

## **THE CHANGES IN THE SURFACE OF FLAT PRESSED WOOD-PLASTIC COMPOSITES EXPOSED TO ARTIFICIAL WEATHERING**

SEFA DURMAZ  
MUGLA SITKI KOCMAN UNIVERSITY  
TURKEY

ÖZLEM ÖZGENÇ  
KARADENİZ TECHNICAL UNIVERSITY  
TURKEY

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### **ABSTRACT**

In this study, the wood flour content's effect on the weathering performance of flat pressed WPC was investigated. The high density polyethylene was reinforced with four different wood flour content (10%, 30%, 50%, 70%). The weathering performance of WPC was determined by the 400 h of artificial weathering test. According to the results, the color change is inevitable as long as the wood flour is used as filler. Surprisingly, the highest color change was obtained from WPC containing 30% WF, contrary to 70% of wood flour. Similarly, the whiteness of the surface of WPC increased with exposure time. The photooxidation resulted in the chain scission and shorter molecules, which were observed by ATR-FTIR analysis. The changes in the intensity of characteristic polymer ( $2914\text{ cm}^{-1}$  and  $2846\text{ cm}^{-1}$ ) and wood peaks ( $1510\text{ cm}^{-1}$  and  $1027\text{ cm}^{-1}$ ) exhibited the photodegradation on WPCs' surface, which resulted in color change. Moreover, the light microscopy investigation showed surface degradation. The extensive weathering conditions caused surface cracks and surface roughness. The visual appearance of WPCs also demonstrated how to change the surface character of WPC during the 400 h of artificial weathering. In conclusion, the increase in the wood content increased the intensity of degradation.

**KEYWORDS:** Artificial weathering, wood content, ATR-FTIR analysis, color change, light microscopy.

## INTRODUCTION

The pressure on the environment forces the manufacturer to use materials efficiently due to the shortage of raw materials. Besides producing with less, there is also a desire for excellent properties. The composites that combine two or more materials have presented superior properties to manufacturers and consumers (Frącz and Janowski 2018). WPC are composites types with more than 80 years of history (Gardner and Murduck 2010). The combination of polymer (mostly thermoplastics) with wood flour has gained a green label for new material and reduced the production cost. Therefore, wood plastic composites (WPC) have been an essential part of the market for more than 20 years (Wei et al. 2021). While the first emergence of WPC is as deck material, they have many usage areas such as furniture, siding, timber, fence, etc. (Xu et al. 2017). As stated, most of the usage areas of WPC are usually outdoor. The materials used in the outdoor conditions are prone to have a low service life due to external factors such as UV light, off-gas, rain, heat, etc.

The degradation of wood under the appropriate conditions is one of the vital threats for both consumers and manufacturers. Besides the biological threat, such as fungi and insects, UV radiation negatively influences wood properties (Rowell 2012). The initial decomposition starts as color changes and continues crack composition with the effect of humidity which causes significant damage (Fabiya and McDonald 2010). The cell wall component, lignin, absorbs almost all UV lights, resulting in inevitable color change. The UV lights lead to changing wood's surface color from yellowish to brownish (Jankowska et al. 2017). The UV absorption of chromophoric groups in the lignin leads to produce paraquinone, which turns the surface color yellowish. However, the photooxidation results in reducing the paraquinone to hydroquinone, which restrains the yellowish surface (Muasher and Sain 2006). The leaching of lignin during the weathering causes cellulose-rich surface, which is also a result of whitening of WPCs' surface. However, the hydroxyl groups on the surface of wood fiber increase as long as degradation proceeds (Oberhofnerová and Pánek 2016). The binding of water molecules to hydroxyl groups increases moisture content, resulting in fiber swelling (Rydzkowski and Michalska-Požoga 2016). This situation is undesirable for WPC, which leads to crack formation on the surface. Therefore, mechanical properties are negatively influenced by humidity. The decrease in the mechanical properties shortens the service life of WPCs, which limits the usage areas where high strength need.

