

The Effect of Vermiculite on Flame Retardancy, Physical and Mechanical Properties of Wood Plastic Composites

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The effect of expanded vermiculite (e-VMT) was evaluated relative to the physical, mechanical, and flame retardancy performance of flat-pressed wood plastic composites (WPCs). Various contents of e-VMT (2.5, 5, 7.5, 10, 15%) were added instead of wood flour (WF) to the matrix. Due to the hydrophilic nature of the WF, water absorption (WA) and thickness swelling (TS) of WPCs increased as compared to neat polymer-based panels. Meanwhile, e-VMT incorporation limited the WA and TS values. As expected, the lowest WA and TS values were obtained from the highest e-VMT-reinforced WPCs. The addition of e-VMT was also found to enhance the mechanical properties of WPCs. As the e-VMT content increased and the WF content decreased, there was an improvement in flexural strength, modulus of elasticity (MOE), and tensile strength. Compared to the neat polymer panels, the MOE of WPCs increased by up to 77%. Additionally, the flame retardancy performance of WPCs improved with e-VMT reinforcement, with limiting oxygen index (LOI) values increasing up to 24%. Scanning electron microscopy (SEM) images also demonstrated the favorable integration of e-VMT with matrix, thereby improving the mechanical properties. The inconsistency between WF and polymer was also well-observed, highlighting the tendency of WF to interact easily with water.

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INTRODUCTION

Composites consist of two or more materials that are different from each other (Kim and Pal 2010). The unique properties of these materials are transferred to new materials, which have potential to render the composites superior. Consequently, the combination of various materials reveals robust new materials. One of the most preferred composites is wood plastic composites (WPCs). The preference for WPCs has increased in recent years. The market share of WPCs was estimated at \$6.41 billion in 2022 and is projected to increase further by 2030 (Grand View Research). Furthermore, the application areas have also expanded with growing demand. Initially utilized primarily for outdoor applications, including siding, fencing, garden furniture, windows, door frames, *etc.* (Kal Xu *et al.* 2023), they can now also be considered for construction purposes.

Polypropylene, polyethylene, and polyvinyl chloride are generally employed as thermoplastic polymers for producing WPCs. At the same time, wood flour is the favored lignocellulosic material (Rowell 2007), while other natural fibers are also being evaluated (Talcott *et al.* 2023). Meanwhile, thermoplastic polymers are considered harmful to the environment, whereas wood rises as one of the world's most sustainable, abundant, and green raw materials. Therefore, the incorporation of wood fibers makes WPC a relatively eco-friendly option. This is particularly pertinent given the current global climate change crisis, where consumers increasingly favor a green label. Compared to petroleum-based polymers, wood is less expensive. Therefore, production costs are reduced by adding WF (Ashori 2008).

The combination of WF and polymer has overcome various deficiencies in both materials. Wood is well-known as a hygroscopic material and is recognized for its susceptibility to dimensional changes with humidity (Gardner *et al.* 2015). Moreover, it is vulnerable to biological threats such as insects and fungi (Mai *et al.* 2004). The weathering conditions also affect the surface properties of wood (Williams 2005). On the other hand, the protection of wood fiber through polymer has mitigated the severity of these dangers. Nevertheless, there are still some drawbacks to WPC. The fire performance of WPC is relatively low due to petroleum-based polymers. Moreover, the mechanical properties may be inadequate in some application areas, such as construction (Durmaz *et al.* 2021). Several studies have focused on improving the technological properties of WPC (Rezaei *et al.* 2009; Durmaz *et al.* 2021; Yeole *et al.* 2021).

