Weathering Characteristics of Wood-plastic Composites Compatibilized with Ethylene Vinyl Acetate

Xiaohong Hu,[a,b] Dongfang Li,[b] Bin Luo,[b,*] and Li Li [b,*]

Poplar wood flour wood-plastic composites (WPCs) prepared with high-density polyethylene and modified with different amounts of ethylene vinyl acetate (EVA) were created. The influence of EVA content on the surface properties and weathering characteristics after ultraviolet (UV) treatment was investigated. The results showed that WPCs treated with EVA had improved thermal stability, and the surface polarity was reduced. The addition of EVA slowed the fading of the WPCs, and the color difference was the smallest when the EVA content was 5%. Both the bending strength and the modulus of elasticity (MOE) of the WPCs decreased after the UV treatment. However, when the EVA content was 5%, the bending strength retention ratio and MOE retention ratio were greatest. Scanning electron microscopy showed that the addition of EVA reduced the amount of pores and gaps on the fractured surfaces of the WPCs. The comprehensive performance of the WPC anti-weathering properties was greatest when the EVA level was 5%.

Keywords: Wood plastic composites; Ethylene vinyl acetate; Weathering characteristics

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INTRODUCTION

Wood-plastic composites (WPCs) are an environmentally friendly material that is prepared mainly with wood flour and plastic under certain technological conditions. They are mainly used in outdoor applications such as decks, guardrails, garden landscapes, and building materials (Friedrich and Luible 2016; Friedrich 2019). Due to their characteristics of both wood and plastic, WPCs have a series of unique advantages, such as resistance to moth attack, corrosion resistance, and low deformation (Li *et al*. 2011; Lazrak *et al*. 2019).

However, traditional WPCs have the problems of low mechanical strength and poor interfacial compatibility. This behavior is because the wood fibers in the composite have strong surface chemical polarity and hydrophilicity, while the plastic matrix is mostly non-polar (or has low surface polarity) and hydrophobic (Bouafif *et al*. 2009; Li *et al*. 2011; Ghahri *et al*. 2012; Turku *et al*. 2017; Ferreira *et al*. 2019). Furthermore, the outdoor use conditions of WPCs should not be ignored. Wood-plastic composites suffer from degradation of mechanical properties and surface color fading when used outdoors, and their poor anti-weathering properties are among the key issues restricting their further development and use (Yang *et al*. 2015; Badji *et al*. 2017).

Over the years, many studies have sought improved coupling agents and compatibilizing agents to enhance the interfacial compatibility (Ratanawilai and Taneerat 2018). Through the continuous efforts of researchers and enterprises, the compatibility problem of wood-plastic composites has been solved to a certain extent, but the mechanical

strength is still far from meeting the requirements of building materials, which still needs further in-depth discussion and research. Besides, from the processing point of view, the increase of wood powder content can reduce the production cost of wood-plastic composites. However, when the content of wood powder is too high, it will cause difficulties in feeding and extrusion, which limited the preparation of wood-plastic composites with high content of wood powder.

EVA is a polar copolymer: One end contains ester groups that can react with the polar groups of the wood powder, and the other end contains vinyl groups that are compatible with resin or nonpolar plastic matrix materials and can theoretically be used as a compatibilizing agent. Furthermore, EVA has good aging resistance, flexibility, and fluidity, which theoretically are beneficial to the anti-weathering properties and extrusion molding process of WPCs. If these theories can be validated, then the adding of EVA can be beneficial to the compatibility between wood fiber and plastic, the extrusion molding process, and the weathering characteristics. This will greatly improve the efficiency of additive use, and it is also of great significance for the research and development of better wood-plastic composite formula and the preparation of high-performance materials.

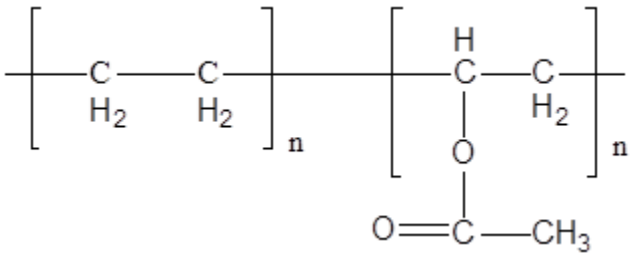

Fig. 1. The molecular structure of EVA

There have been some reports that have demonstrated the possibility of applying EVA to WPCs, but most of them used EVA as a plastic matrix or were focused on the mechanical properties of WPCs that had been supplemented by EVA (Li *et al.* 2010; Zimmermann *et al*. 2014). There has been little research on its impact on the extrusion molding process and anti-weathering performance.

This study used EVA as a compatibilizing agent to improve WPCs' anti-weathering properties. A variety of testing methods were used to evaluate the influence of EVA content on the aging properties and surface properties of the WPCs. Detailed studies were performed to analyze the mechanism of the effect.

