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EFFECT OF WOOD SPECIES ON UV WEATHERING RESISTANCE OF WOOD-PLASTIC COMPOSITES

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Keywords: Wood flour/polyethylene, *Eucalyptus urophylla*, *Acacia mangium*, *Pinus caribaea*, weathering, surface properties

Abstract

The goal of this study was to evaluate accelerated weathering performance of high-density polyethylene (HDPE) -wood flour (WF) composites. The wood flour was obtained from three different plantation species including *Eucalyptus urophylla*, *Acacia mangium*, and *Pinus caribaea*. The samples of WPC were exposed in a QUV accelerated weathering test for a total duration of 2000 h. The flexural strength, impact strength and surface color of the samples were tested. The weathered surface was characterized with Scanning electron microscopy (SEM) and Fourier transform infrared (FTIR) spectroscopy. The results indicate that: (1) the flexural strength and impact strength of the composites were decreased with increasing exposure time of weathering. The highest value of MOE, MOR and impact strength was seen in the *Acacia mangium*/HDPE composite; (2) The change of color and lightness of WPCs during weathering increased with increasing exposure time. The speed of change depends on wood species. WPC oxidation weather which is assessed by the concentration of carbonyl groups increased parallel with exposure duration. WPC filled with *Acacia mangium* wood flour had higher ΔE* and ΔL* values after weathering compared to those of filled with *Eucalyptus urophylla* and *Pinus caribaea*

Từ khóa: Bột gỗ/polyethylene, gỗ Bạch đàn urophylla, gỗ Keo tai tượng, gỗ Thông caribaea; thời tiết, tính chất bề mặt

Ảnh hưởng của loài gỗ đến khả năng chống chịu thời tiết của vật liệu gỗ nhựa

Mục tiêu của nghiên cứu này là đánh giá khả năng chống chịu gia tốc thời tiết của vật liệu tổng hợp nhựa khối lượng thể tích cao (HDPE) và bột gỗ (WF). Bột gỗ được lấy từ ba loài gỗ rừng trồng bao gồm: Bạch đàn urophylla, Keo tai tượng và Thông caribaea. Các mẫu thử được tiến hành thử nghiệm trên máy thử gia tốc thời tiết QUV trong khoảng thời gian là 2.000 giờ. Các chỉ tiêu đánh giá bao gồm độ bền uốn, độ bền va đập và độ biến màu bề mặt. Độ biến màu bề mặt được đánh giá bằng kính hiển vi điện tử (SEM) và quang phổ học hồng ngoại (FTIR). Kết quả chỉ ra: (1) Độ bền uốn và độ bền va đập của vật liệu gỗ nhựa giảm đi khi thời gian tiếp xúc thời tiết tăng lên. Vật liệu gỗ nhựa Keo tai tượng và HDPE có giá trị MOE, MOR và cường độ va đập lớn nhất, (2) Sự thay đổi màu sắc và độ sáng của vật liệu gỗ nhựa trong quá trình tiếp xúc thời tiết tăng lên khi thời gian tiếp xúc thời tiết tăng lên. Tốc độ thay đổi phụ thuộc vào loài gỗ. Sự ôxy hóa theo thời tiết của vật liệu gỗ nhựa được đánh giá bằng mức độ tăng nhóm carbonyl song song với thời gian tiếp xúc thời tiết. Vật liệu gỗ nhựa với bột gỗ là gỗ Keo tai tượng đạt được giá trị ΔE* và ΔL* cao hơn sau khi tiết xúc thời tiết so với vật liệu gỗ nhựa vớ bột gỗ là gỗ Bạch đàn urophylla và gỗ Thông caribaea.

I. INTRODUCTION

The utilization of wood-plastic composites (WPC) has rapidly increased, especially in Europe, Canada, and the United States. The most common types of such panels are produced by a similar manner as plastic-based products (Wolcott *et al.,* 1999; Dong and He. 2012). One of the major advantages of WPC is good biological resistance, for outdoor applications where untreated timber products natural are easily damaged by biology. Additionally, fine particles of wood waste from wood processing plants are low-cost and high availability. These composites are transformed by extrusion processes to obtain structural building applications, such as siding, fencing, pallets, tile roofing, and window frame lineals. A few other products such as pilings, railroad ties, marinas, window blinds, and sound barriers are being prepared by WPC instead of unfilled plastics with improved thermal and creep performance (Wechsler *et al.,* 2007; Morton and Rossi 2003; Kumar *et al.,* 2011).

