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Effects of low concentration coupling agent addition on the physicomechanical behavior of wood fiber/HDPE composite

Jovale Vincent Tongco *

Department of Forest, Rangeland and Fire Sciences, University of Idaho, Moscow, ID, 83844 USA

Abstract: In this study, a simple and efficient preparation method of wood-plastic composites (WPC) from maple wood flour (filler) and high-density polyethylene (HDPE) through extrusion was conducted, and the effects of low concentration (2% w/w) coupling agent addition (maleated polyethylene, MAPE) on their physical and mechanical properties were tested. Tensile test, dimensional stability (water absorption) test, melt flow rate test, and differential scanning calorimetry (DSC) analysis were utilized to assess the physicomechanical properties of the finished composite products. The tensile test quantified the ability of the materials to withstand pulling forces and resulting deformation before breakage. An improvement in stiffness and resistance to breaking under stress was observed. There was a statistically significant difference between WPC and WPC+MAPE in terms of flexural strength and modulus ($p < 0.05$) but no statistically significant difference between the same samples in terms of strain at breakage ($p > 0.05$). There was also no observed difference in the melt flow rate and enthalpy functions of WPC and WPC+MAPE. The minimal addition of a coupling agent (MAPE) to WPC significantly improved dimensional stability, as evidenced by 13% lower thickness swelling and 3% lower mass gain through water absorption.

Keywords: Wood; Wood-plastic composites; Physicomechanical properties; Dimensional stability, HDPE.

1. Introduction

Wood is a versatile renewable material used in various applications, such as construction, furniture, and handicraft manufacturing. Wood is a reliable material regarding mechanical properties, but its organic nature makes it susceptible to degradation, fire, and biological attack. Plastics, conversely, are synthetic or semi-synthetic materials comprising polymers and can be easily molded and processed into different shapes and sizes according to specific applications. Challenges arise in processing and recycling waste plastic materials because of their chemical nature. Plastics are designed to be chemically inert and thermally resistant to serve their purpose. New recycling technologies are recently gaining attention to reduce the amount of plastic waste generated into the environment. A compromise can be attained by manufacturing wood-plastic composites (WPC). WPC is a hybrid material of wood particles and thermoplastic polymers [1]. Wood acts as a fibrous filler material, providing advantages such as better mechanical properties (stiffness and rigidity), lower cost, aesthetic properties of wood, being environmentally friendly, and reducing plastics in the material.[2,3]. Meanwhile, adding plastics imparts better molding capabilities, chemical and moisture resistance, and the ability to use waste plastics as components [4,5]. A study on using wood flour from European Redwood (*Pinus sylvestris* L) to reinforce WPC utilizing polypropylene as the matrix material due to its high crystallinity, stiffness, hardness, and strength [6]. Their work focused on the relationship between the effects of the milling time of the Redwood flour on its dispersion properties and its influence on the mechanical properties of the product WPC. WPCs are mainly produced through extrusion [7,8]. The extrusion process involves several steps. First, the wood fiber material is reduced to smaller particles using a 40 – 120-size mesh, depending on the product specifications and applications. The wood fibers were dried before extrusion and mixed with the polymer (i.e., high-density polyethylene, HDPE) as the plastic component. Other components and additives can also be added to manipulate the chemical and thermal properties of the finished product [9,10]. Figure 1 shows a general process schematic to produce WPC, highlighting the formation of wood flour, mixing with polymer (resin), compounding (additives), extrusion, cooling, and sawing. In some cases, solvent extraction using non-polar solvents is also employed to minimize the effects of hydrophobic components in wood and natural fibers, such as extractives, fatty acids, waxes, alcohols, sterols, glycerides, and suberin, that could otherwise react with the resin and additives incorporated into the production of WPC, affecting its mechanical performance [11–14].

*Corresponding author: Jovale Vincent Tongco
Email address: jtongco@uidaho.edu
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The objectives of the study deal with the production of WPCs from maple wood flour and HDPE and investigate the effects of minimal coupling agent addition (maleated polyethylene, MAPE) on the physical and mechanical properties of the product WPC. Maple wood fiber was used in this study due to its renewable nature. It is valued for its timber and sap and is known for its relatively quick growth rate and wide availability [15]. Wood and HDPE are inherently incompatible due to their different surface chemistries, as wood is hydrophilic, while HDPE is hydrophobic. MAPE acts as a coupling agent or compatibilizer, creating bonds between the wood and the polyethylene matrix [16].

