The Influence of Environmentally Friendly Plasticizer on the Bio-Durability of Wood Plastic Composites

Kaimeng Xu,*[,1] Gaofeng Xu,[1] Yuanbo Huang, Can Liu, Kunyong Kang, and Zhifeng Zheng*

The influence of epoxidized soybean oil (ESO) plasticizer on the mould and algal resistances of wood plastic composites (WPCs) was studied using artificial accelerated tests. The macro- and micro-morphology of the colonization of algae and mould on the surface and fracture morphology of the WPCs samples were observed by digital camera and scanning electron microscopy (SEM). The water absorption and thickness swelling rates of different WPCs specimens with various addition amounts of ESO were also tested. The results indicated that as more of the ESO was added, the mould or algal resistance became weaker, especially on the surfaces of the specimens, which became harshly colonized when the addition amount of ESO was beyond 15 phr. The colonization speed and intensity of the mould were higher than the algae at the same addition level of ESO and the same testing time. The hyphae and spores of mould, but not algae, were found on the inner fracture layers.

Keywords: Wood plastic composites; Bio-durability; Epoxidized soybean oil; Mould; Algae

Contact information: University Key Laboratory of Biomass Chemical Refinery & Synthesis, Yunnan Province; Engineering Laboratory for Highly-efficient Utilization of Biomass, Yunnan Province, College of Materials Engineering, Southwest Forestry University, Kunming 650224, P. R. China;
[1]Kaimeng Xu and Gaofeng Xu are co-first authors;
**Corresponding authors: xukm007@163.com and zhengzhifeng666@163.com*

INTRODUCTION

There is great interest in lignocellulosic fiber and thermoplastic composites due to the rising concerns of resource depletion and environment protection. With fast growth in the global market share recently, wood plastic composites (WPCs) are emerging as green composite materials that combine the advantages of wood and thermoplastics, for example, long-term performance, cost-effectiveness, shape flexibility, and small carbon footprint (Ashori 2008; Naumann *et al.* 2012). Wood plastic compounds are extensively employed in interior and exterior applications, such as building, housewares, automobile components, and various structural members (Muller *et al.* 2013; Ratanawilai *et al.* 2014).

WPCs are often exposed to the surrounding environment, where moisture conditions change rapidly. WPCs products were once considered impervious to the attack of living organisms, such as fungi, mould, algae, marine borer, and termites because the wood particles were assumed to completely be encapsulated by the thermoplastic resin, thereby providing effective protection against biological damage. This assumption was disproven by laboratory testing and practical applications (Defoirdt *et al.* 2010; H'ng *et al.* 2011; Segerholm *et al.* 2012; Catto *et al.* 2016). Major studies on WPCs have focused on polyolefins as a polymeric matrix. In contrast to polyolefins, polyvinylchloride (PVC)

improves the stiffness, creep behavior, weatherability, bio-durability, solvent resistance, flame retardancy, and paintability (Jiang and Kamdem2004; Muller *et al.* 2013; Xu *et al.*2013). Therefore, there is increased use of PVC-based WPCs, with a growth rate of 200 % from 2002 to 2010 (Xu *et al.* 2014), which suggests that it is more important for WPCs applications. There is a repetitive vinyl chloride unit of head-to-tail arrangement for PVC, with various amounts of chain-branching and a low degree of crystallinity. The highly electro negative nature of chlorine leads to high dipoles along the polymer molecules with strong secondary valence forces, which limits the mobility of molecular chains (Matuana 1997). This results in macroscopic brittleness of PVC. Plasticizers are usually incorporated into the formulation of PVC to obtain satisfactory processing and flow properties by modifying its flexibility and distensibility and to reduce the viscosity of the molten compound in heat processing (Vieira *et al.* 2011; Xue *et al*. 2014).