However, exposure of WPC to natural environment changes their color, shape, and mechanical properties. The change in the early emergence of WPC due to weather exposure has been of great interest to consumers (Smith and Wolcott 2005). It is well-established that value-added wood products compete in markets where quality and cosmetic aesthetics often lead to either market success or failure (Ashori *et al.* 2009). Therefore, special attention should be paid to the properties, such as color, of the wood products because they affect the value (perceived quality) of the product. Some of the chemical reactions caused by solar radiation result in a change in color or appearance (Hon *et al.,* 1991). The presence of chromophores in plastic and wood usually increase the absorption of ultraviolet (UV) light, which in turn causes a light decay of the WPC material. The yellowing of weathered wood is due to the decomposition of lignin into water-soluble products resulting in the formation of chromophoric functional groups, such as carboxylic acids and hydroperoxyl radicals (Li *et al.,* 2000; Hon *et al.,* 1991). Metallic additives can also introduce chromophores into WPC. The phenyl and carbonyl groups are produced during the decomposition of wood (Jabarin *et al.,* 2010). Some research groups have been working to describe and understand the changes that occur when WPCs weather (Fabiyi *et al.,* 2010; Wei *et al.,* 2016; Kiguchi *et al.,* 2007). The wood fibers that swell after absorbing moisture and create cracks in the plastic matrix, lead to the loss of composite MOE and strength. Moisture also damages the appearance of wood and reduces the efficiency of stress transmission from the matrix to fiber (Rangaraj 2000; Peng *et al.,* 2014). UV rays erode the surface of the WPCs, making it cracked and broken. The combination of UV and water radiation also leads to the mechanical loss of WPCs. Moreover, the surface is eroded through water spraying, which in turn creates a new surface for degradation (Stark and Matuana 2010). The exposure of high-density polyethylene (HDPE) composite materials with wood pulp to xenon arc radiation, with or without water spray, brightens their colors (Stark and Mueller 2008).

In recent years, the timber industry is booming and become one of the ten key export industries in Vietnam. As a large agricultural country, Vietnam is rich in plant fiber and lack of forest resources, the traditional furniture industry has failed to make efficient use of wood and plant fibers, through the use of wood chips, wood shavings, straw and other deep processing of plant fiber, or through the use of composites and combination. The use of Vietnam's three major species of wood trees to enhance HDPE composite materials, apart from solving the problem of the effective use

of natural fibers can also open new directions for the development of Vietnam's timber industry. Although research and technological development in the area of WPC in Viet Nam is on the rise, till now, no comprehensive work has been done in this field.

There are several studies in this field; Tran Vinh Dieu *et al.,* (2006) investigated the impact strength of PP/rice husk powder composites. The experiment was divided into 30, 35, 40, 45, 50 and 55%, and MAPP 0.5%. The results show that when the content of rice husk powder was 55%, the impact strength (resistance) of composite was 2.5KJ/m^2 which was four times higher than that of the original PP composites. Ha Tien Manh *et al.,* (2011) studied the effect of ratio of wood and polypropylene on the properties of wood-plastic composites. *Acacia mangium* and PP recycled plastic were utilized as raw material in this research. There were three different ratios between wood powder and plastic were applied including (50:50; 70:30; 60:40). The results show that the ratio of wood flour and plastic has minor effect on the performance of wood-plastic composites, Vu Huy Dai *et al.,* (2012) conducted a study on development of composites from wood waste and recycling plastics. *Acacia mangium* and recycling PP, PE and PVC were selected as raw materials in this study. The results of the study: the establishment of a recycling of plastic processing technology, Acacia mangium wood shavings, scrap wood manufacturing technology. The ratio of wood pulp to regenerated plastic was determined by the ratio of wood flour/recycled plastic to the manufacture of plastic particles. The best ratio of wood pulp and recycled plastic was 50% of wood flour, 45% of plastic and 5% of coupling agent. At the same time, Machine Cinnanici TS80 production of wood-plastic composite materials raw materials for wood flour and PP, PE, PVC regeneration. However, so far there have not been studies on *Pinus caribae* and *Eucalyptus urophyla* - plastic composites and also investigations on the influence of weather on wood plastic composites. Moreve, there is no study on the use of *Pinus caribae* and *Eucalyptus urophyla*. The studies only examined the effect on the wood flour ratio on the resin, none of the studies investigated the effect of weather on the type of wood used for composites.