EXPERIMENTAL

Materials

Table 1 shows the materials used in making the WPCs. The ratio of wood flour to plastic was 7:3. Relatively large contents of EVA were used in the composites, so to avoid increases in the proportion of plastic due increasing EVA, the contents of the HDPE and EVA were regarded as a whole (*i.e.*, wood flour : (HDPE + EVA) = 7 : 3). The amounts of materials used in making the WPCs are given in Table 2.

Table 1. Materials Used in Making the WPCs

Material	Melt Index	Mesh	Manufacturer
High-density polyethylene (HDPE), grade 5000S	0.9 g / 10 min	-	Sinopec Yanshan Petrochemical Company (Yanshan, China)
Poplar wood flour	-	100	Xingtai Jinye Wood Flour Factory (Xingtai, China)
EVA, grade 40W (40% vinyl acetate content)	65 g / 10 min at 58 °C	-	Mitsui Petrochemical Industries, Ltd. (Tokyo, Japan)
Paraffin wax (melting point of 54 °C to 56 °C)	-	-	Sinopec Yanshan Petrochemical Company (Yanshan, China)
Calcium carbonate	-	800	Yufeng Company (Shijiazhuang, Hebei Province, China)

Table 2. Material Amounts Used in Making the WPCs

EVA Content (%)	Poplar Wood Flour (g)	EVA (g)	HDPE (g)	Paraffin Wax (g)	Calcium Carbonate (g)
0%	3500	0	1500	105	175
2.5%	3500	87.5	1412.5	105	175
5%	3500	175	1325	105	175
7.5%	3500	262.5	1237.5	105	175
10%	3500	350	1150	105	175
12.5%	3500	437.5	1062.5	105	175
15%	3500	525	975	105	175
20%	3500	700	800	105	175

WPC Processing

The poplar wood flour was initially dried in an oven at 120 °C for 3 h before mixing. To modify the poplar wood flour, seven different amounts of EVA were mixed with the poplar wood flour for 40 min in a high-speed mixer (SHL-100A, Nanjing Haisi Extrusion Equipment Co., Ltd., Nanjing, China). Fourier-transform infrared spectroscopy (FTIR) and thermogravimetric analysis (TGA) were performed on the modified wood flour.

Next, the modified wood flour was mixed with HDPE and other additives in a high-speed mixer for 3 min. After that, the mixtures were first granulated using a single-screw extruder (SJZ35, Shanghai Jwell Machinery Co., Ltd., Shanghai, China). The temperatures of the four zones in the extruder were 160 °C, 170 °C, 160 °C, and 160 °C. The mixtures were then made into WPCs using a twin-screw extruder (SJZ45/100, Shanghai Jwell Machinery Co., Ltd., Shanghai, China). The temperatures of the four zones in the extruder were 165 °C, 170 °C, 165 °C, and 165 °C, while the temperature of the mold was 165 °C. The screw speed was 4 r/min.

FTIR

Fourier-transform infrared spectroscopy was used to examine the changes of the wood flour's surface groups after the EVA treatment. The samples were dried before scanning to reduce the influence of the hydroxyl of the wood flour. Then, the dried samples were measured in a KBr pellet with an infrared spectrometer (Spectrum-100D, PerkinElmer, Fremont, CA, USA).

TGA

The thermogravimetric properties of the poplar wood flour (untreated and treated with EVA) were analyzed using a thermogravimetric analyzer (Shimadzu TGA-600, Kyoto, Japan). The test temperature was ranged from 40 °C to 840 °C with a heating rate of 10 °C/min. To prevent oxidation of the wood flour, the experiment was performed in a nitrogen environment (20 mL/min).

Contact Angle Test

The contact angles of the WPCs with different EVA contents were measured using distilled water and glycerin with a contact angle measurement instrument (DCA-20, Orientec, Tokyo, Japan). The Owens-Wendt-Rabel-Kaelble (OWRK) method was used to calculate the surface free energy of the WPCs. The influence of the EVA content on the contact angles and surface free energies of the WPCs was analyzed.

Ultraviolet (UV) Aging Treatment

Wood-plastic composite samples with different EVA contents were treated under UV light to accelerate aging. Four sets of tubes with a wavelength of 365 nm were used, and the temperature was set at 50 °C. Samples were taken out every 25 h for color testing. All samples were taken out for mechanical and microscopic properties testing when the aging treatment time reached 125 h.

Color Change Analysis

The surface color changes of the samples at different aging times were tested with a spectrophotometer (DataFlash DF110, Datacolor, Lawrenceville, NJ, USA). Five samples of each type of WPC (with different EVA contents) were tested, and five points were tested on each sample. The final result for a given EVA content was the average value of the 25 points.

