

Full Research Paper

Determination of Thermal Properties and Morphology of Eucalyptus Wood Residue Filled High Density Polyethylene Composites

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Received: 16 January 2008 / Accepted: 25 January 2008 / Published: 4 February 2008

Abstract: Thermal behaviors of eucalyptus wood residue (EWR) filled recycled high density polyethylene (HDPE) composites have been measured applying the thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). Morphology of the materials was also studied using scanning electron microscope (SEM). Addition of the EWR into the recycled HDPE matrix reduced the starting of degradation temperature. EWR filled recycled HDPE had two main decomposition peaks, one for EWR around 350 °C and one for recycled HDPE around 460 °C. Addition of EWR did not affect the melting temperature of the recycled HDPE. Morphological study showed that addition of coupling agent improved the compatibility between wood residue and recycled HDPE.

Keywords: Thermogravimetric analysis, differential scanning calorimetry, scanning electron microscope, high density polyethylene, coupling agent.

1. Introduction

In the last twenty years, the use of lignocellulosic fibers as filler in the plastic industry has increased progressively [1]. Thermoplastics are combined with lignocellulosic (wood) fillers to produce wood-plastic composites (WPC). They provide several advantages such as low densities, low cost, nonabrasive nature [1-4], possibility of high filling levels, low energy consumption, high specific properties, biodegradability, availability of a wide variety of fibers throughout the world, and generation of a rural/agricultural-based economy [5-6]. In addition, the utilization of recycled plastic

and waste wood in the manufacturing of such composites may help the environment by reducing the land filling and/or promoting the recycling.

First patented application of WPC was to manufacture of automotive interior substrates using extruded polypropylene and wood flour [1,7-8]. After this Italian patent, other companies started to produce various shapes of automotive parts by extruding polypropylene and wood flour mixtures. In recent times, WPCs have found other application areas including siding, fencing, window frames and decking [1,7-12] by using polypropylene as well as high density polyethylene (HDPE) and polyvinyl chloride (PVC) as matrix resins.

Several studies were conducted to evaluate the performance of WPCs. Most of them has focused on to improve the compatibility between hydrophobic (non-polar) thermoplastics and hydrophilic (polar) wood flours [1,11-14]. In order to enhance similarity and adhesion between wood-flours and thermoplastic matrices, several chemicals have been employed [15-24]. Lu et al. [25] classified the maleic anhydride grafted polyolefin as the most suitable chemicals for polyolefins. The effects of coupling agents on the mechanical properties of HDPE and PP based wood composites were investigated extensively [26-33]. A few researches were conducted to investigate the thermal and morphological behavior of WPCs. It is the purpose of this study to understand the behavior of the thermoplastic matrices with lignocellulosic filler such as eucalyptus wood residues. Study also investigated the effect of maleic anhydride grafted polyethylene as a coupling agent on the mechanical properties and the morphology of the composites.

2. Results and Discussion

2.1. Thermogravimetric analysis (TGA) and Differential scanning calorimetry (DSC) results

TGA analysis was performed on eucalyptus wood residue (EWR), unfilled recycled HDPE (A), EWR filled recycled HDPE composites (B), and EWR filled recycled HDPE composites modified with MAPE coupling agent (C). Figure 1 shows the TGA thermographs of the samples. Initial degradation of EWR was started around 220 °C while recycled HDPE started around 300 °C. Composite materials started degrading 250 °C and 260 °C for sample B and C, respectively. Addition of EWR into the thermoplastic matrix induced recycled HDPE to degrade thermally at low temperatures [34]. It is reported that thermal degradation of wood produces free radicals which accelerate the reaction of thermal degradation of polyethylene at high temperatures [34-35]. Figure 2 shows the TGA and DTGA thermographs of the sample B. The two main decomposition peaks was shown with arrows on DTGA thermograph. The first peak was around 350 °C while the second peak was around 450°C. It is believed that first peak of 350 °C in composite B was mainly coming from the degradation of EWR. The main decomposition temperature of EWR was 360°C (Table 1). Second decomposition temperature peak for HDPE based composites was close to the decomposition temperature of recycled HDPE (470 °C). Similar results were also observed for sample C.