Therefore, the aim of this study was to determine the effects of wood properties on weathering resistance and durability of WPC. Wood species were selected based on their lignin content, mechanical properties, density, natural durability, dimensional stability and color. The three species selected were *Eucalyptus urophylla*, *Acacia mangium*, and *Pinus caribaea*. These three kinds of rapidly growing wood species are highly abundant in Vietnam. We aim to gain insight into the change of the surface of WPC during weathering. The surface characteristics of WPC before and after weathering were analyzed by Fourier transform infrared (FTIR) spectroscopy and Scanning electron microscopy (SEM).

II. EXPERIMENTAL

2.1. Materials

HDPE (grade: 5000s, melting flow index = 0.8 - 1.1 g/10 min at 190^{0}C, density = 0.949 - 0.953 g/cm^3) was purchased from Daqing Petrochemical Co. Ltd. China.

Three kinds of WF were used as fillers, from *Eucalyptus urophylla*, *Acacia mangiu*m, and *Pinus caribaea* The particle size = 40 - 80 mesh, length = 1 - 2.8 mm, aspect ratio = 9 - 12, was purchased from a local market. The coupling agent, maleic anhydride (MAPE; grafting percentage: 0.9%), was obtained from Shanghai Sunny New Technology Development Co. Ltd.

2.2. Preparation of composite samples

WF were dried in a drying oven at 105ºC to reduce the moisture content to less than 3%. Composite formulations were prepared containing 60 parts wood flour powder, 40 parts HDPE, 3 parts maleic anhydride-grafted polyethylene. These materials (HDPE, MAPE, and WF) were mixed in a high-speed mixer for 10 min and then fed into a twins-crew extruder for compounding at 145 - 165°C. The extruded melt mixture was cooled and comminuted into small particles by using a pulverizer. These small particles containing wood flour and HDPE were fed into a single-screw extruder system and processed into lumber with a cross-section of 4 × 40 mm.

2.3. Characterization

2.3.1 Accelerated weathering test

The composites were placed in a QUV accelerated weathering tester (QUV/Spray, Q-Lab Co., USA) according to ASTM G154. Each 12 h weathering cycle consisted of 8 h of UV exposure at 60°C followed by 4 h condensation at 50°C. The UV irradiance was 0.89 W m^{-2} at 340 nm wavelength. The changes in the surface color, surface morphology, flexural properties and impact strength of the tested samples were evaluated after durations of 0, 500, 1000, 1500, and 2000 h.

2.3.2 Colorimetric Analysis

The surface color of the samples was measured using the NF333 photometer (Nippon Denshoku Co.) according to the CIE L^* a^* b^* (Figure 1) color system. Lightness (L^*) and chromaticity coordinates (a^* and b^*) were measured for ten replicate samples. L^* represents the lightness coordinate and varies from 100 (white) to 0 (dark), while a^* represents the red ($+a^*$) to green ($-a^*$) coordinate, and b^* represents the yellow ($+b^*$) to blue ($-b^*$) coordinate. A decrease in L^* signifies that the sample has darkened. The total color change (ΔE) was calculated using *Eq*. 1 according to ASTM D 2244 - 02 (Standard Practice for Calculation of Color Tolerances and Color Differences from Instrumentally Measured Color Coordinates),

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

where ΔL^*, Δa^*, and Δb^* are the differences of initial and final values for L*, a*, and b*, respectively.

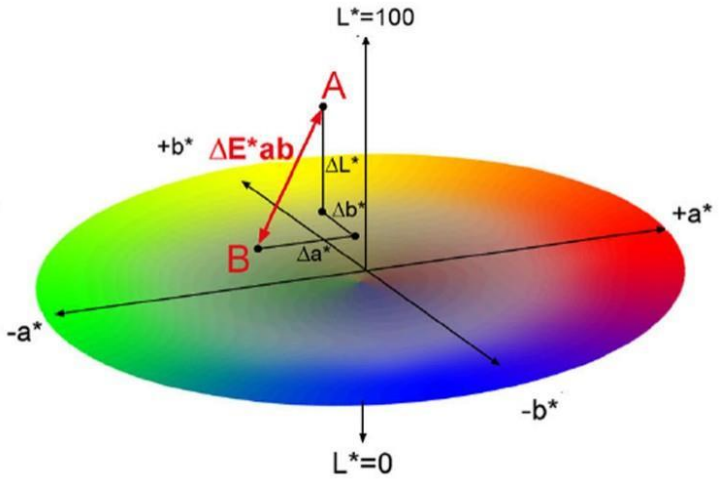

Figure 1. CIELab color space

2.3.3. Scanning Electron Microscopy (SEM)

The surface structure of the WF/HDPE composite samples was analyzed with SEM. The extrusion surfaces of the samples were sputter-coated with gold and analyzed under a scanning electron microscope (SEM; FEI QUANTA 200) at the working distance of approximately 25 mm, voltage of 15 kV, and probe current of 10 A.