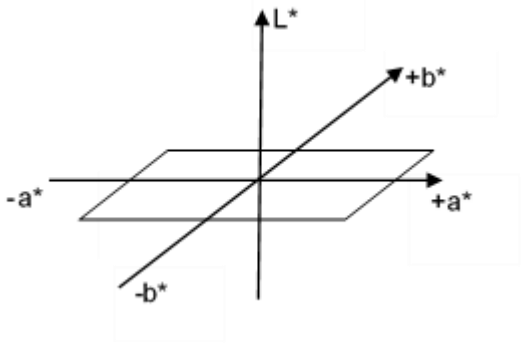

Fig. 2. Model of *L*a*b** color space

The international lighting standard CIE (1976) *L*a*b** (ISO 11664-4 2008) was used to quantitatively analyze the discoloration of the WPCs with aging time and the effect of EVA content on the discoloration (Eq. 1),

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

where ΔE is the overall color difference, L^* is the lightness ($+\Delta L$ represents whitening, and

-Δ*L* indicates the color is turning black), *a** is the green–red color component (+Δ*a* represents the color turning red, and -Δ*a* indicates the color is turning green), and *b** is the blue–yellow color component (+Δ*b* represents the color turning yellow, and -Δ*b* indicates the color is turning blue).

Flexural Properties Change Test

The flexural properties of the WPCs before and after the UV treatment were measured by an electronic universal testing machine (WDW-50D, Sunpoc, Guiyang, China) according to GB/T 1449 (2005). The pressure head acted on the back side of the aged surface, so the aged surface was stretched in the bending process. The speed of the pressure head was 10 mm/min. The test specimen dimensions were 200 mm × 15 mm × 10 mm. Ten specimens were tested to obtain the average value for each sample. Bending strength was calculated according to Eq. 2,

$$\sigma_f = \frac{3P \cdot l}{2b \cdot h^2} \quad (2)$$

where σ_f is the bending strength of the sample (MPa), P is the maximum load of the sample (N), b is the width of the sample (mm), l is the span length of the machine (mm), and h is the thickness of the sample (mm).

The modulus of elasticity (MOE) was calculated according to Eq. 3,

$$E_f = \frac{l^3 \cdot \Delta P}{4b \cdot h^3 \cdot \Delta S} \quad (3)$$

where E_f is the MOE of the sample (MPa), ΔP is the increment of the load in the initial straight segment on the load-deflection curve (N), ΔS is the increment of the deflection in the same initial straight segment (mm), b is the width of the sample (mm), l is the span length of the machine (mm), and h is the thickness of the sample (mm).

The bending strength retention ratio was calculated according to Eq. 4,

$$B = (1 - (P_1 - P_2) / P_1) \times 100 \quad (4)$$

where B is the bending strength retention ratio (%), P_1 is the bending strength before the aging process (N), and P_2 is the bending strength after the aging process (N). The MOE retention ratio was calculated in the same way.

Morphology Study

The morphologies of fracture surfaces were examined using a scanning electron microscope (S-3400N II, Hitachi, Tokyo, Japan) with an acceleration voltage of 15 kV. Samples were mounted on conductive adhesive tape and sputter coated with gold before observation.

RESULTS AND DISCUSSION

FTIR Analysis

Wood flour is composed of cellulose, hemicellulose, lignin, and various wood extractives. Its surface contains many polar groups, such as phenolic hydroxyl groups and hydroxyl groups, resulting in a strong chemical polarity on the surfaces of wood fibers. The main purpose of modifying wood fibers is to reduce the chemical polarity of their

surfaces (Peng *et al*. 2015).

Curve 4 (EVA-15%) in Fig. 3(b) gives an example of treated wood flour, to compare with untreated wood flour and explain what occurred. The absorption peak at 1774 cm^{-1} disappeared, and an absorption peak appeared at 1059 cm^{-1}, indicating that the carbonyl groups disappeared, and ester groups appeared. This result indicated that the adding of EVA has decreased the surface polarity of the wood fiber. This could be a result of EVA coating onto the surface of the wood fiber.