Along with processing time and temperature, coupling agents in optimal concentrations improve the mechanical performance of WPC by facilitating the dispersion of wood flour into the polymer matrix and creating an interfacial interaction between them [17]. Another advantage of using MAPE as a coupling agent is improved moisture resistance. MAPE interacts with the hydrophobic polymer and hydrophilic wood fibers through the olefinic chain and glycidyl or anhydride group [18], resulting in improvements in the interfacial adhesion between the polymers and wood fibers, leading to enhanced properties of WPC, such as increased mechanical strength and decreased water absorption [19]. Several studies have been done concerning the production of WPC from maple fiber and HDPE with MAPE as a coupling agent [20,21]. The present study explored the possibility of adding minimal amounts of MAPE (2% w/w) and quantifying its effect on the physicomechanical properties of the WPC, specifically dimensional stability, which is lacking in the literature. Tensile tests are a series of mechanical tests utilized to evaluate the ability of a material to resist "pulling" forces by applying a force opposite and parallel to the sample. The water absorption test measured the dimensional stability of the WPC samples (shrinkage/swelling). The melt flow rate test quantifies the flow characteristics of molten plastics (HDPE and WPC). Lastly, differential scanning calorimetry (DSC) was employed to measure the thermal properties of HDPE and WPC and determine the materials' % crystallinity (Xc). This refers to the portion of a polymeric material that exists in an ordered state, as opposed to its disordered (amorphous) state, which significantly impacts its physicochemical characteristics, influencing its strength and elasticity.

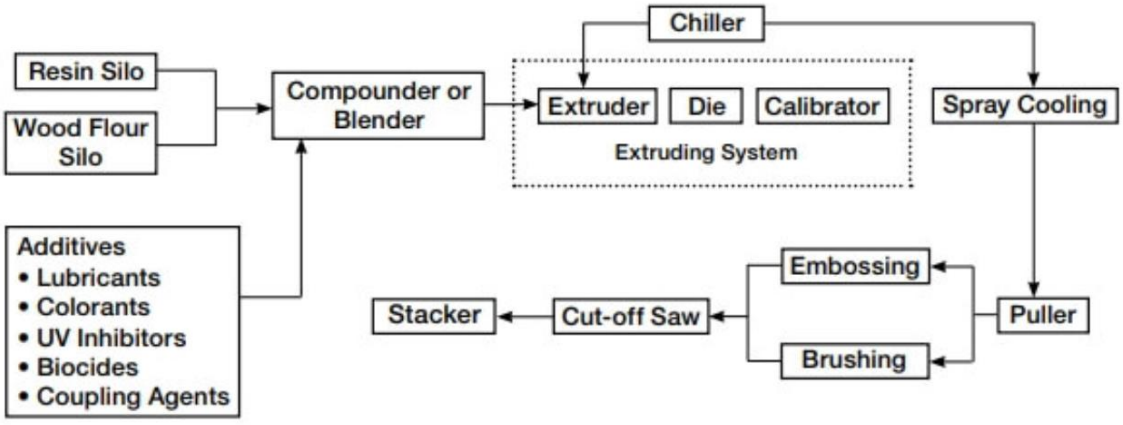

Figure 1. A general process flow diagram for WPC production [22].

2. Experimental

2.1 WPC Preparation

Two different formulations were extruded, with and without the addition of coupling agent (MAPE). The moisture content of the wood fiber (maple wood) used was determined by using an HB43-S Halogen Moisture Analyzer (Mettler Toledo, Columbus, OH, USA), and the mass required per WPC was calculated. The wood flour was screened through a commercial flour sifter to obtain finer particles. Table 1 shows the formulations used in the study.

Table 1. Formulations for WPC production.

Component	w/ MAPE	w/o MAPE
Maple wood flour (dry)	50%	50%
HDPE	48%	50%
MAPE	2%	-

The pre-mixed formulation was added to the feeder of the Leistritz 18 mm co-rotating twin screw extruder (Leistritz, Allendale, NJ, USA). The barrel temperature was set at 160°C and screw speed at 200 rpm. WPC ribbon dies were produced after extrusion and then used for subsequent tests. The produced

WPC ribbons were sanded flat on a belt sander to smoothen the surface. Small pieces were cut from the sanded extruded WPC ribbons for a water absorption test (20 mm x 50 mm). A portion was used to prepare dogbone test strips (165 mm x 20 mm) for tensile test using a Dremel router. HDPE samples were prepared using an injection molding machine.