2.3.4. Fourier Transform Infrared (FTIR) Spectroscopy

FTIR spectra (KBr disk method) were recorded with the MAGNA-IR560 (Thermo Nicolet) spectrometer to study the functional groups on the surface of WF/HDPE composites. Scanning was carried out at the resolution of 4 cm^{-1} in the range of 4000 to 400 cm^{-1}, with 32 scans for each sample.

2.3.5. Mechanical tests

The flexural tests were carried out with a universal testing machine (RGT-20A, Shenzhen Reger Instrument Co., Ltd.) in accordance with the procedure described in "Fiber-Reinforced Plastic Composites-Determination of Flexural Properties" (GB/T 1449 - 2005). The specimens had dimensions of 80×13× 4 mm and a span length of 64 mm; they were tested at the loading speed of 2 mm/min. Five specimens in each group were tested to obtain the values for the flexural modulus and flexural strength.

$$MOR_{ret.ratio} = \frac{MOR_{after}}{MOR_{before}} \times 100 \quad (2)$$

$$MOE_{ret.ratio} = \frac{MOE_{after}}{MOE_{before}} \times 100 \quad (3)$$

where MOR_{before}, MOE_{before}, MOR_{after} and MOE_{after} represent the MOR and MOE before and after weathering.

The unnotched impact strength was examined with an impact testing machine (XJ-50Z, Chengde Precision Testing Machine Co. Ltd.) on the basis of GB/T 1043.1 - 2008 ("Plastics, Determination of Charpy Impact Properties, Part 1: Non-instrumented Impact Test"). The specimens had dimensions of 80 × 10 × 4 mm and the span length of 60 mm. The striking velocity of the tests was 2.9 m/s, and the pendulum energy was 2J. Five specimens of each sample were tested to determine the impact strength.

III. RESULTS AND DISCUSSION

3.1. Color changes of WPC during weathering

As shown in Figure 2, the discoloration of the composites (made from three different wood species) during weathering occurred in three stages. The samples quickly photobleached after exposure for 1000 h and then lightened with further exposure till 1500 h and 2000 h. In particular, two types of wood (*Acacia mangium*, *Pinus caribaea*) took additional 2000 h to turn red-brown. The discoloration of the weathered samples can be attributed to the photo-oxidation of lignin. Muasher and Sain (2006) proposed two competing redox reactions of lignin that could be responsible for photobleaching after weathering due to UV exposure.

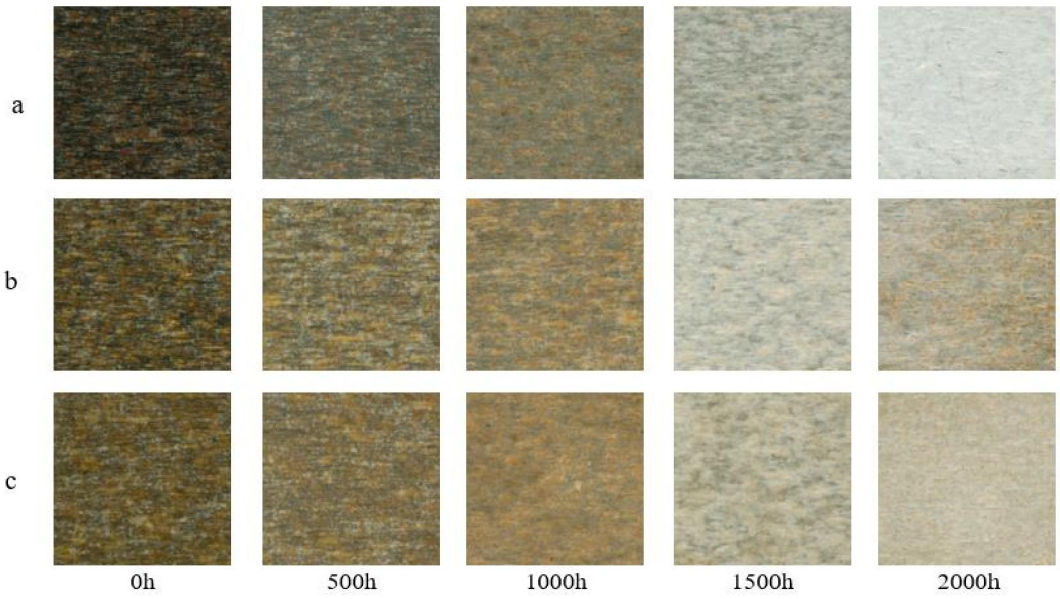

Figure 2. Visual photos all groups of composites as a function of weathering time for (a) *Eucalyptus urophylla*, (b) *Acacia mangium*, and (c) *Pinus caribaea*

