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Some Properties of Eucalyptus Wood Flour Filled Recycled High Density Polyethylene Polymer-Composites

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Abstract: The mechanical properties of eucalyptus wood flours (EWF) filled recycled high density polyethylene (HDPE) composites were investigated. First thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC) analysis were conducted for recycled HDPE and EWF. Later the effects of EWF and maleic anhydride grafted polyethylene (MAPE) concentrations were studied. TGA analysis revealed that EWF and recycled HDPE started degrading around 230 °C and 280 °C, respectively. Melting temperature of the recycled HDPE was around 129 °C. Mechanical property evaluation showed that the increased concentration of EWF in polymer-composites reduced the tensile, flexural, and impact strength while increased the tensile and flexural modulus. The addition of small amount of MAPE coupling agent improved the tensile and flexural strength but this increase was leveled off at higher concentrations. Impact strength of the composites was reduced by the coupling agent. This study showed that polymer-composites can be manufactured utilizing recycled HDPE and eucalyptus wood flours in the process temperature range of 130-230 °C and could provide additional income for the lumber mills and promote the recycling of HDPE.

Key Words: Recycled HDPE, polymer-composites, mechanical properties, coupling agent, TGA, DSC

Dolgu Maddesi Olarak Okaliptüs Unları Kullanılan Geri Dönüşüm Yüksek Yoğunluklu Polietilen Polimer-Kompozitlerin Bazı Özellikleri

Özet: Bu çalışmada, okaliptüs odun unları (OOU) ve geri dönüşüm halindeki yüksek yoğunluklu polietilen (YYPE) kullanılarak üretilen polimer-kompozitlerin mekanik özellikleri araştırılmıştır. Öncelikli olarak, OOU ve geri dönüşüm YYPE üzerinde termogravimetrik analizör (TGA) ve diferansiyel taramalı kalorimetre (DSC) analizleri gerçekleştirilmiştir. Ardından, OOU ve uyumsuzluk giderici MAPE kimyasalının konsantrasyonunun polimer kompozit üzerine etkisi incelenmiştir. TGA analizi sonuçları, termal bozunmanın OOU için 230 °C ve geri dönüşüm YYPE için 280 °C civarında başladığı gözlenilmiştir. DSC sonuçlarına göre; YYPE'nin erime sıcaklığının yaklaşık 129 °C olduğu tespit edilmiştir. Üretilen kompozitlerin mekanik özellikleri dikkate alındığında, OOU miktarındaki artışın kompozitin çekme, eğilme ve darbe dirençlerinde azalmaya, çekme ve elastikiyet modülü değerlerinde ise artmaya neden olduğu belirlenmiştir. Az miktarda uyumsuzluk giderici kimyasalın matrise ilave edilmesi sonucunda ise çekme ve eğilme direncinde artış olduğu, ancak bu artışın doğrusal olarak gerçekleşmeyip bir sure sonra dengelendiği gözlenmiştir. Diğer taraftan uyumsuzluk gidericilerin darbe direnci değerlerinde azalmaya sebep olduğu tespit edilmiştir. Bu çalışma, geri dönüşüm haldeki YYPE ve OOU kullanılarak 130 °C ile 230 °C üretim sıcaklıkları arasında polimer-kompozit üretiminin gerçekleştirilebileceğini ve bu tip bir üretimin kereste fabrikalarına ek gelir getirebileceğini ve geri dönüşümü teşvik edebileceğini göstermiştir.

Anahtar Sözcükler: YYPE, polimer-kompozit, mekanik özellikler, uyumsuzluk giderici, TGA, DSC

Introduction

There is a growing trend in the use of organic fillers in the manufacture of polymer-composites due to their low density, low cost, nonabrasive nature (Clemons, 2002; Mengeloglu and Matuana, 2003), possibility of high filling levels, low energy consumption, high specific properties, biodegradability, and availability throughout the world (Abu-Sharkh et al., 2004; Matuana et al., 1998).