2.2 Tensile test

Six replicates of each WPC formulation and three for HDPE were performed following ASTM D 638 [23] using an Instron 5500R-1122 universal testing machine (Instron, Norwood, MA, USA). The molded dogbone specimens were fitted into the testing zone between the spans. The tensile strength, modulus, and breakage at strain values were obtained and recorded. The resulting data was processed using Bluehill® v3 Instron software.

2.3 Water absorption test

The WPC, WPC+MAPE, and HDPE samples were subjected to a water absorption test by measuring the initial dimensions (50 mm x 20 mm), including thickness, and then weighed at 0.0000 g resolution. The samples were soaked in 700 mL of deionized water (1 L beaker) for 10 days. Every 24 h, the samples were removed from the beaker, with the excess water wiped off. The samples were weighed, the dimensions were measured, and the beaker was placed back in place until the test was over. The test was done in triplicate.

2.4 Melt flow rate test

The melt flow rate was determined using a CEAST model 7024 melt flow indexer (CEAST, Pianezza, TO, Italy) following ASTM 1238 procedure B [24]. For WPC samples, a 15 kg load was applied at a barrel temperature of 190°C. The melt flow was conducted for 10 mins. For HDPE (reference), the same load and barrel temperature were employed, but only for 2 mins. The test was done in triplicate.

2.5 Differential scanning calorimetry (DSC)

The thermal properties of WPC and HDPE samples (5 – 7 mg) were measured from 20 – 160°C with a heating rate of 10°C/min using a PerkinElmer DSC-7 instrument (PerkinElmer, Waltham, MA, USA) [25]. After reaching 160°C, the samples were cooled down to 20°C (3 min), then heated back to 160°C at 10°C/min (second cycle). The melt (T_m on the 1st and 2nd heating cycles) and crystallization (T_c on the cooling cycle) temperatures of the samples were obtained from the DSC curves. The % crystallinity (X_c) of the original HDPE and WPC was calculated, considering the actual HDPE contents in the given sample. X_c was calculated using equation (1).

$$X_c = \frac{\Delta H_s}{\Delta H_m} \, x \, 100 \qquad (1)$$

Where:

ΔH_s = Sample melt enthalpy

ΔH_m = 293 J/g (The heat of melting of 100% crystalline polyethylene)

2.6 Statistical Analysis

The results between different WPC formulations (with and without MAPE) were compared by measuring the mean and standard deviation (within the same formulation) and by employing a t-test (between formulations).

3. Results and Discussion

Wood-plastic composites (WPC) are composite materials composed of wood as the main filler and plastic as a matrix. Like other composite materials, the materials used in WPC are preserved so that the original properties are incorporated or modified into the WPC, leading to improved physical and mechanical properties at a fraction of the cost [26]. Combining wood-based elements with polymers can be easily attained by employing various manufacturing processes such as extrusion, injection molding, and pressing (thermoforming). Wood undergoes degradation at around 220°C, so it is imperative to utilize polyvinyl chloride and polyethylene, which can be easily molded and incorporated into the composite at lower temperatures. Wood is hydrophilic due to hydroxyl groups on the cellulose and hemicellulose chains in the structure. Adding plastic to wood fibers in WPCs improves the moisture resistance of the finished product, making it more suitable for applications such as outdoor decks and benches. In the study, maple wood flour was prepared by screening and sifting. It is then mixed with HDPE as a plastic polymer in an extruder to form WPC. MAPE was added to another batch (WPC+MAPE) to measure the effects of a coupling agent on the physical and mechanical properties of the finished products. HDPE was used as a standard for subsequent tests and prepared using an injection molding machine. A portion of the extruded WPC ribbon (Figure 2a) was sanded and used for subsequent tests.

The manufactured WPC ribbons were cooled and preconditioned for at least a week before testing. A universal testing machine was utilized to measure the tensile strength of the finished products. Dogbone test specimens were prepared from the WPC, WPC+MAPE, and HDPE samples for tensile test, as shown in Figure 2b. The specimens were cut to different dimensions for the water absorption test, melt flow rate test, and DSC analysis. The tensile test applies a force opposite and parallel to the specimen to measure the tensile resistance of the products until breakage (Figure 2c). The test was successful in obtaining the flexural strength (MPa), flexural modulus (MPa), and strain (%) at breakage values for each specimen.