Relative lightness (ΔL^*) and total color change (ΔE^*) of the surface of weathered WPC in Figure 3. ΔL^* was used to understand the degree of lightness of weathered WPC relative to the original WPC color. ΔL^* and ΔE^* increased significantly upon weathering until 1500 h and decreased thereafter. These results are consistent with the other WPC weathering studies (Stark and Matuana, 2010; Fabiyi *et al.,* 2008; Stark, 2003). The total color change of WPC's containing three different wood species ranked as follows: *Acacia mangium* < *Pinus caribaea* < *Eucalyptus urophylla*. However, the surface lightness ranked slightly differently from that of the total color change pattern (*Acacia mangium* < *Pinus caribaea* < *Eucalyptus urophylla*).

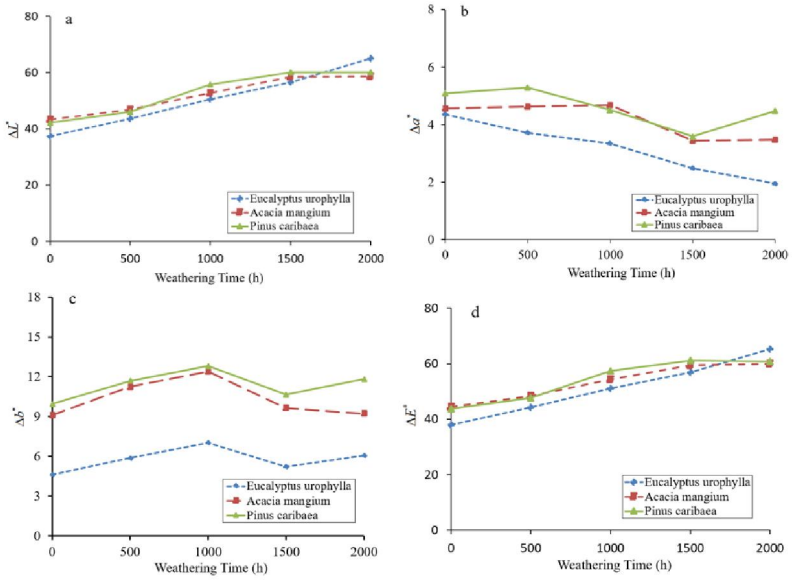

Figure 3. Change of color parameters: (a) L^*, (b) a^*, and (c) c^* and (d) color change (ΔE) of all groups of composites as a function of weathering time.

The results of fourcolor indices (i.e. ΔL^*, Δa^*, Δb^* and ΔE^*) are plotted with exposure time in Figure 3(a)-(d), respectively. The lightness of all WPCs increased dramatically from 37.3 - 42.2 to 43.6 - 45.97 after the first 500 h of exposure. After 500 h of weathering exposure, the lightness continued to increase reaching 58.48 - 59.99 at 1500 h. The sample (with *Acacia mangium*, *Pinus caribaea*) started to decrease, and the decrease continued until the end of the weathering tests reaching the final lightness value of 58.60. The lightness of *Eucalyptus urophylla* increased after 500 h of exposure and continued to increase to reach the final lightness value of 64.97. The lightness values for before and after 2000 h of weathering exposure were extremely close for all the WPC. The increase in lightness suggests the degradation of substances that reflect yellowish and reddish light, causing a bleaching effect on the composite samples. The reason for the color change is believed to be photodegradation of structures in wood components, especially conjugated structures which degrade under UV radiation and water spray.

It is to be noted that color fades with an increase in the wood content of WPC (Stark 2003). This color fade pattern reveals that WPC formulations with a moderate weathering performance can be achieved via proper wood species selection (such as Acacia). The use of acacia mangium for WPC production, with no pigments or ultraviolet absorbers, seems to be a promising step towards a reduction in production cost and improvement of color stability. Hence, based on our results, *Acacia mangium* are preferable wood species for improving WPC color performance.

3.2. Surface morphology

The surface morphology of WPC also changed significantly after weathering. Many authors have observed cracks on the surface of WPCs during natural or accelerated weathering (Stark and Matuana 2004; Kiguchi *et al.,* 2007). SEM micrographs of WF/HDPE composites (see Figures 4) after accelerated UV weathering for 0, 1000, and 2000 h reveal coarsening of the initially smooth surfaces. The effect was visible after 1000 h and the coarsening was followed by matrix cracks and interfacial defects upon extending the weathering duration to 2000 h.