At the beginning companies were skeptical about the use of organic fillers (Clemons, 2002). The sensitivity of the organic fillers to heat and moisture, and the lack of adhesion between hydrophilic organic filler and hydrophobic polymer raised questions about their usage.

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Questions about heat and moisture were answered by choosing polymer having the melting temperature lower than degradation temperature of organic fillers and drying of them before or during processing. Question about adhesion was overcome by improving the similarities between polar organic fillers and non-polar polymer matrices (Mengeloglu et al., 2007). For this purpose several coupling agents have been employed (Woodhams et al., 1994; Jana and Prieto, 2002; Geng et.al., 2004) and maleated coupling agents were found to be the most suitable coupling agent for polyolefin based polymer-composites (Lu et al., 2005).

Polymer-composites might consist of organic fillers, such as agricultural plants residues and wood of various species and polymers such as polyethylene, polypropylene, and polyvinyl chloride. This type of polymer-composite has several application areas including automobile interior parts (Clemons, 2002), siding, fencing, window frames, and decking (Clemons, 2002; La Mantia, 2005; Youngquist, 2005; Mengeloglu et al., 2007). Its low density, high specific properties, non-abrasive nature, availability, etc. can be listed among the advantages of the polymer-composites (Clemons, 2002; Mengeloglu and Matuana, 2003; Panthapulakkal et al., 2006). Several studies were conducted to manufacture polymer composite using agricultural plant residues in flour or fiber form including hemp (Mwaikambo and Ansell, 2002; Prasad and Sain, 2003), bamboo (Chen et al., 1998), flax (Vande Velde and Keikens, 2001), jute (Gassan and Bledzki, 1997), sisal (Joseph et al., 1996), wheat (Hornsby et al., 1997), and kapok (Mwaikambo and Ansell, 2002).

Various wood species were also utilized in polymer-composites. Most pine species (Kim et al., 2006), aspen (Rachtanapun et al., 2003), spruce (Bengtsson and Oksman, 2006), maple (Li and Matuana, 2003), etc. were successfully used as filler in polymer-composites. Previously the use of eucalyptus wood flours in the manufacture of polymer-composites was not investigated thoroughly.

It is well known that during the manufacturing of polymer-composites, recycled plastics as polymer matrix and lumber mill residues as organic filler can be utilized. Turkey produces nearly 3 million tons of plastic waste every year and only small amount of them is recycled (Mengeloglu, 2006). Turkey's forest product industry also generates roughly 500 m^3 of eucalyptus wood residues from milling approximately 20,000 m^3 eucalyptus woods annually. Most of the eucalyptus wood was consumed locally by small companies. Drying eucalyptus wood requires carefully selected drying schedules and well equipped dry kilns; these companies do not have facilities to dry these lumbers properly. They are usually air dried and most often resulted with many cracks on them. Available branches from logging operations, wood residues from milling operations, and lumbers with many cracks create an opportunity to look for alternative use areas for eucalyptus wood as a raw material.

It is obvious that there is tremendous amount of waste material present in Turkey. Considering the available recycled polymer and waste eucalyptus flours, manufacturing of polymer-composite could be a viable option for Turkey. In this study, the effects of eucalyptus wood flour and coupling agent concentrations on the mechanical properties of polymer-composites were investigated.

Materials and Methods

Materials

Recycled high density polyethylene (HDPE) and eucalyptus wood flours (EWF) were used as polymer matrix and filler, respectively. First, water pipes were collected, cleaned from dirt, and sawed into small pieces. Then, these small pieces were grinded into small particles using a Willey mill. The eucalyptus residues were collected from the lumber mills in Tarsus, Turkey. During log cutting with band saw, residues with small amount of bark were generated. Collected residues were granulated into flour form using a Willey mill, screened to 45 mesh-size (354 μ) particles and dried to less than 1% moisture. Maleic anhydrite grafted polyethylene (MAPE) was also used as coupling agent.