The other main component of the WPC is polymer, which has high biological durability, UV resistance, and water repellent compared to wood. Similar to wood fibers; however, the chromophoric groups in the structure of the polymer also absorb UV lights, which leads to photodegradation (Bajracharya et al. 2014). The hydroperoxide, catalyst residue, and carbonyls in polymer structure are the primary trigger of photooxidation for polymers (Kaczmarek et al. 2005, Matuana et al. 2011). The photooxidation results in the chain scission, leading to shorter molecules' formation (Muasher and Sain 2006). Therefore, the change in the density due to photodegradation influences stress transfer, which results in crack formation on the surface of WPCs (Matuana et al. 2011).

As stated above, WPC have been usually exposed to outdoor conditions (UV lights, rain, etc.) due to where they mostly maintain their service life. The main objective of this study was to investigate the effect of wood flour content on the weathering performance of flat-pressed WPC. The high density polyethylene (HDPE) was reinforced with four different wood flour content (10%, 30%, 50%, and 70%) by using the flat pressed method, which is an inexpensive method with large scale production and high production rate (Benthien and Thoemen 2012). The changes on the surface of weathered WPCs were investigated by Attenuated Total Reflection Fourier Transform Infrared spectrometry (ATR-FTIR), light microscopy, and also visual appearance.

## MATERIALS AND METHODS

### Materials

Scotch pine wood flour with a mesh size of 40 to 60 was purchased from a commercial supplier (Marmara Wood Shaving, Istanbul, Turkey). HDPE was obtained as powder form (Ucar Plastic, İzmir, Turkey). The melt flow index (MFI) and density of HDPE were 5.5 g/10 min (190°C/ 2.16 kg) and 0.965 g·cm<sup>-3</sup>, resp. The maleated anhydride grafted polyethylene (MAPE) was used as a coupling agent whose MFI and density were 3 g/10 min (190°C/ 2.16 kg) and 0.92 g·cm<sup>-3</sup>, resp.

### The production of wood plastic composites

The moisture content of wood flour was kept under 2%. The wood flour and polymer were mixed in ratio of 0:100, 10:90, 30:70, 50:50, 70:30 (w/w). MAPE as a coupling agent was added in the production at 2%. The mechanical blender (1200 rev·min<sup>-1</sup>) and a rotary drum blender (30 to 40 rev·min<sup>-1</sup> for 5 min) were used to obtain a homogeneous mixture of wood flour and polymer. The wood flour and polymer mixture were laid on the aluminum caul plate. The wax paper was used to avoid sticking the draft to the aluminum caul plate. The draft was hot-pressed at 170°C for 15 min under 24-26 kg·cm<sup>-2</sup> pressure. The target density and dimensional panels were 1 g·cm<sup>-3</sup> and 500 x 500 x 4 mm<sup>3</sup>. The panels were conditioned according to ASTM D618-21: 2021 after pressing.

### Accelerated weathering test

The weathering performance of flat pressed WPC was evaluated in a QUV/spray accelerated weathering tester (Q-Lab) according to ASTM G-154-16: 2015 during the 400 h. The WPC samples were exposed only to UV/condensation exposures, which are 2 h of UV light (60°C with 313 nm fluorescent UVB lamp) and 2 h of condensations at 50°C (dark conditions without UV exposure), resp. Three samples for each group were exposed accelerated weathering test.

### Color measurement

The changes on the surface of WPC were determined with a Minolta CM-600d spectrophotometer (Konica Minolta) equipped with an integrating sphere according to ISO 7724: 1984. Standard illuminant D65 was selected. The color measurement was carried out

in an area of 8 mm<sup>2</sup> in the 400 - 700 nm wavelength range. Six measurements were taken from the surface of WPCs for each group.