The most commonly used plasticizers in PVC formulations are dioctyl phthalate (DOP) and diisononyl phthalate (DINP), which are becoming limited due to their potentially high toxicity and carcinogenicity (Chiellini *et al.* 2011; Lithner *et al.* 2011; Hines *et al.* 2012; Foghmoes *et al.* 2016). Moreover, the worsening petroleum shortage and environmental pollution have attracted more attention to "green" plasticizers from renewable resources that are characterized by low- or non-toxicity. Epoxidized soybean oil (ESO), a vegetable oil, is used extensively as lubricant, reinforcing agent, plasticizer, *etc.*, in composites because of the inherent advantages of huge quantities, low cost, biodegradation, and non-toxicity (Bueno-Ferrer*et al.* 2010; Zhang *et al.* 2010; Yang *et al.* 2014). Although ESO effectively enhances the thermal stability and processability of PVC products (Bueno-Ferrer *et al*. 2010), there is a lack of information about the effects of plasticizers on the bio-durability of wood flour-filled PVC composites.

The aim of this study was to firstly investigate the influence of ESO plasticizer on the bio-durability of PVC-based WPCs using the artificial accelerated tests with mould and algae. The macro- and micro-morphology of the colonization of algae and mould on the surface and the fracture morphology of WPCs were observed by the digital camera instrument and scanning electron microscopy (SEM). The water absorption and thickness swelling rates of the different WPCs specimens with various additions of ESO were also tested.

EXPERIMENTAL

Raw Materials

Four algal species (*Chlorella vulgaris*, *Ulothrix* (sp.), *Scenedesmus quadricauda*, and *Oscillatoria* (sp.)) and five mould fungi (*Aspergillus niger*, *Chaetomium globosum*, *Penicillium funiculosum*, *Aureobasidium pullulans*, and *Trichoderma viride*) were provided by the Guangdong Institute of Microbiology, Guangzhou, China. PVC resins (DG-800), with an average degree of polymerization of 800 and a density of 1.35 g/cm^3, were purchased from Tianjin Dagu Co. Ltd., Tianjin City, China. Wood flour (WF) with a particle distribution of 80 to 100 mesh was obtained from Xishuangbanna Huakun Biological Technology Co., Ltd, Jinghong City, China. ESO was purchased from Guangzhou Wen Jia Chemical Co., Ltd, Guangzhou City, China. Its epoxy value was no less than 6.0, and its iodine value was no greater than 5.

Preparation of the Composites

The WF particles were oven-dried prior to use. First, the WF and the PVC resin

were blended at 80 °C in a high-speed mixer at 1600 rpm (SHR-10A, Zhangjiagang, China). The mixture was extruded in the shape of a rod by a co-rotating twin-screw extruder (JIEENTE SHJ-20, Nanjing, China) with the temperature range of 150 °C to 180 °C and an average rotation speed of 40 rpm. The granules were transferred to a conical twin-screw extruder (JINWEI SJZ-65, Suzhou, China) to produce 5-mmthicksheet samples. The processing temperature during extrusion was set at the range of 115 °C to 170 °C from hopper to die zone. The rotational speeds of the twin screw and single screw were 20 rpm and 8 rpm, respectively. The composite formulations are given in Table 1.

Table 1. Composite Formulations

Groups	PVC (phr)	WF (phr)	ESO (phr)	Other additives (phr)
ESO-0	100	40	0	6
ESO-5	100	40	5	6
ESO-15	100	40	15	6
ESO-25	100	40	25	6
ESO-35	100	40	35	6

Artificial Accelerated Algal Resistance Test

The artificial accelerated algal resistance tests of the specimens with the four algal species were carried out according to the standard of GB/T 21353 (2008). Each 1.0 mL algal sample was pumped using a sterile pipette from the four algal suspensions and was pipetted into the prepared liquid culture medium. The liquid culture medium was maintained for two weeks under suitable conditions. The mixing suspensions with four algal species were prepared by incorporating the same amount of each algal sample to the suspension media. The sterilized samples with the dimensions of 30 mm (length) × 30 mm (width) × 5 mm (thickness) were placed into petri plates. Subsequently, the surface of each sample was sprayed with an equal amount of algal suspension using a spray bottle. Petri plates were stored at 25 ± 2 °C, 1000 to 3000 lx 14:10 h light/dark cycles, and 80 % relative humidity for 4 weeks.