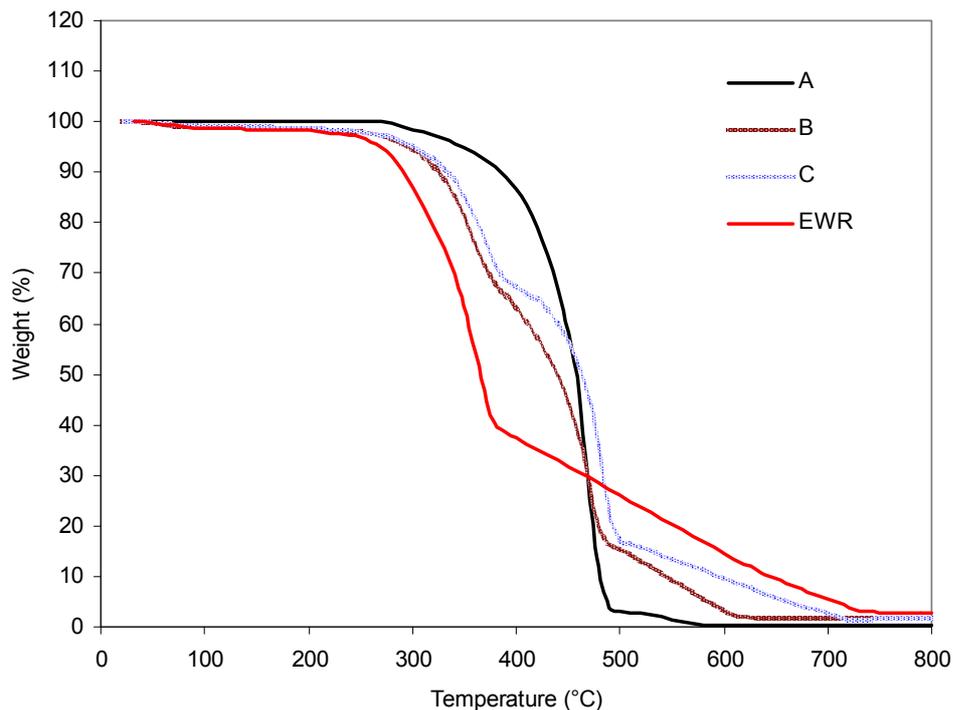


Figure 1. TGA thermographs of unfilled recycled HDPE (A), EWR filled recycled HDPE composites (B), EWR filled recycled HDPE composites modified with MAPE coupling agent (C) and eucalyptus wood residue (EWR).