Methods

Compounding and Composite Manufacturing

The experimental design of the study is presented in Table 1. Two sets of experiment were carried out. One designed to investigate the effect of eucalyptus wood flour (EWF) loading while the other one designed to show the effect of coupling agent loading. During the manufacturing process, depending on the formulation,

Table 1. Experimental design of the study.

	ID Concentration (%)	HDPE MAPE Concentration (%)	EWF Concentration (%)
A	83.3	16.7	0
B	66.7	33.3	0
C	50.0	50.0	0
D	50.0	50.0	2
E	50.0	50.0	4
F	50.0	50.0	6

Note:
EWF: Eucalyptus wood flour.
MAPE: Maleic anhydride grafted polyethylene

granulated recycled HDPE, 45-mesh EWF, and MAPE coupling agent were mixed in a high intensity mixer to produce a homogeneous blend. Then, this blend was compounded in a laboratory scale single screw extruder at 40 rpm screw speed and in the temperature range of 150-180 °C. Extruded samples were collected, cooled, and granulated into pellets. Finally, pellets were compressed into 5 × 150 × 160 mm size composites for 5 min at 175 °C.

Thermogravimetric (TGA) and Differential Scanning Calorimetry (DSC) Analysis

A Shimadzu TGA-50 thermal analyzer was used for thermogravimetric analysis (TGA) of the samples. Heating rate was 10 °C/min under nitrogen with 20 ml min^2 flow rate. Test was performed in the temperature range of 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^2 flow rate, from room temperature to 500 °C.

Scanning Electron Microscope (SEM) Study

JEOL scanning electron microscope (SEM, Model JSM 5500LV) was used to study the fractured surface of the samples. 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.

Property Testing

Testing of the produced composites was conducted in a climate-controlled testing laboratory. Flexural, tensile and impact properties of all boards were determined. The flexural tests were conducted in accordance with ASTM D 790. Test samples were cut in the dimensions of 5 × 13 × 15 mm. The span length of each specimen was 100 mm, with the rest left as overhang. Samples were tested on Zwick 10KN. The rate of crosshead motion was 2.0 mm/min, which was calculated according to the ASTM D 790 standard.

The tensile tests were conducted according to the ASTM D 683. Samples were tested on Zwick 10KN. Tests were performed at a rate of 5.0 mm/min. Dog-bone shape samples were used (Type III). The tensile modulus of the samples was taken as the slope of the curve at stress levels between 0.05% and 0.2%, while the tensile strength was the maximum stress experienced by each specimen.

The impact tests were performed according to ASTM D 256. Impact 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™.

Statistical Analysis

Design-Expert® Version 7.0.3 statistical software program was used for statistical analysis. In this study, effect of eucalyptus wood flour loading was analyzed using 1-factor ANOVA at 3 levels while the effect of coupling agent concentration was analyzed using 1-factor ANOVA at 4 levels.

Results

Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC)

TGA analysis was performed on eucalyptus wood flour (EWR) and recycled HDPE. Figure 1 shows the TGA thermographs of the samples. Initial degradation of EWR was started around 220 °C while recycled HDPE was started around 300 °C. Figure 2 shows the DSC thermographs of the recycled HDPE. Melting temperature of the recycled HDPE was around 129 °C.

Effect of eucalyptus wood flour loading on the mechanical properties of polymer-composites

Recycled high density polyethylene (HDPE) was filled with 16.7%, 33.4%, and 50.0% eucalyptus wood flours (EWFs) to produce polymer-composites A, B, and C, respectively. Mechanical properties of the produced polymer-composites are presented in Table 2. Tensile, flexural, and impact properties were determined. Statistical analysis showed that tensile strength of the polymer-composites was significantly reduced with increase of EWF concentrations ($P < 0.0001$). On the other hand, tensile modulus of the polymer-composites was significantly improved with higher concentration of EWFs ($P < 0.0003$). In the case of elongation at break values, they were significantly reduced with the increased concentration of EWFs ($P < 0.0001$).

Similar to the tensile strength, flexural strength was significantly reduced ($P < 0.0002$) while flexural modulus was improved significantly ($P < 0.0001$) with the increased concentration of EWFs. Increase in the concentration of EWF significantly reduced the notched impact strength of the polymer-composites ($P < 0.0001$).