Artificial Accelerated Mould Resistance Test

The artificial accelerated mould resistance tests of the specimens were conducted in accordance with GB/T 24128 (2009). Five mould fungi species were selected for use in this test. The culture of each fungus was initially inoculated on potato dextrose agar in petri plates at 28 °C and relative humidity of 85 % until the whole surface of the petri plates was covered with fungal hyphae. A few spores were gently scraped using a nichrome inoculating wire and then poured into a test tube with 10 mL of sterile water to make a spore suspension. The suspension was vigorously vibrated in an Erlenmeyer flask with solid glass beads in order to separate spores and break the spore clumps. It was subsequently filtered and centrifuged to recover the precipitate. Finally, the five types of mould mixing suspension were obtained by adding each type of suspension of mould spore at the same addition amount. The composite specimens with the same dimensions as the samples of algal resistance were dried and sterilized prior to the tests. The surface of each sample was sprayed with equal amount of mixing mould inocula suspension and covered. Petri plates were kept at 28 ± 2 °C and 80 % relative humidity for 4 weeks.

Morphological Analysis

The surface and fracture morphology of the specimens were assessed through

imaging with a scanning electron microscope (SEM). The surfaces were prepared by sputter-coating with gold to prevent electrical charging, and were imaged with a Hitachi S-3000N microscope (Hitachi, Tokyo, Japan) with an acceleration voltage of 10 kV.

Water Absorption and Thickness Swelling Tests

Specimens with dimensions of 20 mm × 20 mm × 5 mm (length × width × thickness) were completely immersed in distilled water at 25 ± 2 °C for 24 days. The weights of samples were measured every day. The water absorption and thickness swelling rates of the samples were calculated according to Eqs. 1 and 2. Ten replicate specimens for each group were tested for standard deviations,

$$WA = \frac{m_t - m_0}{m_0} \times 100\% \quad (1)$$

where WA is the water absorption rate (%), m_0 is the weight (g) of the samples before testing, and m_t is the weight (g) of the samples at certain time,

$$TS = \frac{h_t - h_0}{h_0} \times 100\% \quad (2)$$

where TS is the final thickness swelling rate (%), h_0 is the thickness (mm) of the samples before testing, and h_t is the thickness (mm) of the samples after testing.

RESULTS AND DISCUSSION

Visual Appearance Analysis of Algal Colonization

Figure 1 illustrates the algal colonization of the WPCs samples with various addition amounts of ESO after an incubation period of 28 days. There were obvious visual differences in the algal growth on the different composites.

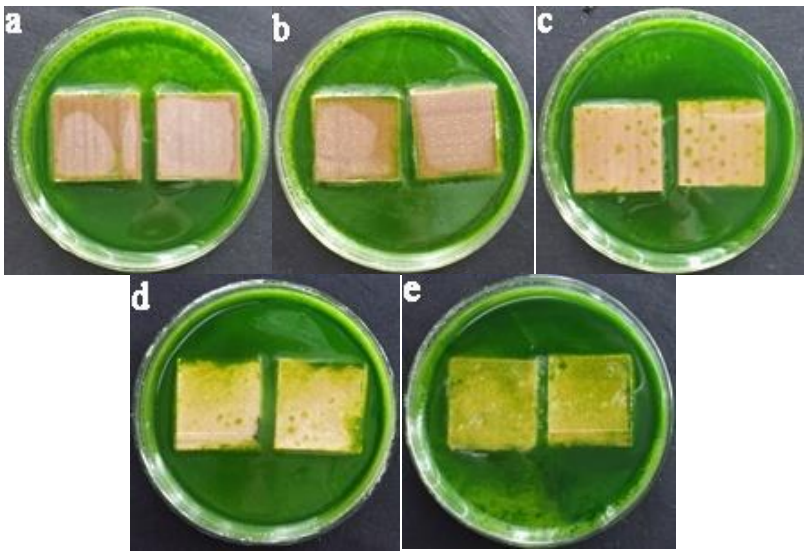

Fig. 1. Algal colonization of WPC with (a) 0 phr, (b) 5 phr, (c) 15 phr, (d) 25 phr, and (e) 35 phr ESO