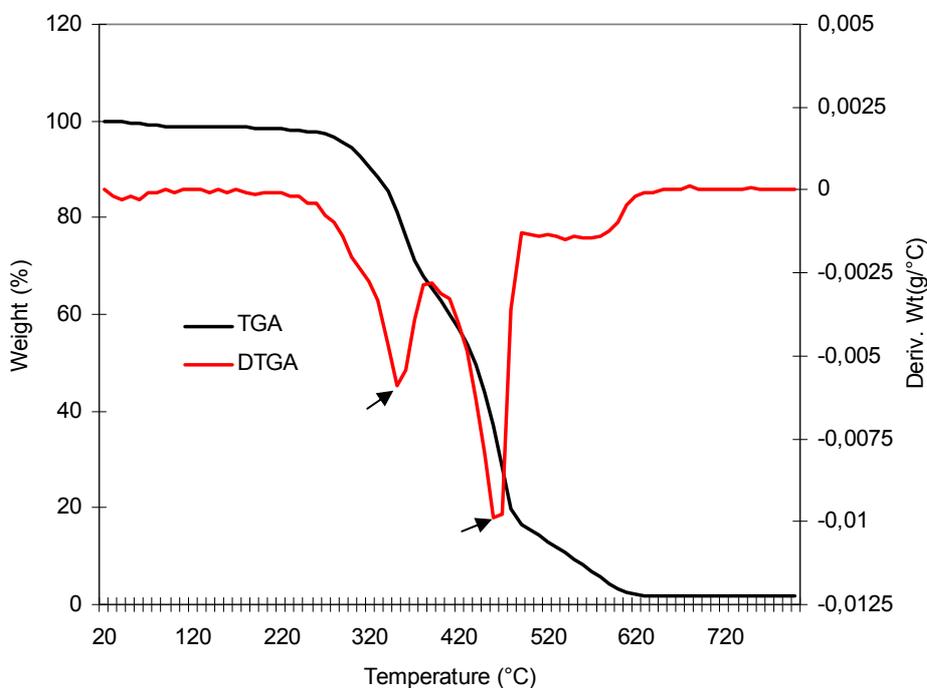


Figure 2. TGA and DTGA thermographs of the eucalyptus wood residue filled recycled HDPE composites.

Summary of onset degradation, peak temperature and residual weight after 500 °C of the materials are also given in Table 1. When the residues of the samples at 500 °C were compared, EWR and recycled HDPE had 26-percent and 3-percent residues, respectively. In composite B and C, residues were around 16-percent and 17-percent, respectively. Addition of EWR into the recycled HDPE increased the remaining residues due to the charring of EWR.

Table 1. Thermogravimetric data for eucalyptus wood residue (EWR), recycled HDPE (A), EWR filled recycled HDPE composites (B), EWR filled recycled HDPE composites modified with MAPE coupling agent (C).

<i>ID</i>	<i>Onset Temperature (°C)</i>	<i>Peak Temperature (°C)</i>		<i>Weight Loss (%)</i>		<i>Residue after 500 °C (%)</i>
EWR	315 (2.4) ¹	360 (7.0)		46 (1.5)		26 (3.0)
A	434 (1.2)	470 (0.8)		71 (2.0)		3 (2.9)
		<i>1st peak</i>	<i>2nd peak</i>	<i>1st peak</i>	<i>2nd peak</i>	
B	404 (1.0)	350 (0.2)	450 (0.15)	19 (0.41)	56 (1.53)	16 (1.0)
C	429 (0.52)	360 (0.07)	480 (0.2)	19 (0.44)	66 (1.32)	17 (0.1)

¹ The value in parenthesis is the standard deviation.

Thermal properties of the materials based on DSC results were presented in Table 2. DSC thermographs of the samples were shown in Figure 3. Melting temperature of the recycled HDPE and their composites was around 128-130°C.

Table 2. Thermal properties of DSC for the recycled HDPE (A), eucalyptus residue filled recycled HDPE composites (B) and eucalyptus wood residue filled recycled HDPE modified with MAPE coupling agent (C).

<i>ID</i>	<i>Wood Content (%)</i>	<i>HDPE Content (%)</i>	<i>Melt Temperature (°C)</i>	<i>Melt Enthalpy ΔH_m (J/g)</i>
A	-	100	130	152
B	50	50	129	88
C	50	46+4	129	72

Based on the TGA and DSC analysis during the manufacturing of the composites, extruder temperatures should be over 129 °C for HDPE to facilitate the melting of the matrix and should be less than 220 °C to prevent the lignocellulosic material from degrading. It should also be noted that residence time of the material in the extruder is also important [36]. Higher processing temperature can be set if the component passes through the extruder in a short time.

2.2. Effect of MAPE Coupling Agent on Mechanical Properties

Table 3 summarizes the mechanical properties of eucalyptus wood residue (EWR) filled recycled thermoplastic composites. Mechanical properties were discussed under three headings; tensile properties, flexural properties and impact properties.