Effect of MAPE concentration on the mechanical properties of polymer-composites

Fifty percent EWF filled recycled high density polyethylene (HDPE) was produced with 0%, 2%, 4%, and 6% MAPE coupling agent and labeled as C, D, E, and F, respectively. Mechanical properties of the polymer-composites are summarized in Table 3.

Statistical analysis showed that MAPE coupling agent significantly affected the tensile strength of the polymer-composites ($P < 0.0001$). Addition of 2% MAPE significantly improved the tensile strength but further increase in MAPE concentration did not affect the tensile

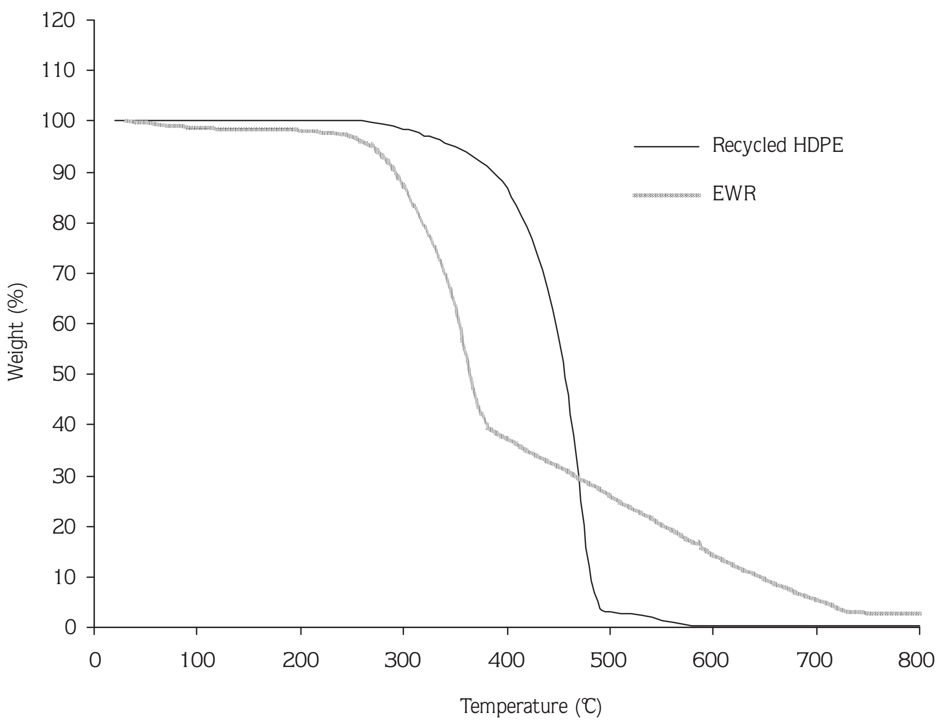

Figure 1. TGA thermographs of eucalyptus wood flour (EWF) and recycled high density polyethylene (HDPE).