The WPCs samples (ESO-0 and ESO-5) in Fig. 1(a, b) that had 0 phr and 5 phr ESO treatment were not significantly affected on their surface, which meant that both had relatively superior resistance to algal growth. However, the surfaces of WPCs samples (ESO-15, ESO-25, and ESO-35) were covered with more algae when the addition amount of ESO increased from 0 phr to 35 phr, as seen in Fig. 1(c to e). This demonstrated that the algal resistance decreased with the increased amount of ESO.

Table 2 shows that there was a relatively stable growth (level 1) for the groups ESO-0 and ESO-5 with the testing time gradually prolonged from 0 to 28 days. The groups ESO-25 and ESO-35 displayed an increasing trend when the testing time varied from 14 to 28 days, corresponding with level 2 to level 3 and level 2 to level 3 to 4, respectively. In addition, the algal growth level of the ESO-15 group gradually increased from level 1 to level 2, and then from level 2 to level 2 to 3, corresponding to the increase of time from 7 to 14 days and then from 14 to 28 days, respectively. This result suggested that the algal colonization was stepwise because the algal growth required a certain time.

Table 2. Algal Growth Level of WPCs with Various Addition Amounts of ESO

Groups	The algal growth level at 7d	The algal growth level at 14d	The algal growth level at 28d
ESO-0	1	1	1
ESO-5	1	1	1
ESO-15	1	2	2 to 3
ESO-25	2	2	3
ESO-35	2	2	3 to 4

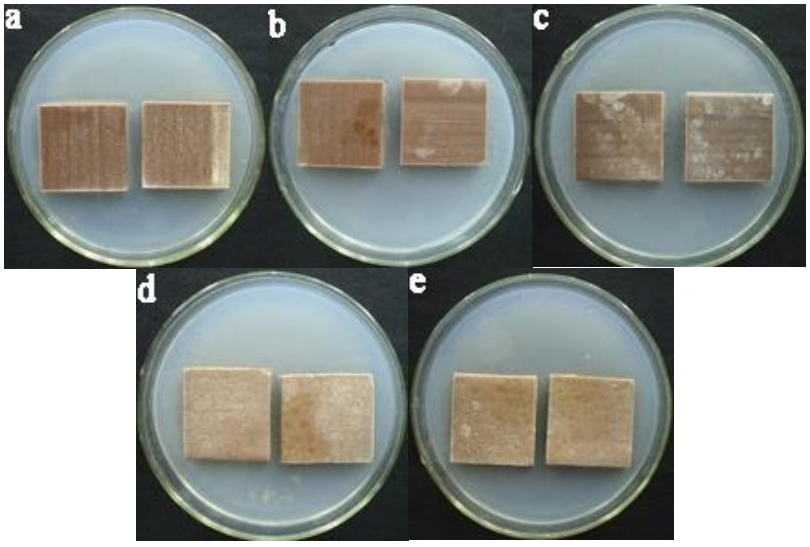

Fig. 2. The mould colonization of the WPCs with (a) 0 phr, (b) 5 phr, (c) 15 phr, (d) 25 phr, and (e) 35 phr ESO