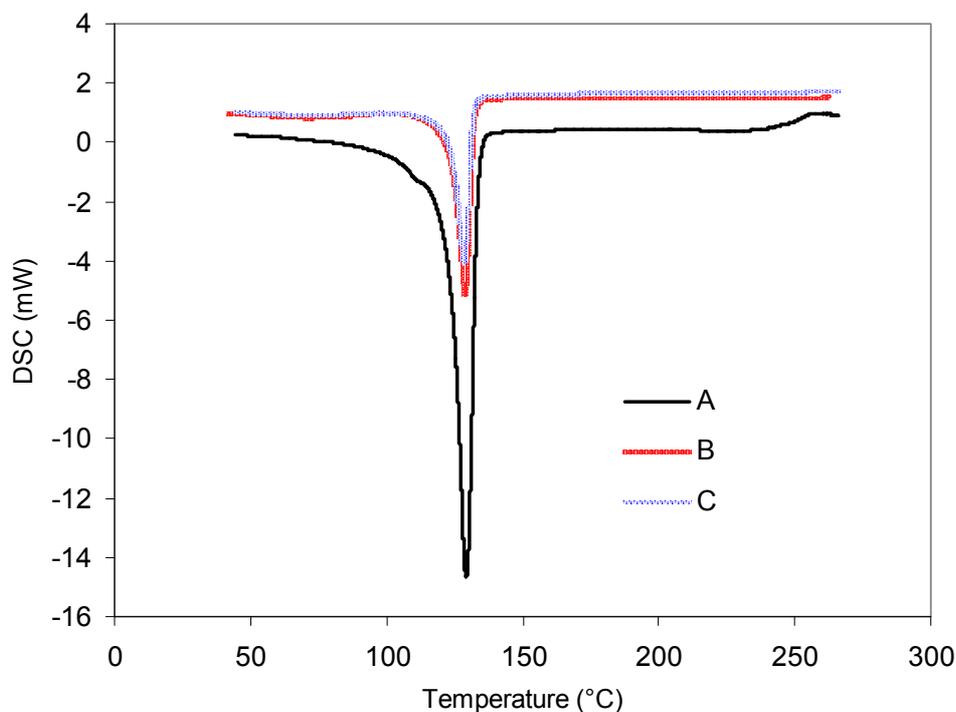


Figure 3. DSC thermographs of the recycled HDPE (A), eucalyptus residue filled recycled HDPE composites (B) and eucalyptus wood residue filled recycled HDPE modified with MAPE coupling agent (C).

Table 3. Mechanical properties of EWR filled recycled HDPE (B) and EWR filled recycled HDPE composites modified with MAPE coupling agent (C).

Specimen ID	Tensile Strength (MPa) ¹	Tensile Modulus (MPa)	Elongation at Break (%)	Flexural Strength (MPa)	Flexural Modulus (MPa)	Impact Strength (J/m)
B (no coupling agent)	6.44 ¹ (0.34) ²	253.6 (30.2)	3.23 (0.36)	14.72 (0.70)	1246.5 (68.0)	30.04 (0.68)
C (4% coupling agent)	9.58 (0.41)	329.3 (32.3)	3.37 (0.21)	17.16 (0.90)	1304.3 (68.0)	28.17 (1.20)

¹Each value is the average of 7 samples tested.

²The value in parenthesis is the standard deviation.

Tensile properties include tensile strength, tensile modulus and elongation at break. Table 4 summarizes the analysis of variance for tensile properties of EWR filled recycled HDPE composites. The ANOVA showed that addition of coupling agent significantly improved the tensile strength ($P < 0.0001$). Similar results were also reported by others [37-40]. It is believed that better wetting of the PE based maleic anhydride to the HDPE matrix polymer is responsible for improved results. Addition of MAPE coupling agent significantly advanced the tensile modulus of the composites ($P = 0.0007$) due to the improved adhesion between plastic and EWRs. Similar results for other wood flours filled polymer composites were also reported [19, 41]. In the case of elongation at break, coupling agent had no significant effect ($P = 0.4087$) on composites.

Table 4. Analysis of variance for tensile properties of eucalyptus residue filled recycled HDPE composites.