Vermiculite is a naturally hydrated silicate containing magnesium, aluminum, and iron formed by the transformation of mica (Benli *et al.* 2020). Its porous structure makes it light in weight due to its relatively low density (Shoukry *et al.* 2016). Therefore, it has low thermal conductivity. It is recognized for its effectiveness as a heat shield owing to its excellent insulating properties – due, in part, to its porous structure (Benli *et al.* 2020; Chen *et al.* 2013a; Shoukry *et al.* 2016). Meanwhile, it is recognized as an insulating, high thermal resistivity, and low conductivity material (Li *et al.* 2021). Li *et al.* (2013), organic vermiculite-reinforced WPC provides improved physical and mechanical properties. Zeng *et al.* (2012) stated that the polymer degradation temperature and tensile strength increased with the addition of vermiculite. De Figueiredo *et al.* (2022) also evaluated VMT as a gap filler for conserving wooden objects. However, there are limited studies on vermiculite-reinforced WPC.

In this study, WPCs were reinforced with e-VMT to improve flame retardancy performance, physical, and mechanical properties. Recycled thermoplastic polymers were selected to highlight environmental awareness. The flat-pressed method is preferred for producing WPC panels on a laboratory scale due to the large-size production possibility and cost-effectiveness (Benthien and Thoemen 2012).

The variations in TS and WA values as physical characteristics were examined over 14 days. The influence of e-VMT on flexural strength, MOE, and tensile strength was also examined. Additionally, the flame retardancy effect of e-VMT on the WPC was evaluated by the LOI. The structural integrity of WPC was also investigated via SEM analysis.

EXPERIMENTAL

Materials

The waste Scotch pine wood flour (*Pinus sylvestris* L.) that was used as a lignocellulosic filler had dimensions ranging from 20 to 80 mesh. The fine-grain recycled high-density polyethylene (~250 microns) (Ucar plastic, Izmir, Turkey) was supplied from a commercial supplier as a thermoplastic polymer. HDPE is one of the three most preferred thermoplastic polymers worldwide. Therefore, it is readily available worldwide as a salvaged material. HDPE has a melt flow index (MFI) is 4.5 g/10 min (190°C/2.16 kg), and its density is 0.963 g/cm³. The super fine e-VMT was supplied from a commercial supplier (Organik Madencilik, Sivas, Turkey) with a 0.5=1 mm dimension.

Methods

WPC production

The WF was oven-dried at 90 °C until the moisture content reached 2%. A constant polymer content (60%) was selected, while WF content varied depending on e-VMT contents, which were 2.5, 5, 7.5, 10, and 15% (Table 1). The constituents were mixed with a mechanical mixer (1200 rev/min) to obtain a homogeneous mixture, then with a rotary drum blender (30 to 40 rev/min). The mixture was laid on the aluminum plate. The draft was hot-pressed at 180 °C under a 100-bar pressure for 15 minutes (Fig. 1). The panels were dimensioned with 500 mm x 500 mm x 4 mm, as shown in Fig. 2. The wax paper was used to prevent the sticking of the draft to an aluminum plate. Panels were left to cool under the press slowly. After pressing, panels were conditioned according to ASTM D618 (2021).

Table 1. The Proportions of WPC Constituents

Groups	Wood (%)	rHDPE (%)	MAPE (%)	e-VMT
rHDPE	-	98	2	-
Control	40	58	2	-
2.5%	37.5	58	2	2.5
5%	35	58	2	5
7.5%	32.5	58	2	7.5
10%	30	58	2	10
15%	25	58	2	15

Water absorption and thickness swelling

WPCs' WA and TS values were determined according to ASTM D570 (2022). WPC samples with dimensions of 50 mm x 50 mm x 4 mm were entirely soaked in the water (20±1 °C). Samples were taken out of the water tank, wiped with a soft cloth, and measured at one day, three days, seven days, and 14 days. Five samples were measured for each group.

Mechanical Properties

The flexural strength and MOE were determined according to ASTM D790 (2017). WPC samples with 127 mm x 12.7 mm x 4 mm were tested with a universal test machine (Marestek, Istanbul, Turkey). The tensile strength was determined according to ASTM D638 (2022). Twelve samples were tested for each group for mechanical tests.