Visual Appearance Analysis of Mould Colonization

Mould resistance analysis showed similar results as the algal resistance analysis. As shown in Fig. 2 and Table 3, a small amount of mould was found on the surfaces of ESO-0 and ESO-5, with growth levels of 2 and 2 to 3, respectively. The surfaces of ESO-25 and ESO-35 were covered with a great amount of mould, as shown in Fig. 2(d, e), which corresponded to the mould growth level of 4 at 28 days. Thus, they exhibited poor

resistance to mould growth. The mould growth level of ESO-15 was in the middle, corresponding to the level 2 to 3. As more ESO was added in the WPCs formulation, the mould growth levels of the WPCs samples were weaker. Comparing the data in Tables 2 and 3, the mould growth levels of the WPCs specimens with the same addition amount of ESO were lower than the mould growth level in the first 7 days, but there was a higher growth level for the mould than the algae after the 28-day period. Specifically, the final mould growth level of WPCs specimens, on average, increased by 1 level compared with final algae growth. Hence, the colonization speed and intensity of the mould were higher than the algae.

Table 3. Mould Growth Level of WPCs with Various Addition Amount of ESO

Groups	The mould growth level at 7d	The mould growth level at 14d	The mould growth level at 28d
ESO-0	1	2	2
ESO-5	1	1 to 2	2 to3
ESO-15	1	1 to2	2 to3
ESO-25	1	2 to3	4
ESO-35	1	3	4

Micro-Morphology Analysis of Algal Colonization

The surface and fracture micro-morphology of the algal growth for the WPCs samples with various addition amounts of ESO are shown in Fig. 3.The surface of the original WPCs samples without ESO was not covered by algae, as shown in Fig. 3(a). Figure 3(b) shows that although the wood particles were embedded in the matrix of PVC resin, there were still a few micro-voids. However, in Fig. 3(c, e, g, and i), the colonization of algae on the surface of the samples tended to increase when the addition of ESO increased from 5 phr to 35 phr, which was consistent with the results mentioned above. The hyphae and spores of the algae were not often found on the inner fracture layers for all of the groups due to the aggregation. Additionally, the interfacial compatibility between the wood particles and the thermoplastic resins increased with 5 phr ESO, which resulted in decreasing voids. Moreover, some co-continuous phase structures were observed with the addition of 15 phr ESO. However, the interface exhibited roughness, and there was an increasing appearance of voids on further addition. It was speculated that the ESO had a positive effect on improving the interfacial bonding of WPCs under a suitable addition amount.

Micro-Morphology Analysis of Mould Colonization

Figure 4shows the micro-morphology of mould growth for the WPCs samples with various amounts of ESO. Generally, there was a similar increasing trend with algal growth. Figures 4 (a, c, e, g, and i) show that more mould grew on the surface of the WPCs samples when the ESO was increased from 0 phr to 35 phr. As mentioned above, there was relatively stronger interfacial bonding with the composite specimens with 5 phr and 15 phr ESO (Fig. 4d and f), which had less voids compared with the original WPCs samples without ESO (Fig. 4b). In addition, the hyphae and spores of the mould were found on the inner fracture layers, possibly due to the low degree of aggregation. This observation indicated that the addition of ESO in the WPCs formulation markedly decreased the mould resistance. It is advised that the addition amount of ESO needs to be controlled when WPCs is applied in outdoor products.