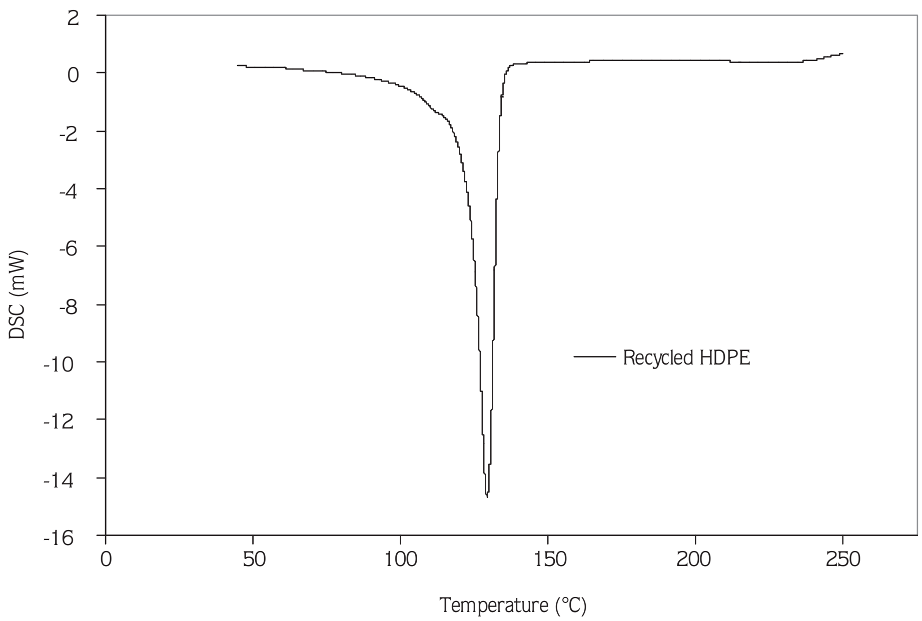

Figure 2. DSC thermograph of recycled high density polyethylene (HDPE).

Table 2. Effect of eucalyptus wood flour (EWF) loading on the mechanical properties of polymer-composites.

ID	Tensile Strength ($N\ mm^{-2}$)	Tensile Modulus ($N\ mm^{-2}$)	Elongation at Break (%)	Flexural Strength ($N\ mm^{-2}$)	Flexural Modulus ($N\ mm^{-2}$)	Impact Strength (J/m)
A	10.75 a (1.92)	204.5 a (71.4)	9.52 a (1.24)	20.23 a (2.47)	813.1 a (114.7)	71.82 a (16.89)
B	8.22 b (1.72)	275.6 b (37.6)	4.40 b (0.54)	17.93 b (3.05)	992.2 b (185.5)	42.65 b (4.02)
C	6.51 c (1.72)	248.3 b (31.7)	3.39 c (0.62)	14.97 c (1.24)	1263.8 c (100.2)	30.32 c (2.21)

Note:
- The numerical value in the parenthesis is standard deviation.
- Different letters indicate significantly different groups ($P < 0.05$)
- N = 10 samples

strength significantly. MAPE addition into the polymer composites also improved the tensile modulus significantly ($P < 0.0039$). Elongation at break values of the polymer-composite stayed unchanged with the addition of coupling agents ($P < 0.2432$).

In the case of flexural properties, presence of MAPE coupling agent in the polymer-composites significantly increased the flexural strength ($P < 0.0044$). However, further increase in MAPE coupling agent concentration did not affect the flexural strength of the polymer-composites. Flexural modulus was increased significantly with MAPE coupling agent ($P < 0.0319$). Notched impact strength values were significantly affected by the MAPE coupling agent concentration ($P < 0.0001$). Notched impact strength was reduced with the MAPE coupling agent.

Table 3. Effect of MAPE coupling agent concentrations on the mechanical properties of polymer-composites.

ID	Tensile Strength (N mm^{-2})	Tensile Modulus (N mm^{-2})	Elongation at Break (%)	Flexural Strength (N mm^{-2})	Flexural Modulus (N mm^{-2})	Impact Strength (J/m)
C	6.51 a (1.72)	248.3 a (31.7)	3.39 a (0.62)	14.97 a (1.24)	1263.8 a (100.2)	30.32 a (2.21)
D	8.53 b (1.25)	286.8 b (62.6)	3.52 a (0.32)	17.72 b (1.89)	1299.3 a (142.3)	26.41 b (1.54)
E	9.60 b (1.26)	321.2 c (41.7)	3.37 a (0.35)	19.11 b (4.42)	1420.7 ab (283.8)	29.06 c (2.43)
F	9.56 b (1.72)	297.8 bc (20.5)	3.58 a (0.41)	18.87 b (1.77)	1463.2 b (139.5)	25.71 c (1.81)

Note:
- The numerical value in the parenthesis is standard deviation.
- Different letters indicate significantly different groups ($P < 0.05$).
- N = 10 samples.