The CIELab color parameters, L\* (lightness) (darkness [-] to lighter [+], as well as a\* (red [+] to green [-] along the x-axis) and b\* (yellow [+] to blue [-] along the y-axis), were calculated using Konica Minolta Color Data Software CM-S100w Spectra Magic NX Lite (ISO 7724-2), and the color difference ( $\Delta E$ ) was calculated according to Eq. 1-4:

$$\Delta L^* = L_d^* - L_f^* \quad (1)$$

$$\Delta a^* = a_d^* - a_f^* \quad (2)$$

$$\Delta b^* = b_d^* - b_f^* \quad (3)$$

$$\Delta E^* = (\Delta L^2 + \Delta a^2 + \Delta b^2)^{1/2} \quad (4)$$

where:  $\Delta L^*$ ,  $\Delta a^*$ , and  $\Delta b^*$  determine the first (f) and different time (d) color changes  $\Delta E^*$ .

### **Attenuated total reflectance - Fourier-transform infrared spectroscopy (ATR-FTIR)**

ATR-FTIR spectroscopy analysis was performed before and after the weathering tests to examine UV light's effect on the surface chemistry of WPC. A Thermo Nicolet iS50 FTIR (Thermo Fisher Scientific Co.) spectrometer equipped with a single-bounce diamond crystal and a deuterated triglycine sulfate detector was used. The samples' surface was in contact with the ATR crystal to ensure that the surface could absorb the evanescent wave. The resultant attenuated radiation produced an ATR spectrum similar to a conventional absorption spectrum. The FTIR spectra of the samples were acquired in the range of 400-4000 cm<sup>-1</sup> with a resolution of 16 cm<sup>-1</sup>. Each spectrum was collected by 32 scans in the absorbance mode. Slight pressure was applied to the ATR crystal to obtain high-quality measurements (Fabiya and McDonald 2005). Three measurements were averaged to produce one spectrum.

### **Light microscopy analysis**

The surface of WPC was investigated with a Zeiss Stemi 305 light microscope and Zeiss AxiocAM erC5-s.

## **RESULTS AND DISCUSSION**

### **Color changes**

The change of the color on the surface of WPC is a vital obstacle that worsens the appearance and decreases the allure of naturalness. The changes in the color on the surface of WPC were examined with four parameters;  $\Delta E$ ,  $\Delta L$ ,  $\Delta a$ ,  $\Delta b$ . According to  $\Delta E$  values, the color changes increased with increasing exposure time during the 400 h. Interestingly, the highest color change was determined in WPC containing 30% of wood flour, contrary to the highest wood flour content (70%). High density polyethylene is well-known for having high UV resistance compared to other polymers such as polypropylene (Klyosov 2007). As seen in Fig. 1, the change in the color was more stable for neat HDPE. Moreover, the WPC containing 10% of wood flour presented a similar trend. After that, the increase in the wood flour content affected

color change significantly. The highest color changes were obtained from WPC containing 30% of wood flour. Moreover, the change in the color was almost all similar to WPC containing 50% of wood flour. However, a decrease was observed after that content. The change in the color on the surface of WPC containing 70% of wood flour was more moderate, contrary to expectation. Likewise, Yang et al. (2015) stated that WPC containing high wood flour content (70%) demonstrated the highest resistance against color change. However, the increase in the wood content negatively influenced the color changes compared to neat HDPE.

$\Delta L$  value indicates the changes in the lightness. The general trend in the WPC samples was rising in lightness with the exposure time. The UV lights negatively influenced the wood and polymer's chemical structure. The breaking of the chemical bond with the effect of UV lights induces irreparable damages, which causes the whitening of the surface (Fabiya and McDonald 2010). Kiguchi et al. (2007) stated that lightness positively relates to the increasing wood flour content. The highest increase in the lightness was obtained from WPC containing 30% of wood flour while the lowest was WPC containing 10%. The increase in the lightness proceeded up to 30% wood flour content. After that, the lightness decreased with an increasing wood flour content. The possible explanation for decreasing lightness with increasing wood flour content is that the crack formation on the surface due to degradation reveals the new surface of inner wood parts with dark color (Yang et al. 2015). WPC with a high wood flour content are prone to crack formation due to weak adhesion than high polymer content (Kaymakci et al. 2016).