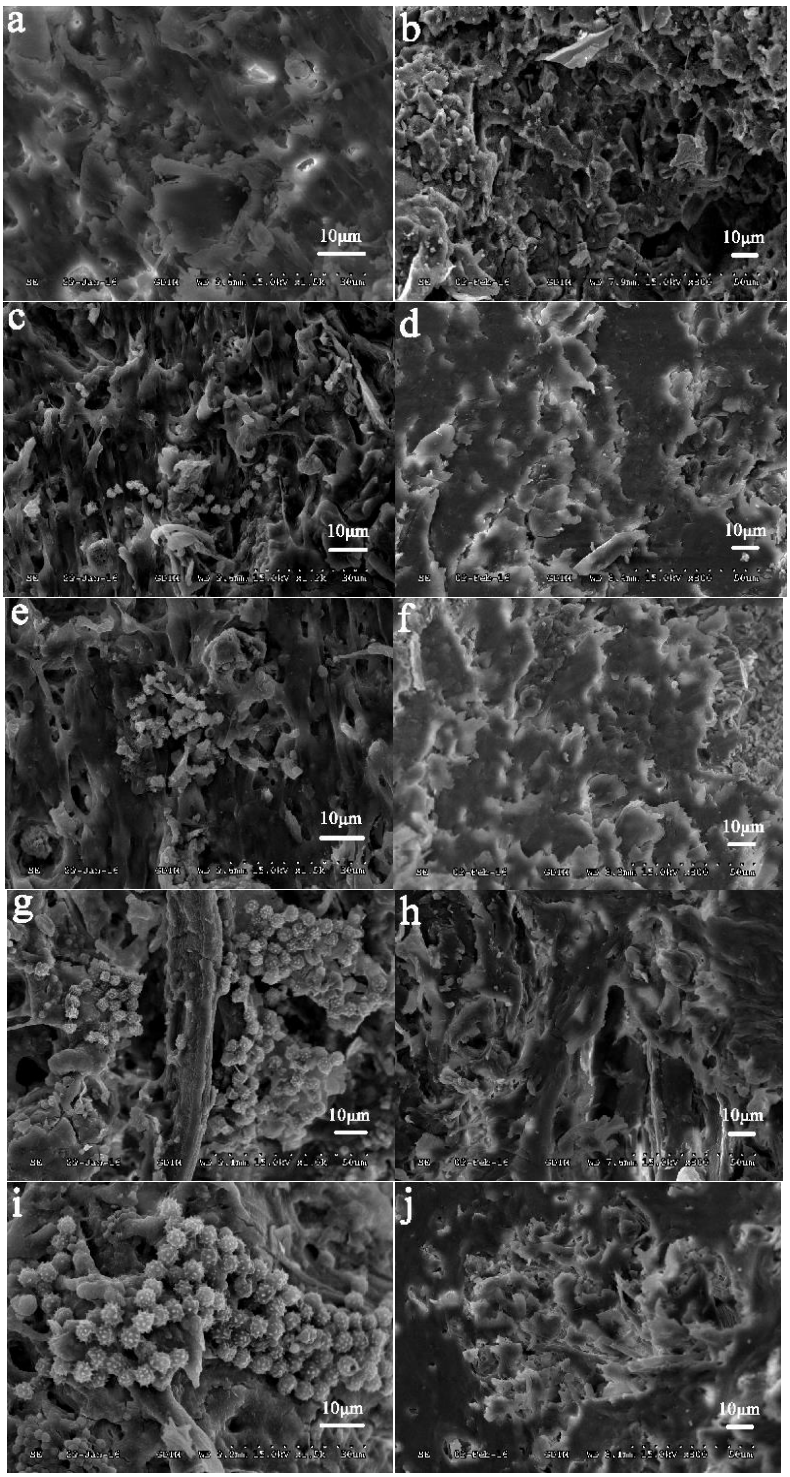

Fig. 3. The surface and fracture micro-morphology of algal growth of WPCs with (a) and (b) 0 phr ESO, (c) and (d) 5 phr ESO, (e) and (f) 15 phr ESO, (g) and (h) 25 phr ESO, and (i) and (j) 35 phr ESO. For each group, surface morphology is on the left, and fracture morphology is on the right