Discussion

Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC)

During the manufacturing of polymer-composites, melting temperature of the polymer and the degradation temperature of the filler have great importance. Based on the TGA and DSC analysis during the manufacturing of the polymer-composites, extruder temperatures should be over 129 °C to facilitate the melting of the polymer matrix and should be less than 220 °C to prevent the eucalyptus wood flour (EWF) from degrading. In other studies, initial degradation of natural fibers such as hemp and flax are reported to be 230 °C (Kozlowski and Wladyka-Pryzbylak, 2008) while jute and wheat straw flour were 190 °C (Choundhury and Adhikari, 2007) and 220 °C (Mengeloglu and Karakus, 2008), respectively. The initial degradation temperature of pine was reported to be around 256 °C (Lei et. al, 2007). It is believed that low temperature degradation is associated with the degradation of hemicelluloses. It should also be noted that residence time of the material in the extruder is also important (Chan and Balke, 1997). Higher processing temperature can be set if the component passes through the extruder in a short period of time.

Effect of eucalyptus wood flour loading on the mechanical properties of polymer-composites

Tensile strength of the polymer composites were significantly reduced with the increased amount of EWFs. Nunez et al. (2002) observed a similar trend with eucalyptus wood flour filled polypropylene composites. La Mantia et al. (2005) also reported similar findings when thermomechanical pulp fiber from softwood was used as filler. It is believed that dissimilarities between polar wood flour and non-polar polymer matrix caused poor adhesion and resulted in lower tensile strength. Poor dispersion of the fillers in the polymer matrix could be another reason for this. Since there was not good bonding between the wood flour and polymer matrix, test samples were broken at lower loads. Figure 3 shows the SEM image of the polymer-composites C. Pulled out wood flours indicating poor adhesion can be seen on this micrograph.

Tensile modulus of the polymer-composites was significantly enhanced when the amount of EWF in the matrix was increased. This could simply be explained by the rule of mixture (Matuana and Balatinecz, 1998). Wood flours have higher modulus compared to polymer matrix as a result their mixture produces modulus values were higher compared to polymer itself. The improvement in the tensile modulus with the increase of wood flour loading has

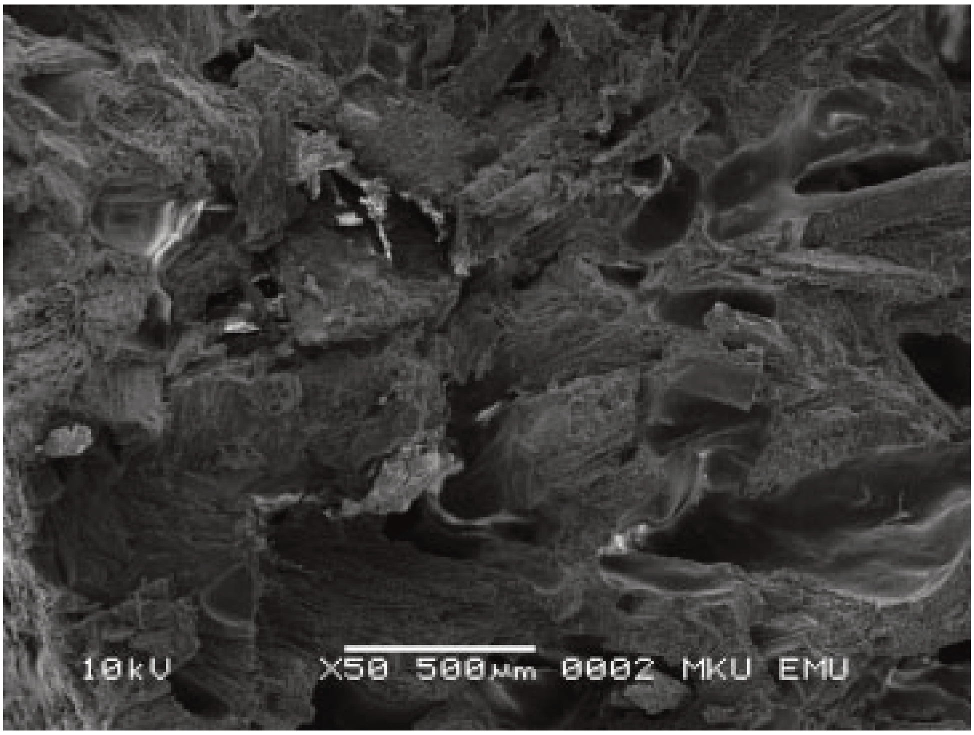

Figure 3. SEM images of polymer-composite C (50% EWF filled recycled HDPE).