After weathering for 2000 h, the surface of each composite was degraded (Figure 4), cracks appeared in WPC's matrix, and wood particles protruded from the composite surface. *Eucalyptus urophylla*/HDPE composite surfaces were more degraded by weathering than that of *Acacia mangium*/HDPE and *Pinus caribaea*/HDPE composites. The protrusion of wood particles was probably due to wood particles' swelling and shrinking after absorbing and desorbing moisture, respectively. This action can result in voids at the wood flour/HDPE interface. These results are consistent with the three stages of degradation in WPC observed by Fabiyi *et al.,* (2008). At first, the surface layer was eroded and small cavities were created (Figure 4). The frequency and size of these cavities increased with extended weathering. Crazing of weathered WPC surfaces was probably caused by polymer chain scission leading to the creation of the highly crystallized HDPE zones. These zones then contracted and expanded during the drying and wetting cycles, respectively. Crazing occurs more readily in the presence of water spray during weathering (Hill and Jones 1996). Finally, small cracks that were formed on the weathered WPC surface were a repeat of the second stage.

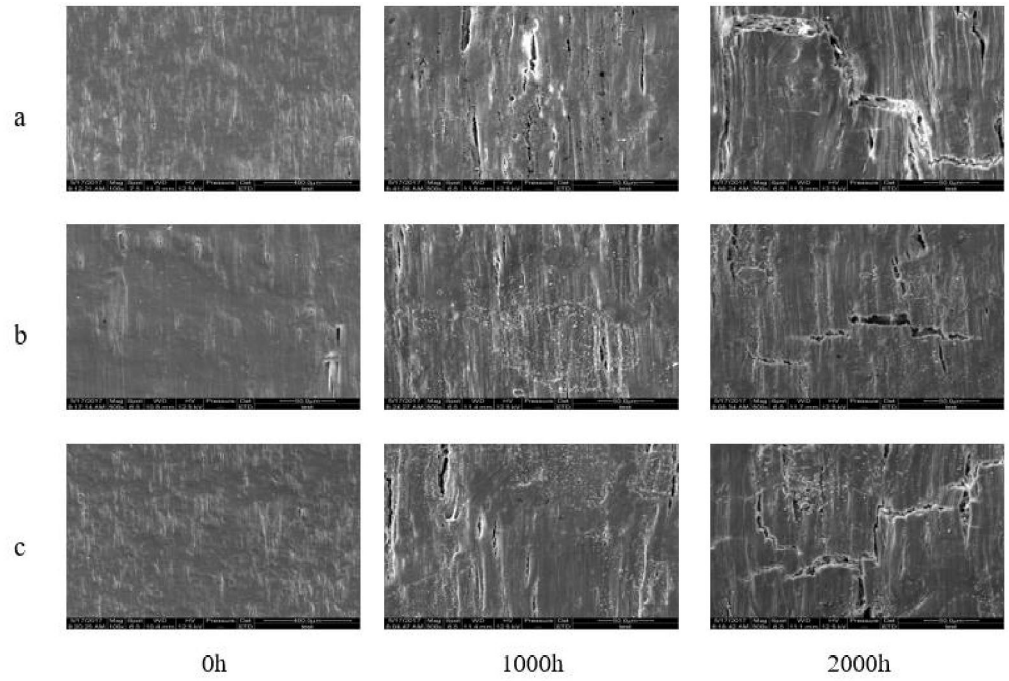

Figure 4. SEM micrographs of (a) *Eucalyptus urophylla*, (b) *Acacia mangium*, and (c) *Pinus caribaea* WPC after accelerated UV weathering for 0 (control), 1000, and 2000 h, respectively