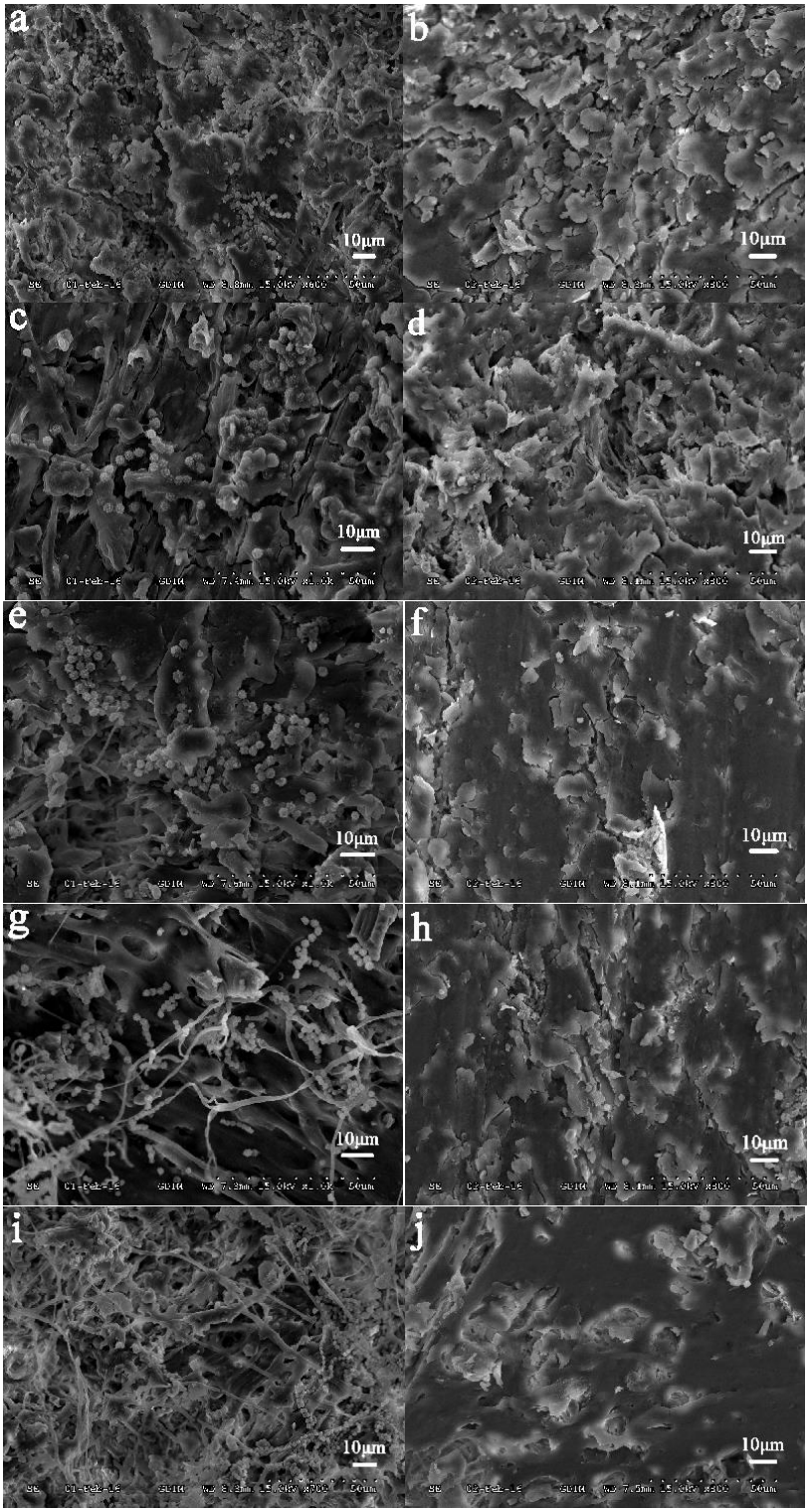

Fig. 4. The surface and fracture micro-morphology of mould growth for the WPCs with (a) and (b) 0 phr ESO, (c) and (d) 5 phr ESO, (e) and (f) 15 phr ESO, (g) and (h) 25 phr ESO, and (i) and (j) 35 phr ESO. For each group, surface morphology is on the left, and fracture morphology is on the right