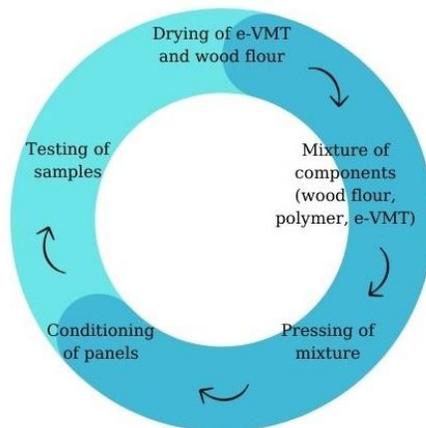


Fig. 1. Production diagram

Limiting oxygen index Test

The LOI test is commonly used to characterize a material's flame retardancy. It is favored as it allows for analyzing small samples. The flame retardancy performance of specimens was determined using LOI tests conducted at Dynisco LOI analyzer instrument (Franklin, USA) according to ASTM D 2863 (2019). The mixture of oxygen and nitrogen was used for combustion during the test. The WPC samples were placed in the center of the glass tube. Afterward, the gas mixture (oxygen and nitrogen) was released to expand into the test environment. Then, the WPC samples were ignited with a pilot fire. The LOI test measures oxygen requirements during combustion caused by flames. An increase in oxygen requirements indicates more excellent fire resistance, whereas a decrease indicates lower resistance. Five samples with 127 mm x 10 mm x 4 mm were tested for each group.

SEM analysis

The structural integrity of WPC plays a crucial role in most technological properties. The cross-section of the samples was investigated by SEM analysis (Zeiss, Evo LS10). The WPC samples were oven-dried until the constant weight, followed by gold-coating of sample surfaces (Emitech, SC7620).



Fig. 2. Flat-pressed WPC panel samples

Statistical analysis

Statistical analysis was conducted to investigate the test data, involving analysis of variance (ANOVA) followed by post-hoc Duncan testing (significance level set at $p < 0.05$). This statistical investigation allowed for revealing the differences between the variables, which makes the test data meaningful.

RESULTS AND DISCUSSION

Physical Properties

This study investigated the impact of e-VMT content on WPC's TS and WA values, as outlined in Table 2. The WA values ranged from 0.08 to 11.98, while the TS values ranged from 0.07 to 7.42. The WA and TS values increased with prolonged exposure time, as it is common knowledge that the hydrophilic nature of wood fiber increases both WA and TS (Faure *et al.* 2019; Borysiuk *et al.* 2020). The control groups (without e-VMT) yielded the highest WA and TS values, as hydroxy groups in the chemical structure of wood fiber make it more vulnerable to water (Adhikary *et al.* 2008; Chen *et al.* 2008). Therefore, incorporating wood fiber into the polymer increased the values of WA and TS compared to rHDPE, which is hydrophobic.

Table 2. The WA and TS values of WPCs

Groups	Water Absorption (%)				Thickness swelling (%)			
	1	3	7	14	1	3	7	14
rHDPE	0.08	0.26	0.28	0.46	0.07	0.15	0.24	0.32
Control	4.70	7.96	11.12	11.98	3.48	5.75	7.20	7.42
2.5%	4.50	7.82	10.87	11.76	3.40	5.64	7.06	7.33
5%	3.32	6.53	9.16	11.16	2.85	4.50	6.30	7.09
7.5%	3.62	6.45	9.37	10.62	2.83	4.61	6.13	6.66
10%	3.42	6.06	9.10	10.57	2.74	4.83	6.11	6.78
15%	3.15	5.40	8.35	10.16	2.21	3.96	5.36	6.51