3.3. Mechanical properties

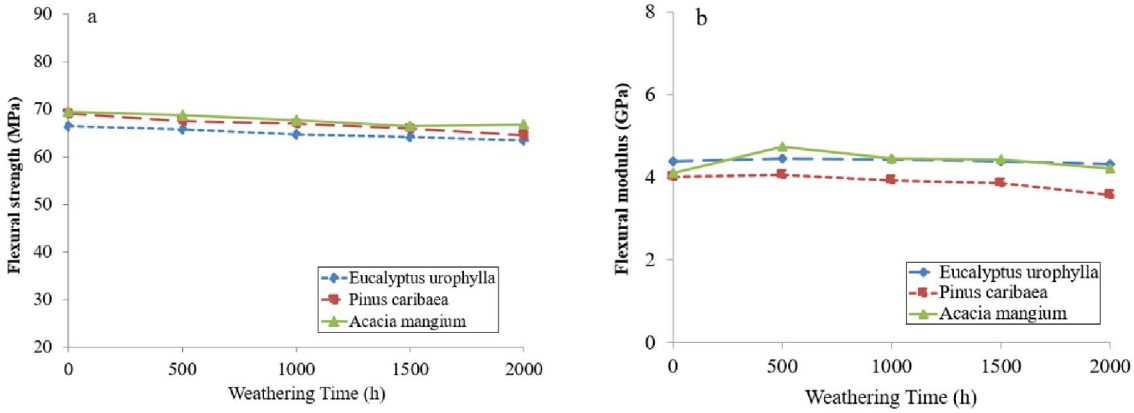

Figure 5. (a) Flexural strength and (b) modulus of the composites as a function of weathering time

The changes in the flexural properties of the WPC during weathering exposure are presented in Figure 5(a) - (b). The flexural MOR and MOE of all the composites decreased with increasing weathering time. After 2000 h, the MOR retention ratios of the WPC were in the order: *Acacia mangium*/HDPE (96%) > *Eucalyptus urophylla*/HDPE (95%) > *Pinus caribaea*/HDPE (93%). Among these, polyethylene-based WPCs of HDPE/acacia mangium composite retained the greatest strength over the weathering period, while the polyethylene-based WPCs of pinus caribaea retained the least. Similar to the trend observed for the flexural strength, the MOE

retention ratios of all WPC after 2000 h were in the order: *Acacia mangium*/HDPE (103%) > *Eucalyptus urophylla*/HDPE (99%) > *Pinus caribaea*/HDPE (89 %). In general, the mechanical properties of the WPC were not only influenced by the nature of the wood and plastic materials but also by the interfacial adhesion in the composites. Doan *et al.,* (2007) reported that improving the interfacial adhesion between the wood fibers and the matrix led to lower values of the equilibrium water content and diffusion coefficient. The majority of flexural MOR and MOE changes occurred during the first 1000 h (Table 1). These results are similar to those reported by Stark *et al.,* (2004), who investigated the loss in MOR and MOE for 50% wood flour-HDPE composites after weathering.

Table 1. Flexural properties (MOR, MOE) of composites before and after weathering.

WPC	MOR (MPa)	MOR retention ratio (%)				MOE (GPa)	MOE retention ratio (%)			
		500h	1000h	1500h	2000h		500h	1000h	1500h	2000h
Eucalyptus urophylla/HDPE	66.4±0.77	99±0.01	97±0.50	96±0.66	95±0.50	4.3±0.1	101±0.83	101±0.14	100±0.02	98±0.63
Acacia mangium/HDPE	69.5±0.82	98±0.57	97±0.86	95±0.52	96±0.14	4.1±0.17	115±0.33	108±0.52	107±0.79	102±0.68
Pinus caribaea/HDPE	69.2±1.48	97±0.91	96±0.41	95±0.24	93±0.35	4.0±0.3	100±0.75	97±0.51	95±0.77	88±0.81

Both MOR and MOE values decreased after weathering due to UV radiation and water spray. The MOE retention ratio of all the composites displayed a similar trend as in MOR retention ratio. As HDPE is exposed to UV radiation, chain scission occurs. Additionally, exposure to water degrades mechanical properties of WPC primarily due to swelling of the wooden particles. The swollen particles cause microcracks in the matrix, leading to a decrease in flexural strength and reduction of efficiency of stress transfer from fiber to the matrix.

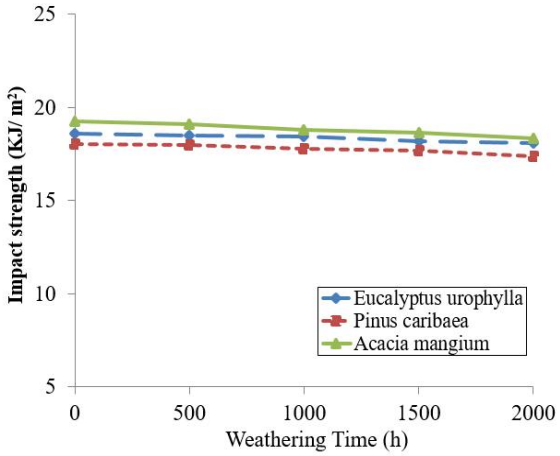

Figure 6. Variation of impact strength of the composites with weathering time

As shown in Figure 6, the impact strength of all composites decreased with increasing weathering time. After 2000 h, the impact strength of the composites decreased by 97%, 95%, and 96% for HDPE/*Eucalyptus urophylla*, HDPE/*Acacia mangium*, and HDPE/*Pinus caribaea*, respectively.