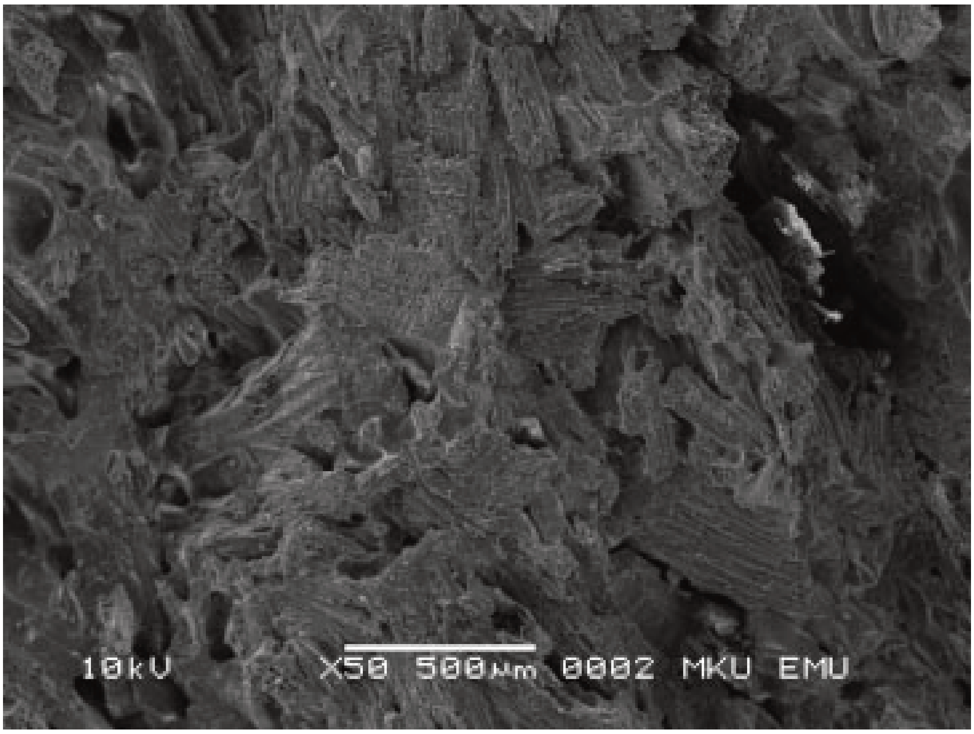

Figure 4. SEM images of polymer-composite E (50% EWF filled recycled HDPE modified with 4% MAPE).

also been reported by others (Stark and Berger, 1997; Nunez et. al., 2002; La Mantia et. al., 2005). This is one of the advantages of the use of wood flour as filler. In some applications, certain modulus values were desired and could not be provided by polymer by itself.

Elongation at break values was significantly reduced with the increased concentration of EWFs. Polymer-composites became stiffer when the amount of wood flour was increased. As a result, the elongation at break values reduced. Usually in composites, lower elongation at break values was observed with increased modulus (Chan and Balke, 1997; Sain and Panthapulakkal, 2006; Yang et al., 2007).