$\Delta a$  value gives information about the color changes of the surface from greenish to reddish. As seen in Fig. 1, the trend for WPC samples was greenish, except for neat HDPE and WPC containing 10% of wood flour. The degradation of lignin during the weathering process decreases  $\Delta a$  values (Badji et al. 2017). Therefore, the increased wood flour content caused to occur the greenish surface. The highest greenish surface was determined on the surface of WPC containing 30% of wood flour, while the lowest one was neat HDPE.  $\Delta b$  value gives information about the color changes of the surface from bluish to yellowish. Paraquinone chromophoric groups, which tend to turn the WPCs' surface yellow, form due to lignin oxidation during the weathering process. However, paraquinone is reduced to hydroquinone with increasing exposure time, which prevents a yellowish surface (Muasher and Sain 2006). Therefore, all of the samples tended to be bluish surfaces. The intensity of the degradation increased with the increasing exposure time. The lowest change was determined in the neat HDPE. Moreover, WPC containing 10% wood flour content has a similar trend. Both of these groups were least affected by UV radiations. However, the increased wood flour content up to 50% increased the bluish surface.

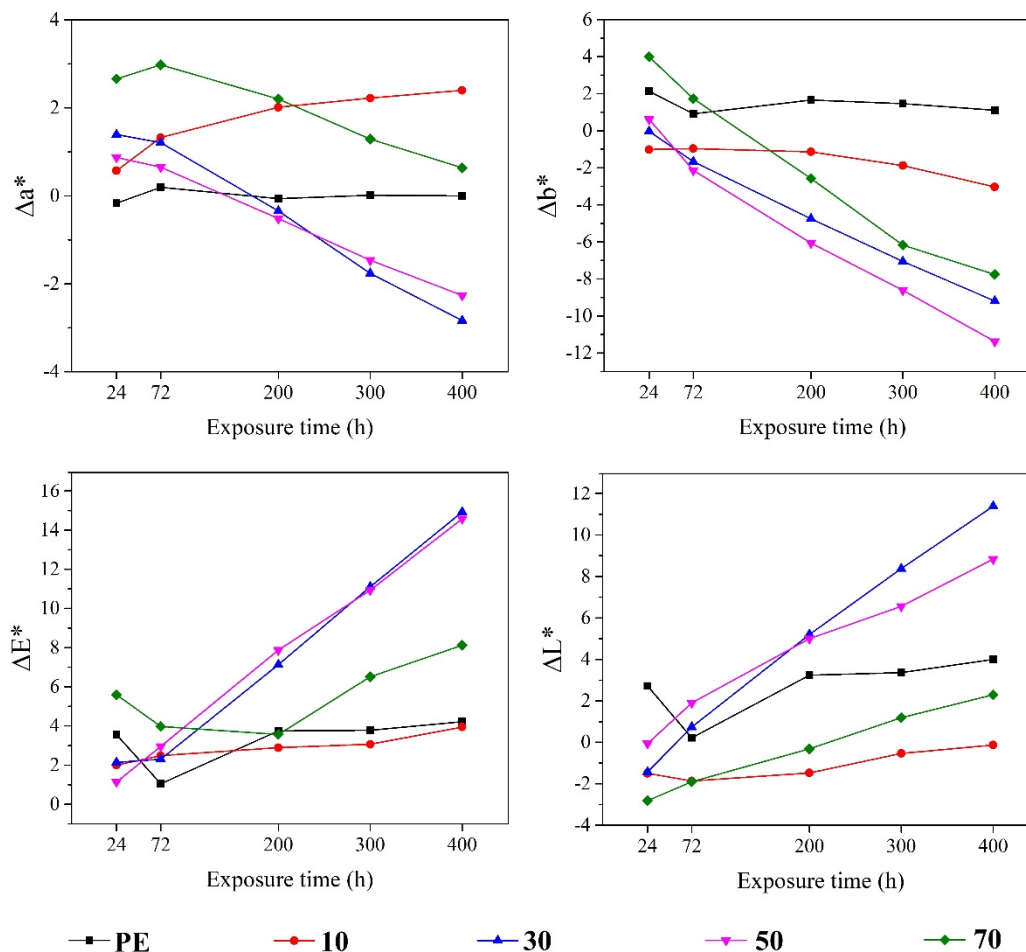


Fig. 1: The change in the color on the surface of WPC.