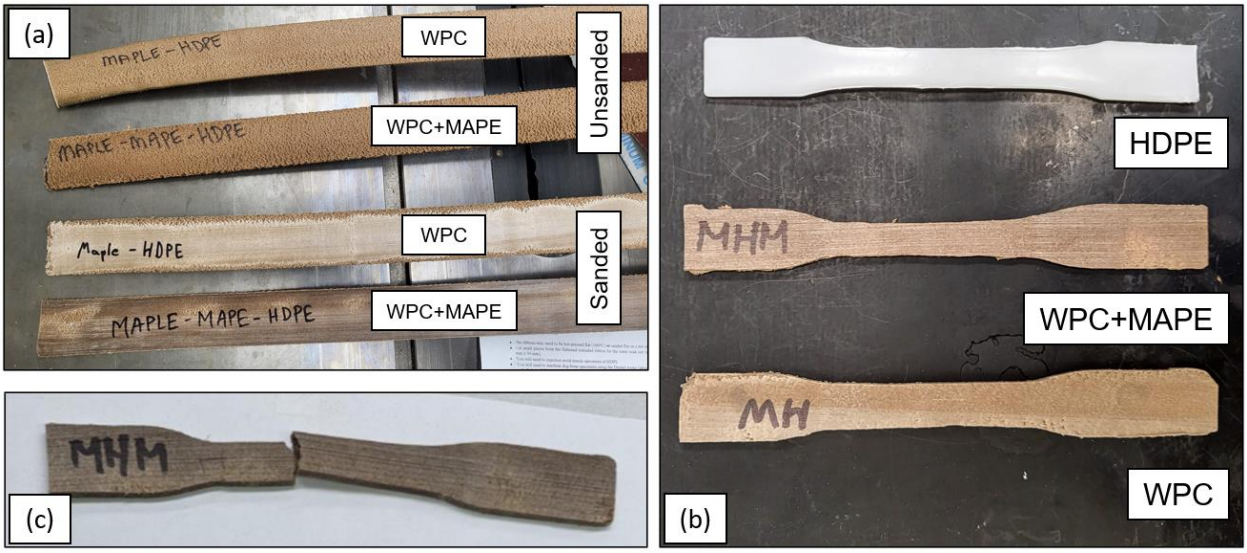

Figure 2. (a) Extruded WPC ribbon products. (b) Dogbone specimens for tensile test (c) and subsequent breakage.