3.4. Fourier Transform Infrared Spectroscopy

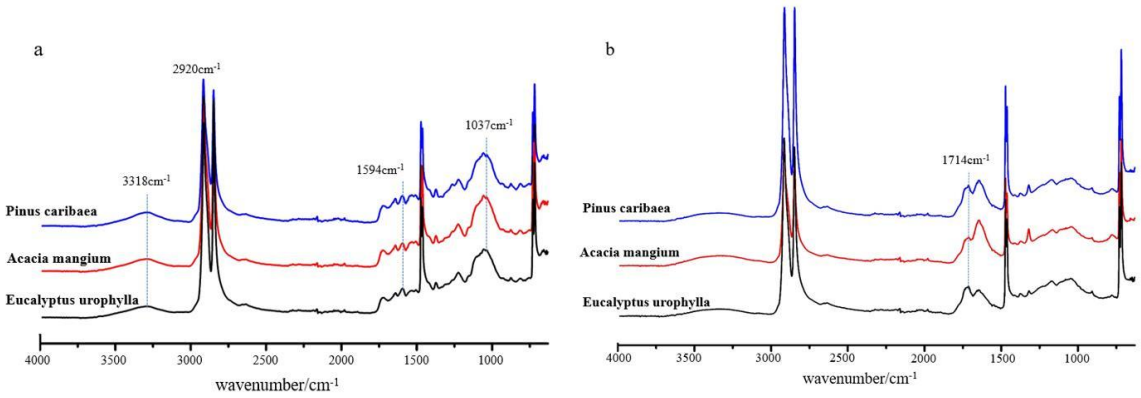

Figure 7. FTIR spectra of different types of composites before and after 2000h weathering

FTIR spectroscopy is a crucial technique to analyze the structure of wood components and chemical changes on wood surface induced by UV-irradiation (Stark and Mutuana 2007). FTIR spectra of IR in the range of 4000 to 2500 cm^{-1} are shown in Figure 7a. A strong band observed at 2920 cm^{-1} in all unweathered samples (Figure 7a) is assigned to alkane CH vibrations of methylene groups (-CH2 -) of *Pinus caribaea*/HDPE (Stark and Mutuana 2010). However, the intensity of this band decreases rapidly in *Eucalyptus urophylla*/HDPE and *Acacia mangium*/HDPE group after exposure, while it is still visible in the control group and *Pinus caribaea/*HDPE. The intensity of the peaks assigned to cellulose at 3318 cm^{-1} (free -OH) decreased significantly after 2000 h exposure (see Figure 7b), indicating that more WF appeared on the weathered surface.

The intensity of the band at 1850 - 900 cm^{-1} assigned to the carbonyl groups (carboxylic acids at 1714 cm^{-1}) showed a remarkable increase. This implies that weathering caused the oxidation and photodegradation of cellulose. This also suggests that the composites were vulnerable to further degradation because the carbonyl groups are photo-labile. After exposure, a significant decrease in the intensity of peaks at 1037 cm^{-1} (C-O in cellulose and hemicelluloses) occurred on the surface of WPC. This phenomenon suggests that the wood degraded extensively upon weathering. After 2000 h of aging, the characteristic peak at 1594 cm^{-1} representing lignin had nearly disappeared. This result is consistent with the changes shown in Figure4, where it is apparent that the surface bonding substance (lignin and pectin) was lost, leaving gaps between the fibers. These changes in the characteristic peaks imply that cellulose, hemicelluloses, and lignin underwent serious degradation.

IV. CONCLUSIONS

The UV weathering resistance of WPC are greatly affected by the type of WF. FTIR analysis demonstrates that there were significant changes in the surface chemistry of the composite material and characteristic vertices of cellulose and lignin after exposure The flexural properties of the composites declined significantly between 0 h and 2000 h of aging while the impact properties improved insignificantly during the same time. Among the studied WPC, *Acacia mangium*/HDPE composite exhibited higher MOR and MOE retention ratios after 2000 h accelerated UV

weathering comparing to *Eucalyptus urophylla*/HDPE *and Pinus caribaea*/HDPE. In addition, the color change of the *Acacia mangium*/HDPE composite was the lowest. Accordingly, *Acacia mangium*/HDPE composite showed the best weathering resistance in these three studied WPC.

We conclude that *Acacia mangium* is preferable for applications where color and mechanical properties are of prime importance.

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