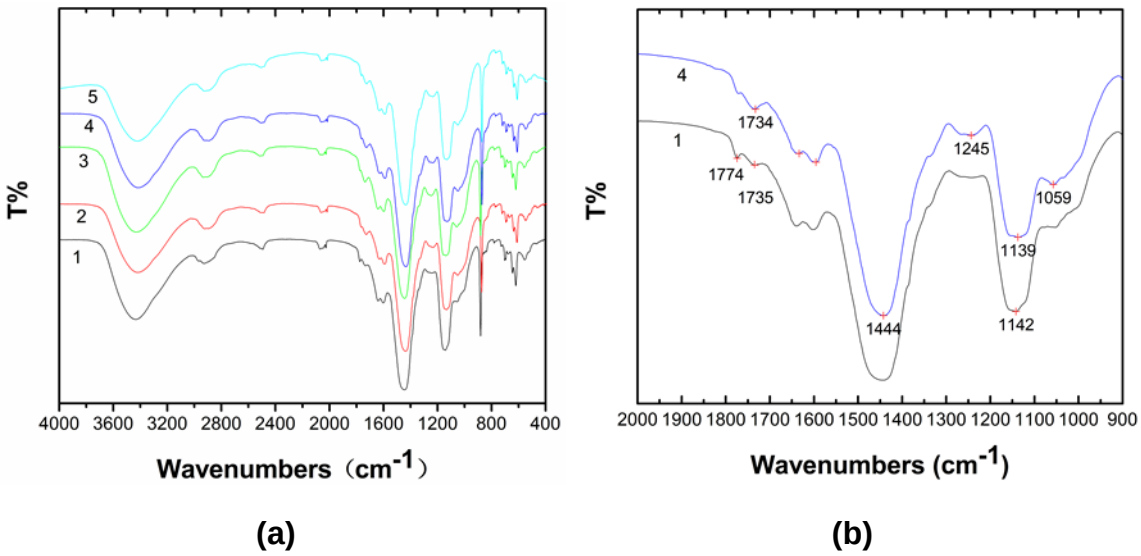

Fig. 3. (a) FTIR spectra (4000 cm^{-1} to 400 cm^{-1}) for different EVA contents of wood flour: 1) 0% EVA, 2) 5% EVA, 3) 10% EVA, 4) 15% EVA, and 5) 20% EVA; (b) FTIR spectra (2000 cm^{-1} to 900 cm^{-1}) for wood flour with 1) 0% EVA and 4) 15% EVA

Thermal Properties

As shown in Fig. 4, the thermal degradation of the unmodified poplar wood flour had four stages.

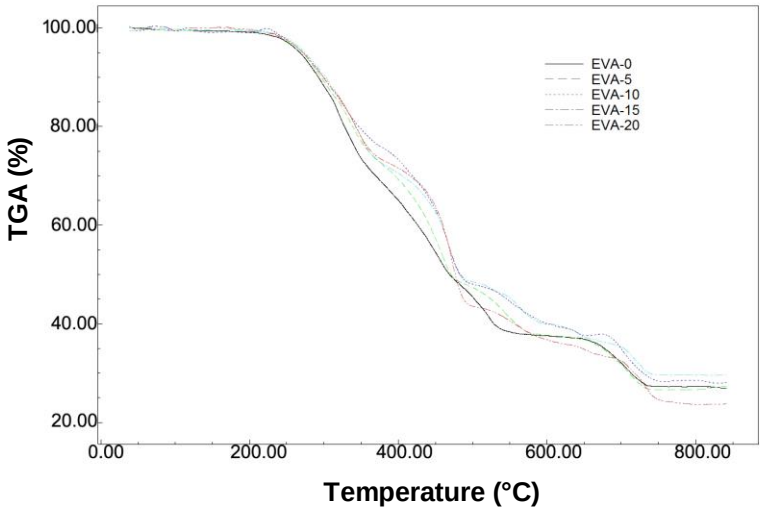

Fig. 4. Effect of EVA content on thermal behavior of the poplar wood flour

The stages were evaporation of water in the wood flour; thermal degradation of hemicellulose, cellulose, and lignin; degradation of other components of the wood flour; and degradation of the wood flour.

Compared with the unmodified wood flour, the modified wood flour had five stages of thermal degradation; a stage of EVA degradation was added. At temperatures less than 200 °C, the curves of the modified and unmodified wood flours had no apparent difference. Between 200 °C and 350 °C, hemicellulose, cellulose, and lignin were degraded in turn; and in this stage, the mass loss of the EVA-modified poplar flour was noticeably less than that of the unmodified wood flour. Moreover, the initial degradation temperature of the EVA-modified wood flour was greater than that of the unmodified wood flour. It was speculated that the chemical reaction of the modified poplar flour may have improved the thermal stability. Furthermore, the addition of EVA could improve the remaining amount of mass.

Contact Angle

As shown in Fig. 5, when the detecting liquids were distilled water and glycerol, the addition of EVA decreased the contact angles. The contact angle continued decreasing with increasing EVA content, indicating that the addition of EVA increased the surface wettability of the WPCs. This result will be beneficial for coating and glue spreading in the manufacture of WPCs.