It is crucial to have a strong adhesion between the polymer and wood fiber to prevent water molecules from easily penetrating and bonding with hydroxyl groups. The coupling agent was added to the matrix to improve the bonding. The anhydride groups in the coupling agent react with the -OH groups in the cell wall, and ester linkages are thereby formed (Rowell 2012). Hence, the bonding between polymer and wood fiber was much more robust. Incorporating e-VMT into the matrix instead of wood fiber improved the water resistance of WPC. As the content of e-VMT increased, both the WA and TS values were observed to decrease. The WPC specimens containing 15% e-VMT had the lowest WA and TS values. After 14 days, a decrease in WA values of 15% was observed, while the decrease in TS values was 12% compared to the control. Li *et al.* (2013) suggested that vermiculite acts as a barrier against water, effectively decreasing WA values. It is believed that the porous structure of e-VMT enhances the interfacial bonding with the polymer, ultimately limiting water flow and penetration (Zeng *et al.* 2012). Therefore, the increase in WA and TS values was restricted by e-VMT acting as a barrier to water. The decrease in the WF content with increasing e-VMT also played an essential role in reducing WA and TS values.

Mechanical Properties

The effect of e-VMT on the mechanical properties of WPC was investigated; results are summarized in Table 3. Incorporating WF into the polymer decreased mechanical properties compared to rHDPE, specifically a decrease of up to 43% for flexural strength and 46% for tensile strength were measured. This reduction can be attributed to the contrast in properties between the wood and polymer, which prevents effective load transfer in the matrix, resulting in weakened adhesion and, subsequently, lower strength. Previous studies also highlighted that the chemical differences between the components as the polar structure of wood and the apolar structure of the polymer, make chemical bonding difficult (Durmaz 2022; Ndiaye *et al.* 2011). As stated above, coupling agents play a crucial role in reducing incompatibility. Nonetheless, the addition of e-VMT demonstrated a favorable influence on mechanical properties, specifically a significant boost in flexural strength. The highest flexural strength was obtained from WPCs containing 15% e-VMT. The comprehensive statistical investigation also showed the differences among the variances. The MOE of the WPC was significantly improved up to 77% compared to rHDPE with the addition of e-VMT and WF. This is attributed to the limitations imposed on the polymer chain's mobility, resulting in an overall improvement in performance.

Table 3. Mechanical Properties of WPC Specimens

Group	FS	MOE	TS
rHDPE	31.63a	1167a	17.44a
Control	17.90b	1450b	9.36b
2.5%	17.92b	1477b	9.39b
5%	18.55bc	1846c	9.43b
7.5%	18.86bc	1781c	9.41b
10%	18.54b	1857c	9.78b
15%	19.57c	2063d	10.77c

Note: Letters indicate the differences ($P < 0.05$) between groups depending on the Duncan test.

Similar results were also obtained for tensile strength. The addition of WF decreased the TS values compared to rHDPE. Meanwhile, the increase in the e-VMT content with decreasing WF improved the tensile strength, as well as flexural strength. The highest tensile strength was obtained from WPC containing 15% e-VMT. It is considered that the porous structure of e-VMT is vital for improved mechanical properties. The polymer could penetrate the porous structure of e-VMT and improve mechanical anchorage bonding, which resulted in increased mechanical properties. The well-bonding is essential for stress transfer. Zeng *et al.* (2012) also stated that the addition of vermiculite made the fracture surface of composites porous, which is better for mechanical properties. Li *et al.* (2013) highlighted that homogeneous dispersion of vermiculite improved stress transfer, significantly enhancing mechanical properties.

LOI Tests

The performance of e-VMT on the effect of flame retardancy was investigated by the LOI test. Oxygen is an essential element in combustion. As can be seen in Fig. 3, the LOI test determined the oxygen needs for e-VMT-reinforced WPC, which were between 17.5 and 24%. Due to its petroleum-based structure, the rHDPE demonstrated the lowest fire performance (17.5%). The polymer chain degradation takes place easily and rapidly during the flaming combustion process, in which some free radicals such as alkyl or alkyl peroxide are revealed (He *et al.* 2012). These radicals transform into CO, CO₂, and H₂O,

which decrease flame resistance due to low char formation. Moreover, the low char formation contributes to an increase in the dropping of the polymer during flaming combustion, as shown in Fig. 4. The flaming droplets then flow and disperse the flame, further accelerating the degradation process.