Water Absorption Behavior

The water absorption and thickness swelling rates of the different WPCs groups are plotted in Fig. 5. The water absorption rates of all the WPCs groups with various amounts of ESO generally increased rapidly in the initial stage (within five days), increasing from 0 % to 1.18 %, 1.61 %, 1.40 %, 3.55 %, and 4.05 % with the ESO addition of 0 phr, 5 phr, 15 phr, 25 phr, and 35 phr, respectively. The water absorption rate gradually slowed down. The maximum water absorption rates of the specimens after adding 25 phr and 35 phr ESO (6.13 % and 7.71 %) were much higher than the samples with 0 phr, 5 phr, and 15 phr ESO (2.49 %, 2.90 %, 3.31 %, respectively). Additionally, the two curves of the ESO-5 and ESO-15 groups crossed each other; the reason may be a result of improvement of the interfaces within WPCs at the suitable addition level of ESO. This agrees with the analysis mentioned above. However, the excess ESO led to the decrease in water resistance for WPCs. Similar variation trends occurred in the thickness swelling rates of the WPCs, as illustrated in Fig. 5b. As more ESO was added, the thickness swelling rate increased. The average maximum thickness swelling rate for the samples was the ESO-35 group, which increased by 149 % compared with the control group.

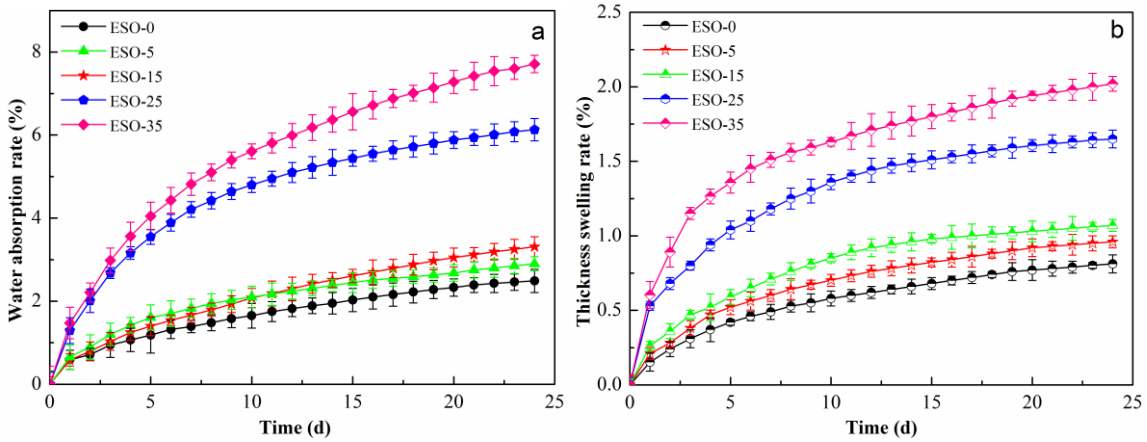

Fig. 5. The water absorption and thickness swelling rates of WPCs samples without and with various amounts of ESO

CONCLUSIONS

1. Epoxidized soybean oil (ESO), as a green bio-plasticizer, can be successfully added into the formulation of polyvinylchloride (PVC)-based wood-plastic composites (WPCs) with good processing characteristics. However, the addition of ESO had a markedly negative effect on the bio-durability (mould and algal resistance) of the WPCs.
2. The more ESO added in the formulation of WPCs, the weaker the mould or algal resistance of WPCs samples was. This was especially true for the surfaces of the specimens that were harshly colonized by mould or algae when the addition amount of ESO was above 15 phr. The colonization speed and intensity of the mould were higher than the algae at the same addition level of ESO, at the same testing time.
3. The hyphae and spores of mould were clearly found on the inner fracture layers, but

not for algae. Additionally, it was deduced that there was a relatively stronger interfacial bonding with fewer voids for the composite specimens after the addition of added 5 phr and 15 phr ESO.

4. It is advised that the addition amount of ESO needs to be controlled when WPCs are applied to outdoor products due to the weak bio-durability.

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