### ATR-FTIR Analysis

The changes in the chemical structure on the WPC surface were examined by ATR-FTIR analysis, as seen in Fig. 2. As explained in detail above, the change on the surface of WPC exposed to weathering conditions is inevitable. The main interest in this study is to reveal the effect of wood flour content in this aspect. The bands at  $2914\text{ cm}^{-1}$  and  $2846\text{ cm}^{-1}$  assigned to the C-H stretch in polymer chemical structure were intense (Kaczmarek et al. 2005, Turku and Kärki 2016). The intensity of these bands increased due to the photodegradation after weathering. As the wood content increases, the polymer separation from the surface of wood fiber is easier with the effect of UV lights. Therefore, the bands' intensity decreased for WPCs containing 30% and 50% of wood flour. Wei et al. (2021) also stated that the increase in the wood content paves the way for the scission and oxidation reaction in HDPE chemical structure. Moreover, the light microscopy images also supported this situation. The band at  $1743\text{ cm}^{-1}$  in the carbonyl regions indicates a carboxylic acid related to MAPE, showing the ester bonds between wood cell wall components and maleic anhydride groups (Wei et al. 2021). The ester concentration increased due to photodegradation, showing the polymer structures' scission reaction. Therefore, this band increased during the weathering, indicating UV absorption and photodegradation. However, the most notable increase was recorded for WPC containing 10%

of wood flour, which WPC also changing the least color. The shorter molecules resulting from chain scission might restrict the oxygen contact, which reduces photooxidation, resulting in the slightest color change (Muasher and Sain 2006).