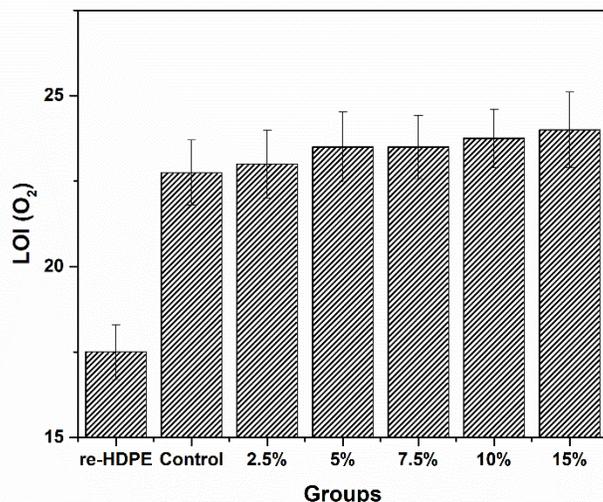


Fig. 3. The LOI values of WPC specimens



Fig. 4. The LOI samples after testing

On the other hand, the incorporation of WF into the polymer resulted in an increase in LOI values. There was an increase of nearly 35% thanks to WF, which has a low heat release rate (Kozłowski and Władyka-Przybylak 2008). Contrary to rHDPE, the carbonization of the wood surface enhances the char formation, which restricts O₂ penetration and heat conduction. Therefore, the addition of WF enhanced the flame retardancy. Meanwhile, only a limited improvement was achieved with the addition of e-WMT. However, the highest LOI value (24%) was obtained from specimens containing 15% e-VMT. It is considered that e-WMT replaced the WF as its concentration increased in the matrix while the polymer remained stable. Additionally, e-VMT has a lower permeability coefficient (Wang *et al.* 2016), making it a barrier against oxygen and improving fire resistance. Consequently, the porous structure of e-VMT restricted heat conduction and oxygen circulation. Moreover, dripping occurred exclusively in e-VMT,

which played a crucial role dispersion of flame. Yan *et al.* (2017) also stated that the non-homogeneous dispersion of e-WMT could result in a decrease in LOI values over 10%. Moreover, Chen *et al.* (2013b) highlighted that the higher e-WMT content created a diffusion barrier for certain gases during polymer degradation, hindering char formation and resulting in lower flame retardancy performance.

SEM Analysis

The effect of e-VMT on the structure of WPCs was investigated through SEM analysis. The e-VMT appeared as a flake-like structure within the matrix, as shown in Fig. 5. The addition of WF and e-VMT resulted in a more porous structure as compared to rHDPE. However, the incorporation of e-VMT into the matrix positively affected the structural integrity. The good bonding between polymer and e-VMT is clearly evident in the images. Therefore, a remarkable coherence between the polymer and e-VMT was observed. In a previous study, raw e-VMT was investigated by SEM analysis and characterized as an accordion-like shape (Sutcu 2015). The porous e-VMT structure improved integration through polymer penetration into the gaps of e-VMT, which affected the mechanical bonding, as can be seen in Fig 5e.