<i>Dependent Variable</i>	<i>Source of variation</i>	<i>SS</i>	<i>DF</i>	<i>MS</i>	<i>F</i>	<i>P</i>
Tensile Strength	Effect of Coupling Agent (CA)	34.54	1	34.54	241.27	<0,0001
	Pure Error	1.72	12	0.14		
	Total	36.26	13			
Tensile Modulus	Effect of CA	20068	1	20068	20.48	0.0007
	Pure Error	11760	12	979.97		
	Total	31828	13			
Elongation at Break	Effect of CA	0.64	1	0.64	0.73	0.4087
	Pure Error	1.06	12	0.88		
	Total	1.12	13			

Flexural properties include flexural strength and flexural modulus (Table 3). Table 5 summarizes the analysis of variance for flexural properties of EWR filled recycled HDPE composites. Addition of coupling agent significantly improved the flexural strength ($P = 0.0001$). Similar results were also reported in the flexural strength of other wood flour filled thermoplastic composites [37-39]. For polyolefin-based plastic lumber decking boards, ASTM D 6662 (2001) standard requires the minimum flexural strength of 6.9 MPa (1,000 psi). All composites produced in this study provided flexural strength values (14-17 MPa) that are well over the requirement by the standard. In the case of flexural modulus, addition of coupling agent improved the flexural strength but this increase was not statistically significant ($P = 0.1378$). ASTM D 6662 (2001) standard requires the minimum flexural modulus of 340 MPa (50,000 psi) for polyolefin-based plastic lumber decking boards. All composites produced in this study provided flexural modulus values (1250-1300 MPa) well over required standards.

Table 5. Analysis of variance for flexural properties of eucalyptus residue filled recycled HDPE composites.

<i>Dependent Variable</i>	<i>Source of variation</i>	<i>SS</i>	<i>DF</i>	<i>MS</i>	<i>F</i>	<i>P</i>
Flexural Strength	Effect of CA	20.86	1	20.86	32.02	0.0001
	Pure Error	7.82	12	0.65		
	Total	28.68	13			
Flexural Modulus	Effect of CA	11678	1	11678	2.53	0.1378
	Pure Error	55440	12	4620		
	Total	67118	13			

Table 6 summarizes the analysis of variance for impact strength of EWR filled recycled HDPE composites. Addition of coupling agent reduced the Izod impact strength significantly ($P=0.0037$). In composite materials, improved adhesion usually changes the mode of failure from “fiber pull out” to fiber breakage, which usually requires less energy. Similar findings were reported in other studies for different wood flours or fibers [2].

Table 6. Analysis of variance for impact properties of eucalyptus residue filled recycled HDPE composites.

<i>Dependent Variable</i>	<i>Source of variation</i>	<i>SS</i>	<i>DF</i>	<i>MS</i>	<i>F</i>	<i>P</i>
Impact Strength	Effect of CA	12.22	1	12.22	12.93	0.0037
	Pure Error	11.34	12	0.95		
	Total	23.56	13			

2.3. Morphology

Morphology of the eucalyptus wood residue (EWR) filled recycled HDPE composites were studied using SEM micrographs. Figure 4 shows the fractured surface of unfilled recycled HDPE, EWR filled recycled HDPE composites and MAPE modified EWR filled recycled HDPE composites. When Figure 4a and b was compared, it could be seen that matrix with eucalyptus wood residue was stiffer than other. In Figure 4 b, the arrows show the individual eucalyptus wood residues in the matrix. This result was due to the poor adhesion between the not compatible EWRs (hydrophilic) and recycled HDPE matrix (hydrophobic). Figure 4c presents the EWR filled recycled HDPE composites with 4-percent MAPE coupling agents. Some wood residues were embedded into the recycled HDPE matrix indicating improved adhesion. However, there were still individual wood residues on the interface of eucalyptus wood residue and recycled HDPE matrix.