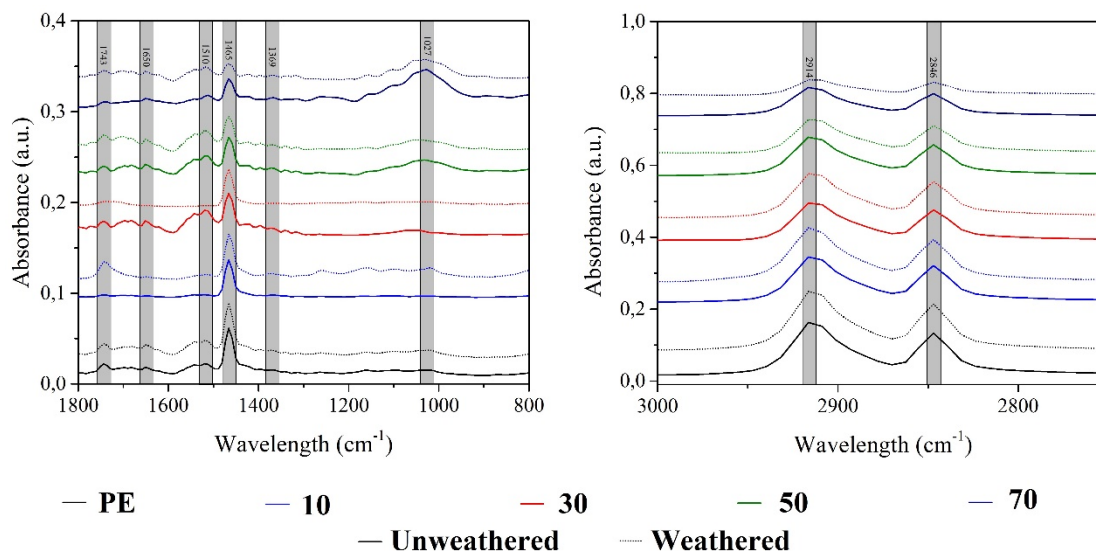


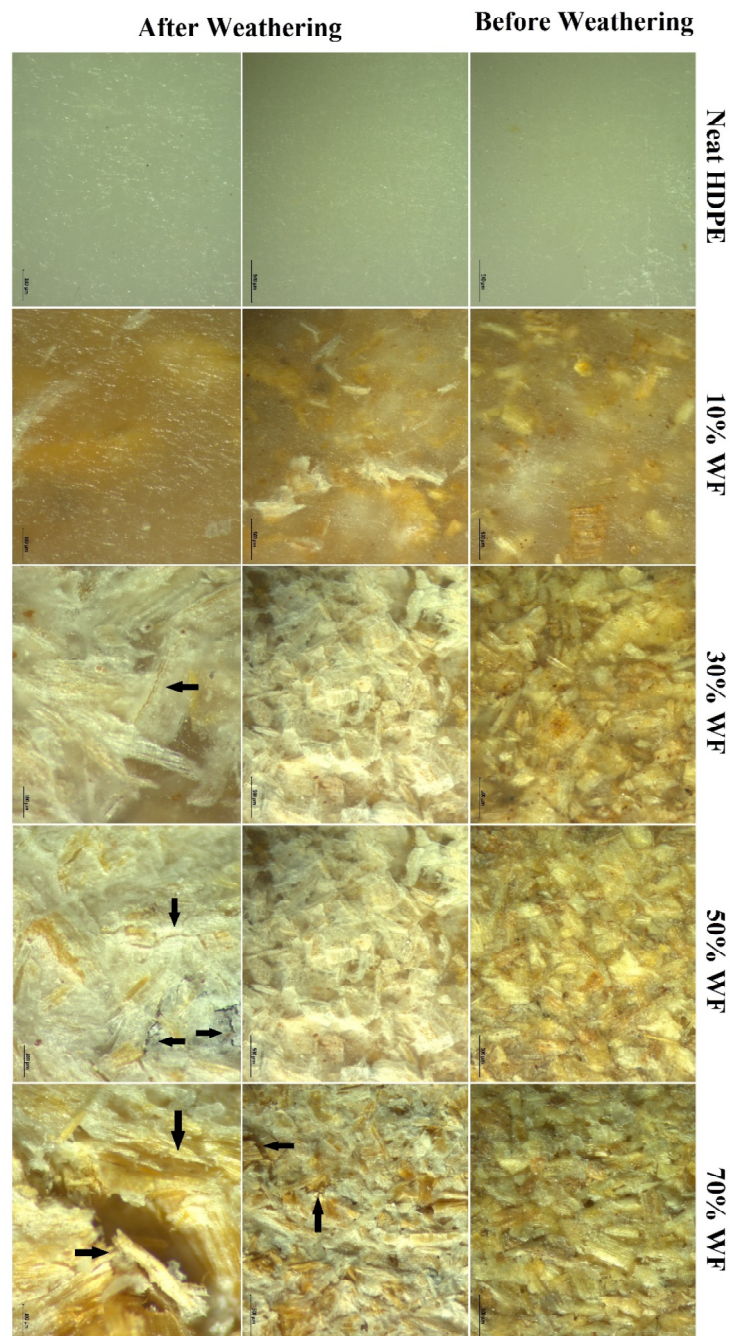
Fig. 2: ATR-FTIR spectra of WPC.