Flexural strength of polymer-composites reduced when the amount of EWF was increased. Similar results were reported by Nunez et al. (2002) for eucalyptus wood flour filled PP composites. This result could be caused by lack of good adhesion between hydrophobic polymer matrix and hydrophilic wood flour because of the hydrophilicity of wood flour and hydrophobicity of plastics (Li and Matuana, 2003). Similar to the tensile modulus, flexural modulus significantly increased with EWF concentration. Similar behaviors for the composite produced with various wood flours were observed in other studies (Li and Matuana, 2003; Wang et al., 2003). ASTM D 6662 (2001) standard requires the minimum flexural strength of 6.9 N mm^{-2} (1000 psi) and flexural modulus of 340 N mm^{-2} (50,000 psi) for polyolefin-based plastic lumber decking boards. Polymer-composites produced in this study provided flexural strength values (15-20 N mm^{-2}) and flexural modulus values (813-1,263 N mm^{-2}) that are well over the requirement by the standard. Increase in the concentration of EWF significantly reduced the notched impact strength of the polymer-composites. Wood flour increased the brittleness of the polymer-matrix (Matuana and Balatinecz, 1998; Mengeloglu et.al., 2000; Li and Matuana, 2003).

Effect of MAPE concentration on the mechanical properties of polymer-composites

Poor adhesion between polar wood flour and non-polar plastic matrix can be overcome with the use of coupling agents (Clemons, 2002; Li and Matuana, 2003, Lu et al., 2005). In this study, 0%, 2%, 4%, and 6% MAPE coupling agent was used in recycled HDPE composites filled with 50% EWFs. The addition of MAPE coupling agent improved the tensile strength of the composites. However this increase was not linear with the coupling agent concentration, which increased and leveled of between 2% and 4%. It is believed that MAPE coupling agent improved the similarities between the wood flour and polymer matrix resulting in good bonging between them. Figure 4 shows the SEM image of the polymer-composites E. Small wood flours shown with white arrows were embedded in the polymer matrix showing a fairly good adhesion in the polymer-composites. However, there was some pulled-out flour in the polymer-composites meaning that the potential of strength improvement was not fully exploited.

Similar trend was seen in tensile modulus. MAPE addition into the polymer composites improved the tensile modulus but, at higher loading, it leveled off. Elongation at break values of the polymer-composite stayed unchanged with the addition of coupling agents.

Similar to the tensile strength, flexural strength improved with the addition of the MAPE coupling agent; however, at higher concentration, their effect leveled off. This increase might be due to the better wetting of the polymers with the use of coupling agent (Li and Matuana, 2003; Lai et al., 2003; Yang et al., 2007). Flexural modulus was increased significantly with the addition 4% MAPE coupling agent but further increase did not change the tensile modulus significantly. It should be noted that one of the major application areas of the polymer-composites is the decking applications. ASTM D 6662 (2001) standard requires the minimum flexural strength of 6.9 N mm^{-2} (1,000 psi) and flexural modulus of 340 N mm^{-2} (50,000 psi) for polyolefin-based plastic lumber decking boards. Polymer-composites with MAPE coupling agent provided much higher properties than required standard.

Notched impact strength values were significantly affected by the MAPE coupling agent concentration ($P < 0.0001$). Notched impact strength reduced with the MAPE coupling agent. This result was expected since the use of the coupling agent enhanced the interaction between filler and polymer resulting in increased brittleness of the composite (Selke and Childress, 1993). This might have changed the mode of failure from “fiber pull-out” to “fiber breakage”. Former one requires a larger amount of energy during the crack propagation compared to fiber breakage where the crack goes through the brittle wood-fiber (Matuana and Balatinecz, 1998; Mengeloglu et. al., 2000).

In conclusion, based on the TGA and DSC studies, processing temperature should be adjusted between 129 °C and 230 °C for polymer-composites utilizing EWF and recycled HDPE. Increase of the EWF concentration in polymer-composites reduced the tensile strength, elongation at break, flexural strength, and impact strength. Addition of 2% to 4% MAPE coupling agent improved the tensile strength, tensile modulus, flexural strength, and flexural modulus of the polymer composites filled with 50% EWF.

The successful application of EWF filled recycled HDPE polymer-composite could reduce the land filling and promote the recycling in Turkey. It might help the rural economy by opening new markets for waste materials.

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