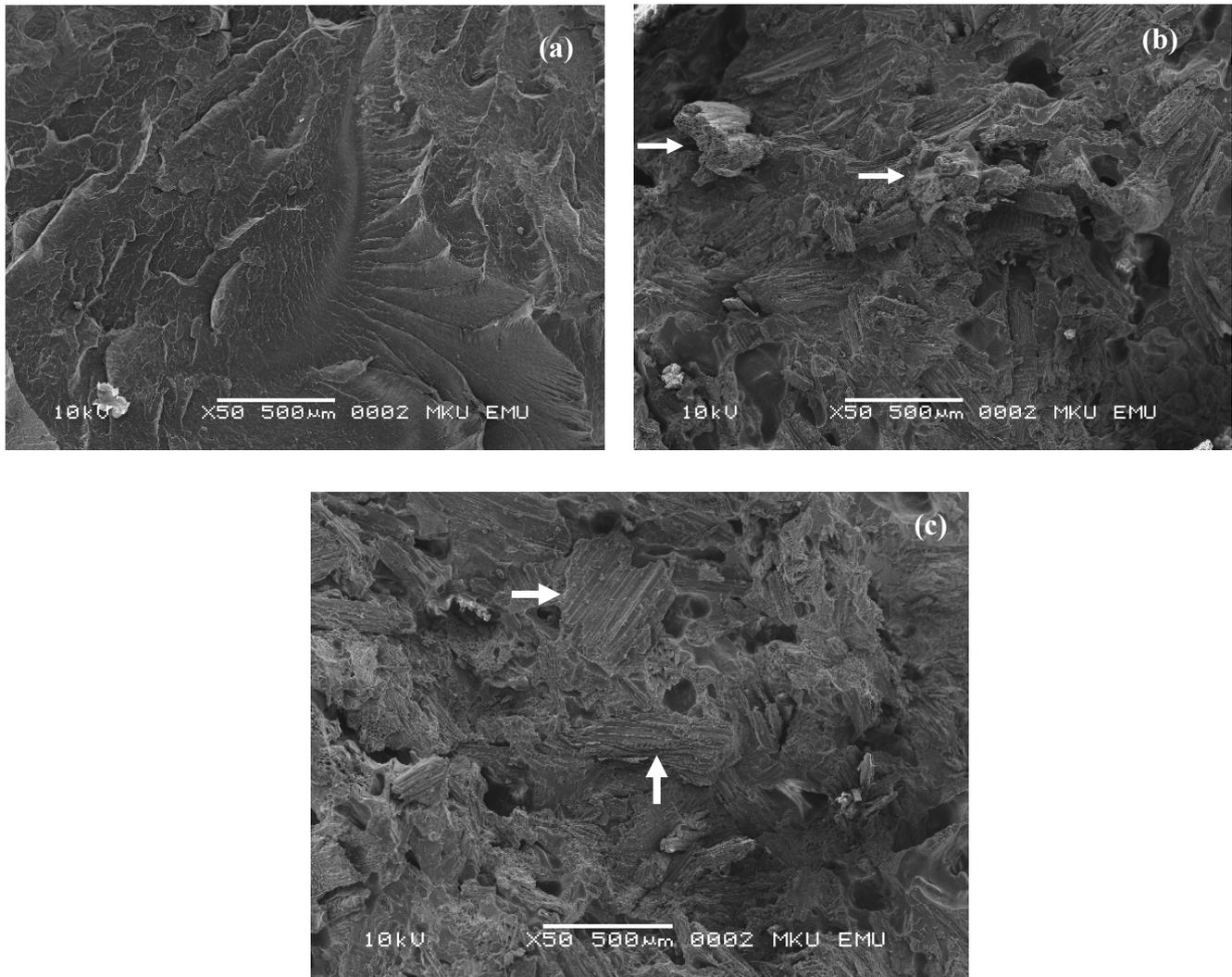


Figure 4. SEM micrographs of the a) unfilled recycled HDPE, b) eucalyptus wood residue filled recycled HDPE composites and c) MAPE modified eucalyptus wood residue filled recycled HDPE composites