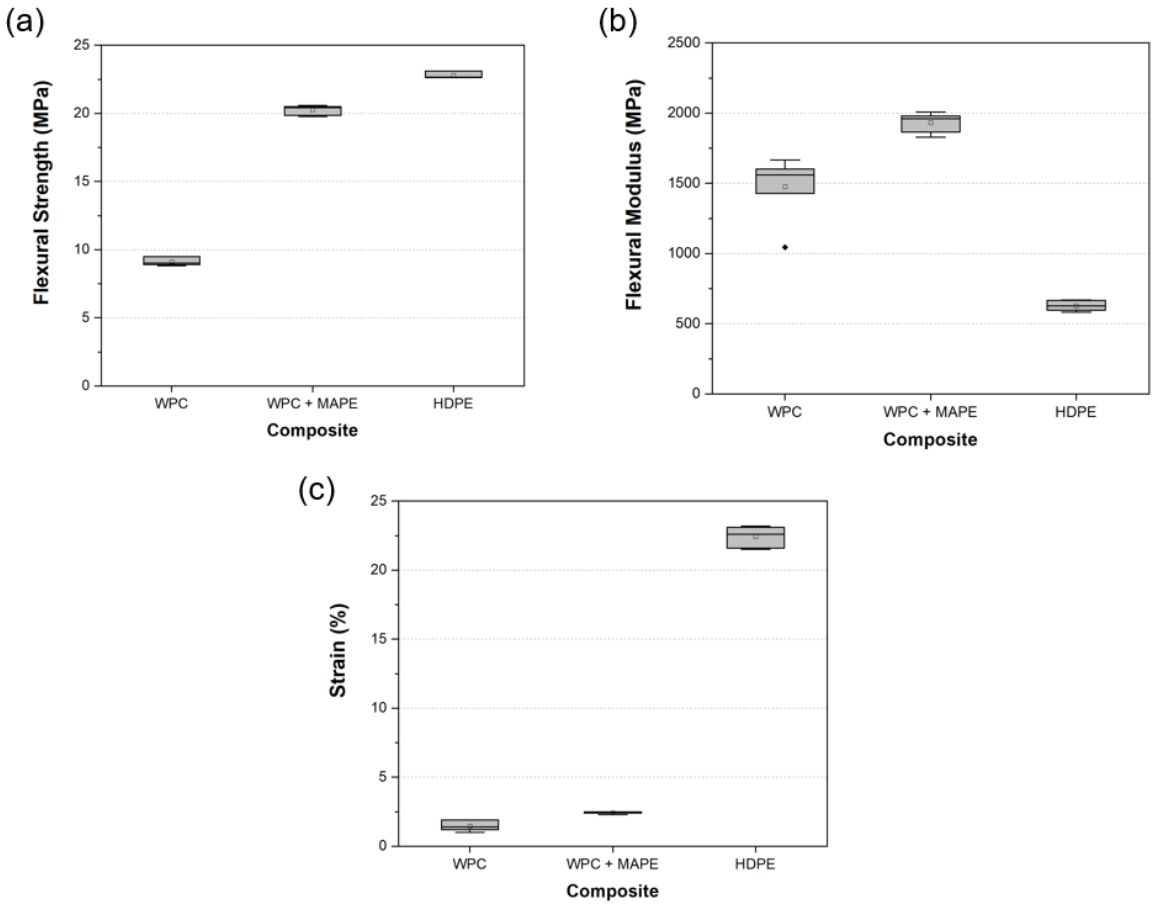

Figure 3. (a) The modulus of rupture and (b) modulus of elasticity of WPC, WPC+MAPE, and HDPE samples are expressed as flexural strength and flexural modulus, respectively—units in MPa. (c) Strain at breakage of the same samples, expressed in %.

Figure 3 highlights the box plots of tensile test results of WPC, WPC+MAPE, and HDPE. Box plots were useful in visually presenting the minimum and maximum values, the mean, and the quartiles of the parameters evaluated on composite samples. Significant differences were in the flexural strength and flexural modulus between WPC and WPC+MAPE, but no significant differences were observed in the strain at breakage. WPC exhibited the lowest flexural strength at 9.1 ± 0.3 MPa, followed by WPC+MAPE at 20.3 ± 0.0 MPa, and HDPE at 22.8 ± 0.3 MPa. This signifies that HDPE withstood the highest stress in bending. WPC was the least in terms of withstanding maximum stress in bending, failing at

just around 50% modulus of rupture for HDPE. Adding a coupling agent (MAPE) improved the modulus of rupture of WPC. For flexural modulus (a measure of the stiffness in bending of a given material), there were differences between WPC, WPC+MAPE, and HDPE modulus of elasticity values at 1476.3 ± 225.8 MPa, 1943.0 ± 70.7 MPa, and 634.3 ± 45.0 MPa, respectively. The addition of MAPE also enhanced the modulus of elasticity of WPC, while HDPE exhibited the lowest stiffness in bending compared to composite materials. Lastly, for the strain at breakage of the materials, WPC, WPC+MAPE, and HDPE afforded the values 1.5 ± 0.4%, 2.4 ± 0.1%, and 22.1 ± 1.0%, respectively. The flexibility of HDPE led to a significantly higher strain value when breaking the material than the composite samples. Statistically, both WPC treatments differed substantially from HDPE in all tensile tests. This is due to the flexibility of HDPE as a plastic ($p < 0.05$). Another study using maple as wood filler compounded with isotactic polypropylene (50:50 w/w) reported a tensile strength of 25 MPa and a tensile modulus of 3000 MPa [27]. Using polypropylene with a higher molecular weight than PE resulted in higher flexural strength and modulus than the present study. Trex, a company manufacturing composites for decking and outdoor furniture, reported a modulus of rupture (tensile strength) of 25.9 MPa and a modulus of elasticity (tensile modulus) of 2757.9 MPa for their products (e.g., Trex Select® and Trex Transcend® Lineage) which are comparable to the values obtained in this study. Their composite decking comprises linear low-density polyethylene (LLDPE) and recycled wood fibers [28].