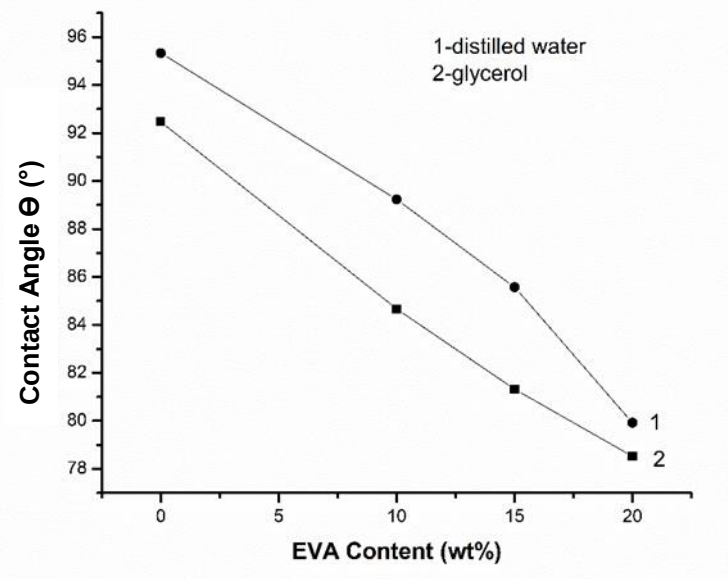

Fig. 5. Effect of EVA content on the contact angles of the WPCs

Surface Free Energy and Surface Polarity

According to Zisman's critical surface free energy theory, when fluid comes into contact with a solid surface to form a new interface, the condition for its stability and mutual fusion is that the surface free energy of the fluid is less than or equal to the surface free energy of the solid. When the wood fiber is in contact with the plastic surface, an increase in the surface free energy of the wood fiber increases the likelihood of the plastic to infiltrate into the wood fiber. As shown in Table 3, with the addition of EVA, the dispersion (non-polar) component of free energy did not change to an important degree; however, the polar component of free energy increased with increasing content of EVA.

This indicates that the surface wettability between the plastic and the wood fiber had increased. The plastic infiltrated into the wood fiber more thoroughly, which increased the interfacial fusion performance and the overall performance of the WPC. Additionally, the increase of the surface free energy would be beneficial for the spreading of glue on the WPC surface.

Table 3. Effect of EVA Content on the Surface Energy and Surface Polarity of WPCs

EVA	SE (mJ/㎡)	*D* (mJ/㎡)	*P* (mJ/㎡)
0%	34.89	34.25	0.64
10%	36.06	36.06	1.54
15%	36.99	36.99	2.29
20%	36.94	36.94	4.06

SE – surface energy; *D* – Dispersion component of surface free energy; *P* – Polar component of surface free energy

Color Change after Aging Treatment

After 25 h of sample aging, the value of ΔE was 0.61, and there was an obvious discoloration (Fig. 6). As the UV treatment time increased, the value of ΔE gradually increased. The changes of ΔL and Δb were similar to that of ΔE, which increased with increasing aging time. This result suggested that the discoloration in the WPC was mainly due to its turning white and yellow. However, the value of Δa did not change much with the aging time, indicating that it was not the main factor affecting the value of ΔE.

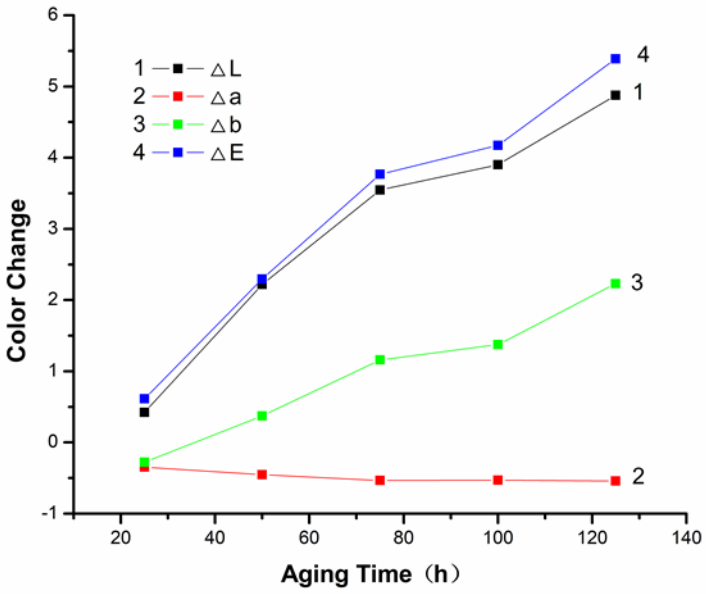

Fig. 6. Surface color changes of WPCs without EVA during the weathering process

The aging of WPCs results from complex changes of different components, including the degradation of cellulose, hemicellulose, and lignin in the wood fiber and the degradation of plastic and EVA. Furthermore, synergistic effects result from the interfacial compatibility damage caused by the degradation of components, stress concentration, and cracking inside the material.