3. Experimental Section

3.1. Materials

Recycled high density polyethylene (HDPE) and eucalyptus wood residues were used as thermoplastic matrix and organic filler, respectively. Recycled HDPE pellets were produced from waste water pipes while eucalyptus residues were collected from lumber mills in the city of Tarsus, Turkey. They were granulated into 40-mesh size flours using Wiley mill. Maleic anhydride grafted polyethylene (Licocene PEMA 4351 by Clariant) was utilized as coupling agent. Descriptions of coupling agents are provided in Table 7.

3.2. Compounding of the composites

Compounding of the composites was implemented using a single screw extruder. Three different panels in Table 8 were manufactured. Depending on the groups, granulated HDPE, eucalyptus wood

residue (EWR) and MAPE coupling agent were mixed in a high intensity mixer for 5 minutes to produce homogeneous blend. Then this homogenous mixture was compounded in a laboratory scale single-screw extruder at 40 rpm screw speed. Extruder temperatures were set as 170 175 180 185 185 °C. The extrudates were collected, cooled and granulated into pellets. Finally, pellets were compression molded in the hot press for 5 minutes at 175 °C and cooled for 20 minutes. Panels with the size of 5x150x200 mm were produced.

Table 7. Descriptions of the coupling agents used in this study.

Descriptions	Licocene PE MA 4351 (MAPE)
Appearance	White fine grain
Softening point	123°C
Acid Value	43 mg KOH/g
Density at 23°C	0.99 g/cm ³
Viscosity at 140 °C	300 mPa.s

Table 8. Description of the manufactured samples.

ID	Recycled HDPE (%)	Eucalyptus wood residue (%)	MAPE (%)
A	100	-	-
B	50	50	-
C	46	50	4

3.3. Thermogravimetry and Differential Scanning Calorimetry

Thermogravimetric analysis (TGA) of the samples was done in a Shimadzu TGA-50 thermal analyzer using a scanning rate of 10 °C/min heating rate under nitrogen with 20 mL/min flow rate, from room temperature to 800°C. Differential scanning calorimeter (DSC) analysis was performed in Shimadzu DSC-60 using 10 °C/min heating rate under nitrogen with 30 mL/min flow rate, from room temperature to 500 °C.

3.4. Mechanical Property Testing

To evaluate the effect of MAPE coupling agent on the mechanical properties of eucalyptus wood residue filled recycled HDPE composites, testing of the flexural, tensile and impact properties were determined in a climate-controlled testing laboratory. The flexural tests were conducted in accordance with ASTM D 790. Ten samples were tested on Zwick 10KN for each group with a crosshead motion rate of 2.0 mm/min. ASTM D 683 standards were used for tensile tests with a 5.0 mm/min test speed. The impact tests were performed according to ASTM D 256. Ten samples for each group were cut from the manufactured composites. The notches were added using a Polytest notching cutter by RayRan™ and notched samples were tested on a HIT5.5P impact testing machine, manufactured by Zwick™. At least seven samples were tested for each property.

3.5. Scanning Electron Microscope

The fractured surface of the samples was also studied by using JEOL scanning electron microscope (Model JSM 6400). The samples were first dipped into liquid nitrogen and snapped to half to prepare the fractured surfaces. Then samples were mounted on the sample stub and were sputtered with gold.

3.6. Statistical Analysis

Design-Expert® Version 7.0.3 statistical software program was used for statistical analysis. In this study, the general factorial design for one factor was chosen to determine effect of addition of 4-percent MAPE coupling agent on the mechanical properties of eucalyptus wood residue filled recycled HDPE composites.

Acknowledgements

This research was supported by The Scientific & Technological Research Council of Turkey (Project # TOVAG 1060548). Authors would also like to thank Mr. Kadir Karakus and Mr. Murat Ertas for the help during composite manufacturing and instrumental analysis.

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