Portions of the unused samples used for the tensile test were subjected to a water absorption test, melt flow rate test, and DSC analysis. The water absorption test was employed to measure the changes in dimensions and water absorption property (mass gain) of the composites (WPC and WPC+MAPE) and plastic standard (HDPE). Two properties were measured and obtained using the water absorption test, namely, % thickness swelling (%TS) and % mass gain (%MG). Figure 4 illustrates the samples' %TS 10 days after the water absorption test. There was a positive correlation between the two properties, as an increase in %TS corresponds to the rise in %MG due to water absorbed, affecting the samples' dimensions and mass, especially the wood composites. Wood is inherently hydrophilic, and the hydroxyl groups on the wood fibers bind to water molecules even in the presence of a plastic matrix and coupling agent, leading to an observed significant increase in %TS and %MG of the WPC and WPC+MAPE relative to HDPE, which is hydrophobic. There was a significant increase between the first and last days of soaking for WPC and WPC+MAPE samples. The observed increase for %TS of WPC and WPC+MAPE was 55.0 and 41.9%, respectively, while the rise for the same period for %MG of WPC and WPC+MAPE was 32.3 and 29.2%, respectively. Maximum absorption was observed around the 8th day for both %TS and %MG. This result corresponds to previous studies showing that the water uptake (%) for WPC with MAPE as matrix reached a plateau at around 10 days, and for PP-based WPC, reaching maximum absorption (%) at around 200 h (~8 days) [16,29]. WPC exhibited the highest final %TS and %MG due to the nature and concentration of wood fibers in the composite relative to WPC+MAPE and HDPE (hydrophobic). This signifies that a minimal amount of MAPE addition significantly improved the dimensional stability of the WPC. A related study on aspen and HDPE composite reported comparable values for %TS and %MG with the present study, at around 50% and 30% difference between the initial and final (1000 h) period, respectively [30]. Water absorption is important when manufacturing composite materials, especially for outdoor applications like decking. Trex maintains the water absorption of their products to less than 1% [28].

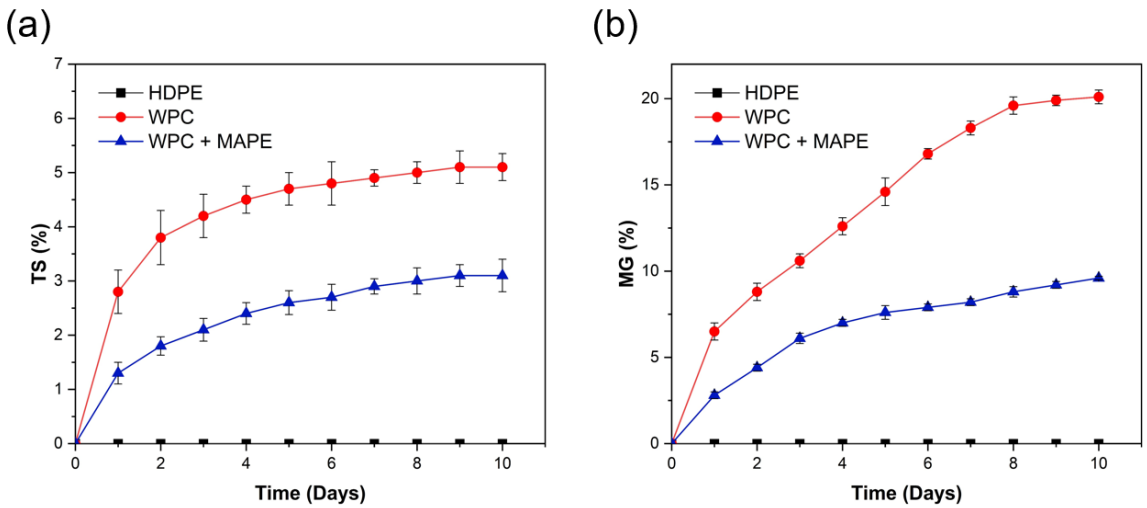

Figure 4. (a) Thickness swelling (%TS), and (b) mass gain (%MG) of WPC, WPC+MAPE, and HDPE during 10 days of water absorption test.