After the UV aging treatment, chromophoric groups such as para-quinone could be produced as a result of oxidation reactions, as lignin is highly sensitive to UV light, heat, and oxygen, which caused discoloration. Additionally, the molecular chain of HDPE was broken during the aging process due to photodegradation. In addition, degraded HDPE chains having reduced molecular mass migrated to the surface, causing a certain degree of discoloration. Moreover, HDPE mainly forms para-quinone after photodegradation, followed by degradation into hydroquinone, which has a photobleaching effect and causes the discoloration of WPCs (Matuana *et al*. 2011; May-Pat *et al*. 2013). Finally, lignin will generate chromophoric functional groups such as carboxylic acids, quinone, and peroxy hydroxyl species after photodegradation, which make WPCs appear yellow (Kiguchi *et al*. 2007; Stark and Matuana 2007; Fabiyi *et al*. 2008; Butylina *et al*. 2012; Badji *et al*. 2017; Yáñez-Pacios and Martín-Martínez 2018).

As shown in Fig. 7, compared with curve 1 (EVA content of 0%), the overall changes of other curves (added EVA) were relatively gentle. This result indicates that the addition of EVA reduced the fading and increased the aging resistance of the WPCs. Among them, curves 2, 3, and 4 (with EVA contents of 2.5%, 5%, and 7.5%) were similar to curve 1. As the aging time increased, ΔE gradually increased. However, curves 5, 6, 7, and 8 (with EVA contents of 10%, 12.5%, 15%, and 20%) showed different behavior. After 25 h of aging, there was a severe discoloring, but between 25 h and 75 h, ΔE gradually decreased. This result indicated that the colors of the WPCs deepened during this period of time, which could be due to the aging of EVA. Between 75 h and 125 h, ΔE increased again gradually, and the colors of the WPCs continued to fade.

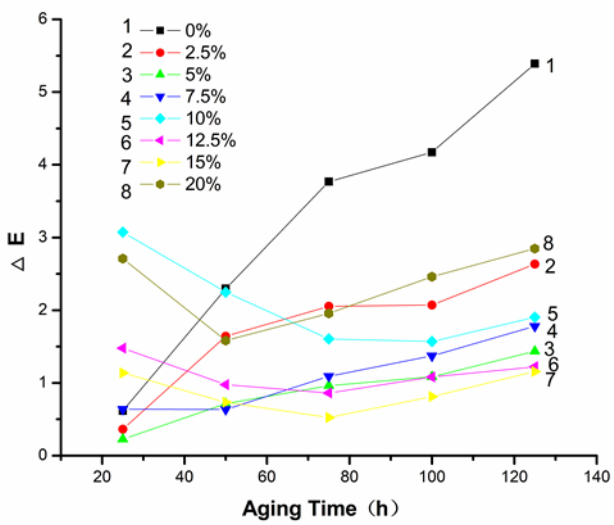

Fig. 7. Degrees of fading of WPCs with different EVA contents during the weathering process

The addition of EVA reduced the discoloration of the WPCs. Ethylene vinyl acetate has good qualities of optical transparency and flexibility. With the addition of the EVA, the compatibility between the wood fiber and the plastic matrix was increased. The flowability formed a better transition layer between the wood fiber and the plastic, which enabled the plastic to form a better covering layer on the surface of the wood fiber. The degradation of lignin in the wood materials was reduced, as was the generation of color functional groups such as carboxylic acid, quinone, and peroxy hydroxyl species.

When the EVA content was greater than 10%, the WPCs' anti-aging performances

decreased, which might be because EVA has high optical transparency. When more of the HDPE was replaced by EVA, there was more EVA on the surface of the wood fiber. Its high optical transparency transmitted more of the UV radiation to the wood fiber, which caused a more severe degradation of cellulose and hemicellulose. More cracks appeared, and the interfacial combination between the wood fiber and plastic was also destroyed.

As shown in Fig. 8, as EVA content increased, ΔE first decreased and then increased. When EVA was not added, after 125 h of the aging treatment, ΔE reached 5.39. With the addition of EVA, the discoloration of the WPCs decreased sharply. When the EVA content was 5%, ΔE value decreased to 1.44, a decrease of 73.3%. As shown, the addition of a small amount of EVA greatly decreased the discoloration of the WPCs. As the EVA content continued to increase, ΔE presented a small range of fluctuation at first and then increased when the EVA content exceeded 15%.