The  $1510\text{ cm}^{-1}$  band, assigned to the characteristic lignin peak, decreased during the weathering exposure (Pandey 1999). The photooxidation of lignin produces water-soluble products during the weathering process (Badji et al. 2017). The extensive weathering conditions resulted in the separation of wooden particles from the structure, which caused to decrease in the intensity of the lignin-related peak (Volkmer et al. 2013). The band's intensity at  $1650\text{ cm}^{-1}$  increased as a result of photodegradation for all groups, except WPC containing 30% of wood flour. The photooxidation resulted in an increase in vinyl groups in the polymer structure. Moreover, the degradation of lignin, which is the responsible component for absorbing UV radiation for wood fiber (Liu et al. 2018), causing reveal more cellulose and hemicellulose components, which are also responsible for whitening of surface as well as increasing the intensity of this band (Kiguchi et al. 2007). The band at  $1465\text{ cm}^{-1}$  is related to the amorphous structure of polyethylene (Nguyen et al. 2021). The increase in the intensity of this band was observed in WPC having high polymer content. On the other hand, this band decreased for WPC containing 50% and 70% WF. The scission reactions lead to shorter molecules due to the degradation of the HDPE (Muasher and Sain 2006), which increased the intensity of related bands for WPCs having high polymer content while it was a decrease for high wood content. The changes in the absorbance intensity of bands at  $1369\text{ cm}^{-1}$  and  $1027\text{ cm}^{-1}$  indicate cellulose and hemicellulose degradation (Mburu et al. 2008, Mohebbi 2005). The changes were more remarkable for WPC having high wood content.

### Light microscopy

The surface of WPC exposed to artificial weathering was examined by light microscopy, as seen in Fig. 3. The effects of UV radiation were seen on the surface of WPC after weathering

test. The change in the wood flour content increased the effect of the intensity of artificial weathering.



*Fig. 3: The light microscopy images of WPCs. Black arrows show crack formation.*

As shown in Fig. 3, there were almost no changes on the surface of neat-HDPE and WPC containing 10% of wood flour, except for increased surface roughness. However, the increased wood flour content negatively affected WPCs' surface character. The scission reactions occur in the polymer structure, which triggers the formation of cracks (Fabiya and McDonald 2008). Moreover, the decreasing polymer content brings wood fiber into the forefront, which increases the crack formation on the surface of WPC. Black arrows show the cracks occurred on



the surface after weathering exposure. The increase in the humidity also enhanced the intensity of degradation. Due to weathering conditions (UV radiation and condensation), the cracks expanded with increasing wood flour content (Yang et al. 2015). Meanwhile, WPC containing 70% wood flour content was evidence of weak binding and splitting of fibers, which negatively affected the usage of WPC. Moreover, the increase in the wood flour content whitened the surface of the samples. As explained, the interaction with the UV lights of wood fiber influenced the color changes more than polymer.

### Visual appearance

The visual appearance of WPC is shown in Fig. 4. The degradation after artificial weathering was shown in its entirety. The color change showed up clearly with increasing exposure time. The glossiness left its place to matteness with exposure time. The surfaces of WPC were grayish as wood flour content increased. However, a remarkable amount of grayish was shown in WPC containing 30% to 50% of wood flour. After that content, the graying decreased and initiated to turn red. Meantime, the changes in the color on the surface of neat HDPE were more stable than WPC. Likewise, the color stability was better for WPC containing 10% of wood flour. The yellowish surface increased during the 200h, after this time it decreased relatively. The increasing wood content damages the structural integrity of WPC. The decrease in the polymer content weakens adhesion between wood and polymer. The swelling of wood fibers during the weathering test breaks the polymer encapsulation, which causes the wood fibers to expose to UV light (Fabiya and McDonald 2010). Consequently, color changes are inevitable, and the grayish surface results from the weathering process. Besides this, the erosion on the surface is much more prominent for WPC with lower polymer content. Wood flour also caused irregularities in the structure of WPC. Moreover, the degradation of wood particles and polymer from the surface increased the irregularities, which resulted in increased surface roughness (Badji et al. 2017).