Figure 5 shows the melt flow rate of the samples. HDPE exhibited a low viscosity and high melt flow rate of 46.5 g/min, significantly higher than There was no significant difference between the melt flow rate of WPC and WPC+MAPE ($p > 0.05$). This signifies that the addition of MAPE did not affect the ability of the WPC to flow when melted, indicating no measurable difference in viscosity and molecular weight. The molecular weight distribution of the polymer matrix is one of the most critical parameters that influence the melt flow of composites, as reported by a study measuring the effects of varying 30 – 60% wood flour content during WPC extrusion [31]. A lower melt flow rate indicates higher molecular weight and degree of polymerization for a given material. This enhances the composite's strength, toughness, and thermal properties. However, an extremely low melt flow rate can also cause challenges in WPC processing, like high shear stress, difficulties dispersing additives, and a higher rate of physical degradation.

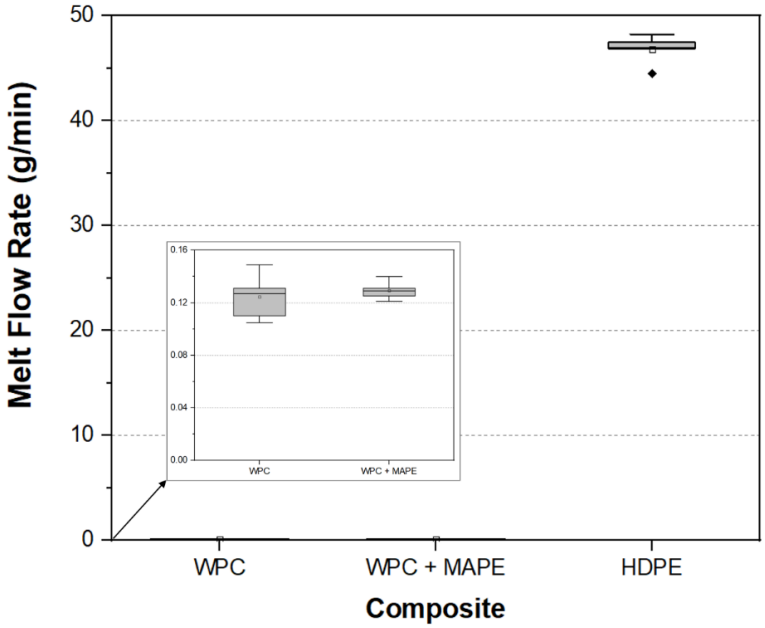

Figure 5. Box plot of the melt flow rate (MFR) of WPC, WPC+MAPE, and HDPE samples. Note the significant difference between the MFR of HDPE and the composite samples. Inset: WPC and WPC+MAPE MFR box plots.

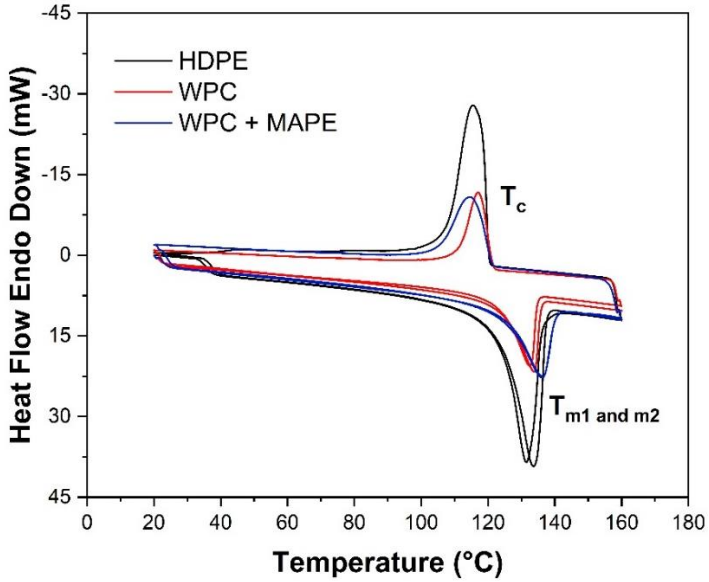

Figure 6. DSC curves of WPC, WPC+MAPE, and HDPE.

Differential scanning calorimetry (DSC) is an analytical technique utilized for measuring the difference in the amount of heat required to increase the temperature of a material against a reference as a function of temperature [32]. The sample and reference materials are maintained at the same temperature throughout the run, and the temperature program can

be set so that the sample holder temperature increases linearly with time.