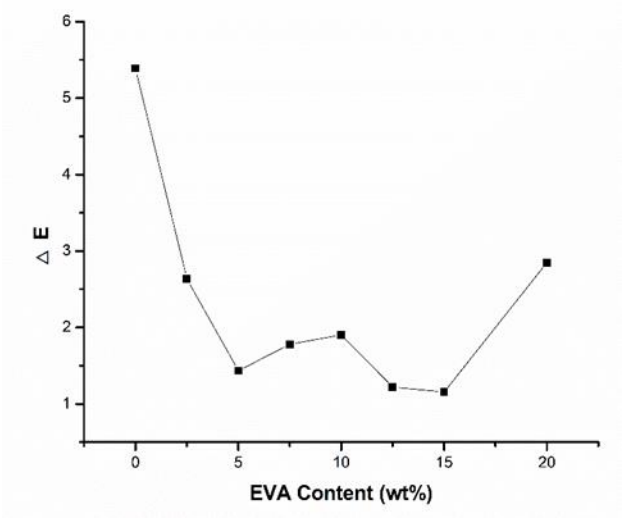

Fig. 8. Effect of EVA content on the degree of fading of WPCs after 125 h of UV treatment

Overall, the addition of EVA greatly decreased the discoloring of the WPCs, achieving a notable effect when the EVA content was 5%. As the EVA content continued to increase, the improvement decreased.

Flexural Properties after Aging Treatment

Figure 9 shows that, after the aging treatment, the bending strengths and MOE values of the WPCs decreased by different degrees. As shown in Fig. 9a, when the EVA content was between 2.5% and 5%, the bending strength retention percentage of WPCs increased and reached 98.99% when the EVA content was at 5%.

This behavior was attributed to the addition of EVA, which appeared to enhance the interfacial connection performance between the wood fiber and the HDPE. When the EVA content was between 5% and 10%, the bending strength retention rate noticeably decreased. When the EVA content was 12.5%, it increased slightly and then continued to decline. When the EVA content was 20%, the bending strength retention rate was only 76.6%.

The behavior of the MOE shown in Fig. 9b was similar to that of the bending strength. When the EVA content was at 20%, the retention percentage of MOE was only 54.8%, which was less than the retention percentage of bending strength. This result indicated that the aging had a greater influence on the MOE in the WPCs.

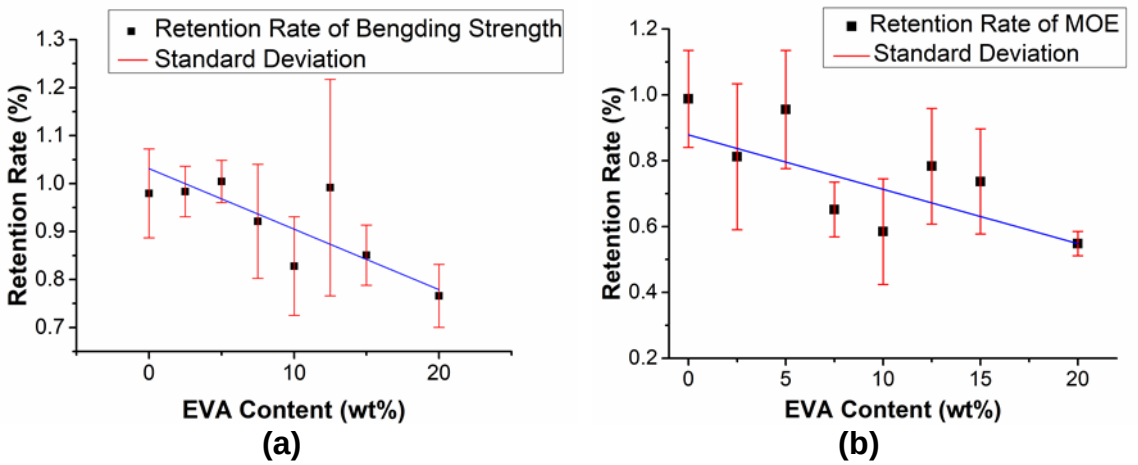

Fig. 9. Influence of EVA content on bending properties of WPCs after UV treatment

The mechanical properties of the WPCs decreased after UV irradiation. Studies have shown that this result is due to changes in crystallinity, surface oxidation, and hygroscopicity of materials during this process, which lead to interfacial separation. The aging of wood-plastic composites is accompanied by the degradation of all components. At first, the degradation of HDPE caused cracks inside the material, which reduced the stress transfer efficiency in the material, consequently decreasing the flexural properties of the composites (Yang *et al*. 2015; Vedrtnam *et al*. 2019). Meanwhile, the degradation also destroyed the compatibility between the wood fiber and the HDPE. Additionally, in the wood part, the degradation of lignin, cellulose, hemicellulose, and other components also decreased the overall mechanical properties of the material (Fabiyi *et al*. 2008; Soccalingame *et al*. 2016; Ferreira *et al*. 2019). Moreover, the destruction of the WPC material surface exposed more wood fiber to the air, facilitating water absorption and accelerating the aging of the material.

With the addition of EVA, the compatibility between the wood and the plastic matrix increased. More plastic was wrapped onto the surface of the wood fiber, which made the wood fiber more protected and reduced the oxidation points, thus reducing the loss of mechanical properties.