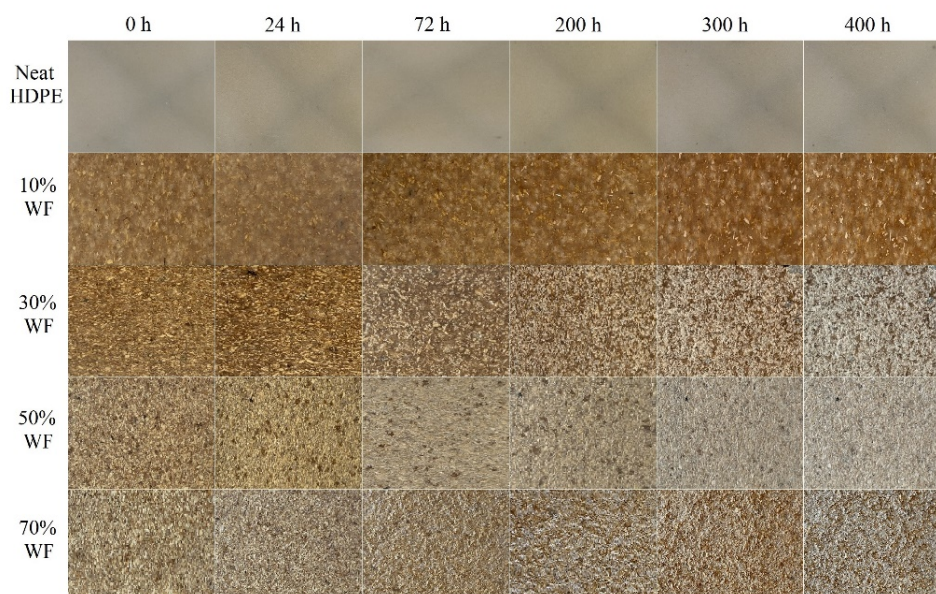


Fig. 4: The visual appearance of WPC during the artificial weathering.

## CONCLUSIONS

In this study, the effect of wood content on the artificial weathering performance of flat pressed WPC was investigated by some physical, analytical, and microscopic tests. The color change as the main problem for WPC is inevitable during UV exposure. The surface character of WPC rapidly changed during the 400 h of the artificial weathering test. The whitening also revealed itself with increasing exposure time. Not only did color change, but the cracks were also observed after the weathering test. The increase in the wood content weakens the adhesion between wood and polymer, facilitating cracking formations. Therefore, the crack formation was more pronounced with the increase in the wood content. The cavity and hills are accompanied by surface degradation. The surface roughness increased with increasing wood content as a result of photodegradation. As a consequence, the surface character worsened with increasing wood content.

The chemical structure of the surface also changes during the weathering. The changes in the band's intensity at  $1510\text{ cm}^{-1}$  assigned to the characteristic lignin peak indicated the photooxidation. Lignin oxidation is one of the causes of color changes. The degradation in the amorphous structure of polyethylene was also detected with changes in the band's intensity at  $1465\text{ cm}^{-1}$ . The shorter molecule formations resulting from the chain scission reactions facilitate polymer layer separation in WPCs with high wood content, also shown in light microscopy images. According to these results, the increase in the wood flour content complicates the process and results in troubles in the usage areas.

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SEFA DURMAZ\*  
MUGLA SITKI KOÇMAN UNIVERSITY  
KAVAKLIDERE VOCATIONAL SCHOOL  
DEPARTMENT OF FORESTRY AND FOREST PRODUCTS  
MUGLA, 48500  
TURKEY

\*Corresponding author: sefadurmaz@mu.edu.tr

ÖZLEM ÖZGENÇ  
KARADENİZ TECHNICAL UNIVERSITY  
FACULTY OF FORESTRY  
DEPARTMENT OF FOREST INDUSTRY ENGINEERING  
TRABZON, 61080  
TURKEY