Figure 6 shows the DSC curves for the WPC, WPC+MAPE, and HDPE samples, highlighting the 1st and 2nd melt ($T_{m1\ and\ m2}$) and crystallization (T_c) temperatures. The melt enthalpy (ΔH_s) was obtained by integrating the area under the melting peak (T_m) and dividing the result by the heating rate and initial sample weight. These calculations were done through the built-in software. The heat of melting (ΔH_m) is a literature value and, for 100% crystalline PE, corresponds to 293 J/g. Another study reported a T_c for maple at 128°C and T_m at 170°C using polypropylene as a matrix [27]. Using polypropylene resulted in higher X_c and T_m than in the present study.

The X_c of the WPC, WPC+MAPE, and HDPE samples were calculated using the enthalpy functions obtained from DSC. Table 2 shows the T_c, $T_{m1\ and\ m2}$, ΔH_s, and ΔH_m, and calculated X_c.There was no significant difference between WPC and WPC+MAPE in terms of T_c, T_m, and ΔH_s. This means that adding a coupling agent (MAPE) does not significantly affect the thermal properties of WPC [33]. The literature value for the X_c of HDPE is around 60%, consistent with the 57% obtained by calculations in this study [34].

Table 2. T_c, $T_{m1\ and}$ T_{m2}, ΔH_s, and ΔH_m, and X_c values of WPC, WPC+MAPE, and HDPE obtained from DSC.

Composite	T_c (°C)	T_{m1} (°C)	T_{m2} (°C)	ΔH_s (J/g)	ΔH_m (J/g)	X_c
WPC	117.1	132.7	133.7	87.9	146.5	60
WPC+MAPE	114.6	136.0	136.4	86.5	140.6	61
HDPE	115.6	131.5	133.5	165.9	293.0*	57

* = Literature value [34,35]

Statistical analysis of the tensile test results was performed to determine if there were statistically significant differences between the WPC formulations (with and without MAPE) by t-test. The p-values of 5.5×10^{-14} for the flexural strength, 8.0×10^{-4} for the flexural modulus, and 1.0×10^{-6} were orders of magnitude below the significance level $\alpha = 0.05$, meaning there is no statistically significant difference between WPC and WPC+MAPE. Therefore, adding a coupling agent (MAPE) at minimal amounts does not significantly affect the flexural strength, flexural modulus, and strain at breakage of WPC.

The intersection of sustainable materials development and climate action represents a critical area of focus in meeting the UN Sustainable Development Goals (SDGs). Recent research provides significant insights into how wood-plastic composites (WPCs) align with these global sustainability objectives. A recent study quantified the greenhouse gas (GHG) emissions from WPC production using recycled plastics, obtaining a baseline GHG emission value of around 3500 kg CO_2e/t [36]. They also found that setting the recycled material rate of plastic materials to 100%, instead of just using new and non-recycled plastics, would reduce the GHG emissions to around 1300 kg CO_2e/t (28% decrease). Complementing this quantitative approach, another recent study positioned WPCs as an essential part of carbon-negative materials [37]. The researchers stated that this can be attained through efficient materials processing, renewable resource utilization, and improved recycling processes. These studies demonstrate that WPC production, particularly those incorporating high percentages of recycled plastics and other materials, represents a significant opportunity for reducing carbon footprints in developing construction materials.

4. Conclusion

Wood-plastic composites (WPC) were produced by mixing maple wood flour (filler), HDPE (plastic matrix), and MAPE (for coupling agent-treated samples only) in an extruder. HDPE samples for testing were prepared using injection molding. The effects of the addition of MAPE on the mechanical and physical properties of the finished composites were investigated in terms of tensile strength, thickness swelling (%TS), and mass gain (%MG) through water absorption, melt flow rate, and thermal properties (DSC). The results of the study revealed that there were significant differences between WPC and WPC+MAPE in terms of flexural strength and modulus, signifying an improvement in terms of stiffness and resistance to yielding under bending stress ($p < 0.05$), but no statistically significant differences between the WPC treatments in terms of strain at breakage (resistance to deforming under stress) as evidenced by employing t-test ($p > 0.05$). There was also no significant difference between WPC and WPC+MAPE in terms of melt flow rate as measured by melt flow index rheometer and enthalpy functions as demonstrated by DSC ($p > 0.05$). Adding low-concentration MAPE (2% w/w) to WPC as a coupling agent improved dimensional stability, yielding a lower %TS and %MG (water absorption) at 13% and 3%, respectively. The study recommends using recycled materials to reduce the carbon footprint of WPC production.

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Conflicts of interest

The author declares no conflicts of interest.

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