Morphology

As shown in Fig. 10, after the UV treatment, the interface of the WPC changed greatly. The surface of the material before aging was smoother, and more wood powder was wrapped with HDPE. After the aging treatment, the WPC surface showed (a) more slender features, exposed wood fibers, and (b) gaps, which might be caused by the fracture of plastics.

As shown in Fig. 11, a small amount of EVA could greatly improve the interfacial properties of the WPCs. The amount of pores and gaps on the fractured surfaces of the WPCs was reduced. When the EVA content was 5%, the surface was smooth, and the UV treatment did little harm to the material's surface. However, as the EVA content continued to increase, especially beyond 10%, the arrangement became disordered, and (b and d) more gaps and holes were visible. This result may be because excessive EVA increased the stickiness of the plastic, such that the EVA clumped together (item c in Fig. 11) rather than coating the wood fibers better.

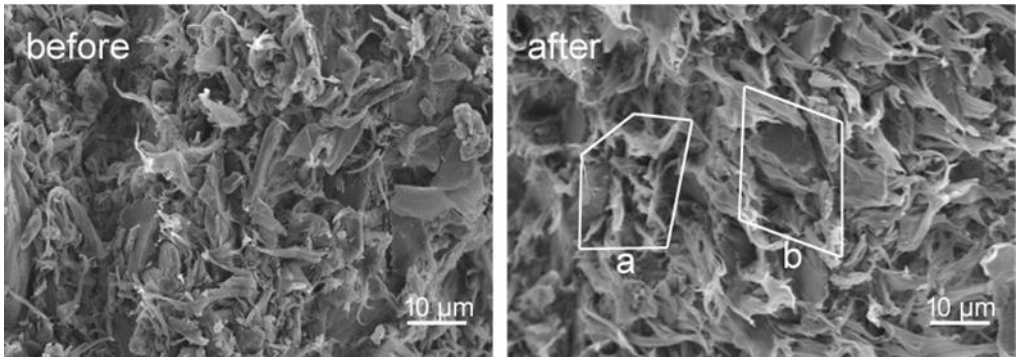

Fig. 10. Comparison of WPC sections before and after UV treatment

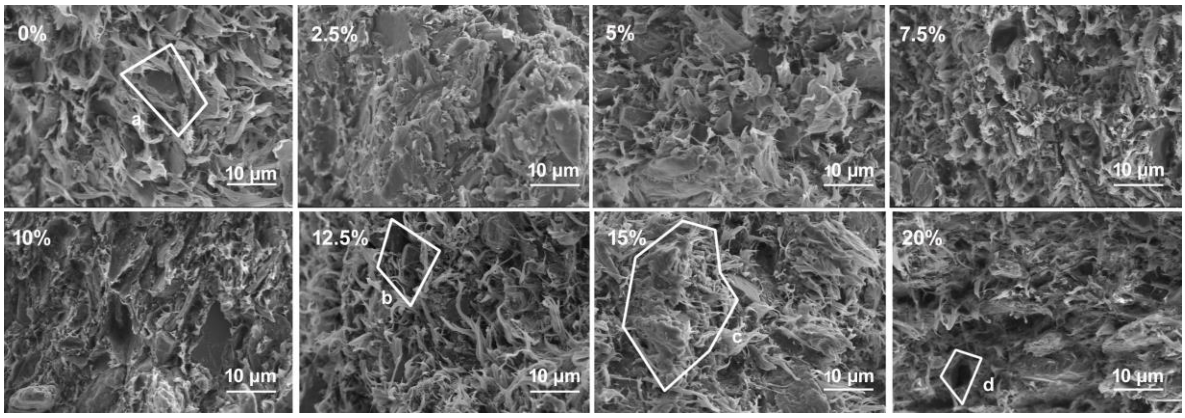

Fig. 11. Comparison of WPC sections with different EVA contents after the aging treatment

CONCLUSIONS

1. The addition of ethylene vinyl acetate (EVA) improved the interfacial properties between the poplar wood fiber and the high-density polyethylene (HDPE), resulting in better anti-weathering properties of wood-plastic composites (WPCs).
2. Fourier-transform infrared spectroscopy and thermogravimetric analysis (TGA) showed that poplar wood flour treated with EVA had improved thermal stability, and the surface polarity was reduced.
3. With increasing EVA content, the surface contact angle of the WPCs decreased, and the wettability of the WPCs and the surface free energy increased, which were beneficial for coating and gluing.
4. The WPCs mainly suffered from discoloring and mechanical loss after aging. The addition of EVA greatly slowed the fading of the WPCs, and the color difference was the smallest when the EVA content was 5%. Both the bending strength and the modulus of elasticity (MOE) of the WPCs decreased after the aging process. However, when the EVA content was 5%, the bending strength retention ratio and MOE retention ratio were greatest.
5. Scanning electron microscopy showed that the addition of EVA reduced the amount of pores and gaps on the fractured surfaces of the WPCs.

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