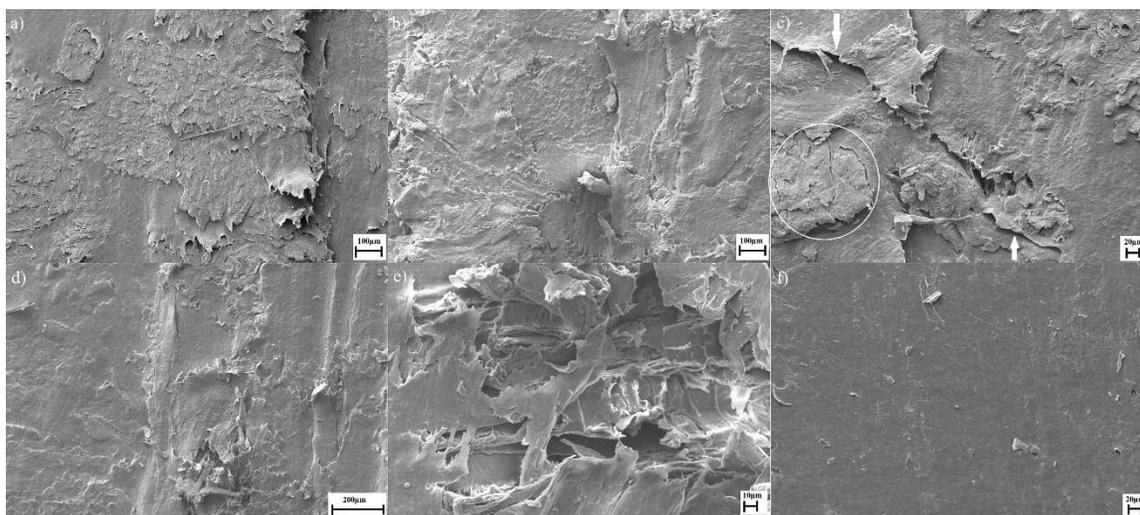


Fig. 5. The SEM images of WPCs: a) 2.5% e-VMT, b) 5% e-VMT, c) 7.5% e-VMT, d) 10% e-VMT, e) 15% e-VMT, f) neat-rHDPE

A good bond between the components is crucial for stress transfer. The flake-like structure of e-VMT is integrated well with the polymer (Fig. 5a), leading to improved stress transfer compared to WF. On the contrary, the inconsistency in surface affinity between wood (polar) and polymer (apolar) resulted in poor mechanical properties. The differences in chemical structure are clearly seen in Fig. 5c (white circle). The weak adhesion between the WF and polymer indicates why there was low mechanical strength. Moreover, the incompatibility is also a reason for capillary gaps between the polymer and WF. As a result, water molecules easily penetrate from these areas to WF, resulting in swelling. Therefore, as stated above, WA and TS values increased with increasing exposure time. Contrary to these, the physical and mechanical properties improved with increasing e-VMT content due to strong bonding.

CONCLUSIONS

1. The hydrophilic nature of natural fibers is well-recognized, which was also observed in this study. On the other hand, the incorporation of expanded vermiculite (e-VMT) into the matrix instead of wood flour (WF) resulted in a significant improvement in dimensional stability. Moreover, water absorption (WA) and thickness swelling (TS) values decreased with increasing e-VMT content.
2. The flexural strength increased by 43%, while the tensile strength increased by 46% with the incorporation of e-VMT. The modulus of elasticity (MOE) of wood plastic composite (WPC) also improved by 77% with the incorporation of e-VMT. The improvement in the mechanical properties was aligned with increasing e-VMT content.
3. The structural integrity was also investigated by scanning electron microscopy (SEM) analysis. The low adhesion between wood and polymer was observed, while e-VMT exhibited good bonding with the polymer. As the WF content decreased, the structural integrity improved, resulting in better stress transfer during loading and improved mechanical properties. A similar phenomenon was evident in physical properties. The favorable bonding of e-VMT restricted the water penetration, which means e-VMT acted as a barrier and improved the dimensional stability.
4. The neat recycled high-density polyethylene (rHDPE) had the lowest LOI value. Adding WF to the polymer significantly increased the LOI values (23.8%). This is due to the carbonization of the wood fiber surface, which obstructed oxygen penetration, leading to an enhancement in LOI values. Moreover, there was also an improvement as WF was substituted by e-VMT. The porous structure of e-VMT hinders heat transfer, which provides a retardant effect during fire.
5. This study demonstrated that e-VMT has the potential as a valuable replacement for WF in the production of WPCs. The incorporation of e-VMT allows for enhancement in physical and mechanical properties and flame retardancy. This research is promising in addressing the drawbacks of conventional WPCs and providing an eco-friendly solution, particularly in the face of growing concerns related to global climate change and sustainable material choices.

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