and acetic anhydride as the reagent, an increase in dimensions of the reacted wood species has occurred, because of swelling of the wood cell wall. In most cases, chemically modified wood has a lower capacity for water absorption, with lower equilibrium moisture content at a specified atmospheric relative humidity, compared to unmodified wood (Teaca *et al.*, 2014).

## 4 CONCLUSIONS

### 4. ZAKLJUČAK

In this research, WPCs were made from acetylated wood flour. The highest and lowest WPG levels were achieved for 300-120 min for PITs-H/RTs and 180-60 min for PITs-H/RTs, respectively. The test results revealed that the chemical modifications of WF

in different PITs and H/RTs improved the mechanical and physical properties of the WPCs. The highest mechanical properties were found in the WPCs produced with wood acetylated at PITs-H/RTs for 60-60 min with the exception of impact strength, while the least WA and TSw were found in the PITs-H/RTs for 60-120 min and PITs-H/RTs for 300-60 min, respectively. The WPCs produced with wood acetylated at PITs-H/RTs for 180-60 min affected the physical characteristics, while the positive effect did not affect all the mechanical properties compared to unmodified or control samples. Regarding the physical and mechanical properties of the WPCs, it was observed that surface modification by both anhydride acetic and reaction times improved the tensile and flexural strengths as well as WA and TSw of the composites. This was because the surface modification could improve the compatibility between wood flour and PP matrix, leading to fewer microvoids and fiber-PP debonding in the interphase region. Simultaneous use of pre-impregnation and reaction times had a synergic effect on the physical and mechanical properties.

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