

Performance Characterization of Composite Materials Based on Recycled High-Density Polyethylene and Ground Tire Rubber Reinforced with Short Glass Fibers for Structural Applications

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ABSTRACT: This study addresses the issue of using recycled materials to obtain low-cost structural products for practical applications. Through the use and re-extrusion of virgin high-density polyethylene (HDPE), the effects of the degradation level of HDPE as a matrix phase on its mechanical properties and the mechanical performance of composites produced with the degraded polyethylene have been examined. The degradation level of HDPE caused by re-extrusion has been evaluated by the measurement of the melt flow index and mechanical properties of virgin and degraded HDPEs. The results indicate that the modulus and strength of HDPE significantly increase with the addition of polypropylene filled with 30 wt % glass fiber [PP-GF(30)] without any other compatibilizer. However, the final properties of composites with specified glass-fiber contents are

dependent on the degradation level of the matrix phase. In addition, the role of ground tire rubber (GTR) in HDPE/PP-GF(30) systems has been examined by the preparation of composites with various GTR contents without any treatments. The results show that the presence of GTR in the final product results in lower stiffness because of its role as a soft filler, but the elongation of the product increases slightly. Furthermore, GTR does not produce any improvement in the impact properties, probably because of the low interfacial adhesion with the matrix phase; therefore, its content should be kept low in the final composition. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 104: 1–8, 2007

Key words: degradation; polyethylene (PE); recycling; reinforcement

INTRODUCTION

Annually, thousands of tons of postconsumer polymeric material wastes are generated all over the world, and they mainly end up in landfills. This is now becoming a concern because of the annual increase in polymeric waste generation and limited landfill areas, particularly in large cities. The reutilization of postconsumer polymeric materials and their return to industry could be a suitable way of solving environmental concerns. On the other hand, recycled polymeric materials offer low-cost sources of raw materials that can be used to produce practical products with low prices. Consequently, various aspects of postconsumer materials, including economical recycling procedures, the performance of recycled-material-based products, and the discovery of new applications for such products, have been the subjects of many studies in the past few decades.^{1,2}

One interesting area for recycled polymers is the production of structural components for engineering

applications.^{3,4} Because structural products based on virgin polymers are often expensive, their use is economically infeasible. One industrial application of structural materials based on recycled polymers is recycled-plastic lumber, which can be used in many structural applications for which chemically treated wood, especially wood treated by creosote, is traditionally used.⁵ A very important example is railroad crossties or sleepers.⁶ Currently, thousands of wooden crossties are replaced every year worldwide; therefore, producing recycled plastic crossties can help reduce the volume of waste in landfills. In addition, replacing wooden crossties with recycled composites helps to save many trees from being cut down and prevents hazardous chemicals from being introduced into the environment. As a result, serious research has been conducted to develop an appropriate candidate based on recycled polymers for wooden crossties since the early 1990s. The patents on this subject claim significant property improvements of recycled materials for such applications. In most cases, the appropriate materials are a proper combination of different disposal polymers, including postconsumer high-density polyethylene (HDPE), postindustrial glass-fiber-filled polypropylene (PP-GF), recycled polystyrene, ground

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TABLE I
Mechanical Properties of Recycled HDPE Filled
with PP-GF(35)

PP-GF(35) (wt %) in HDPE	Tensile modulus (GPa)	Tensile strength (MPa)
0	1	14
10	1.1	16
20	1.5	20
30	2	25
35 ^a	3	30
40	2.7	28
50	3.3	31

The data are approximate values taken from Figures 1 and 2 in ref. 10.

^a This compound was reported in ref. 10 to have a discontinuity in its mechanical properties.

tire rubber (GTR), and other additives.⁷⁻⁹ Mostly, HDPE forms the main part of the composition of the recycled plastic crosstie. This may be due to the fact that it constitutes a large portion of postconsumer plastics and has appropriate mechanical properties, suitable processability, and a reasonable price.

Despite industrial successes with recycled-plastic-composite crossties, few details have been reported in the literature concerning the formulation of the compounds and their performance. For instance, Nosker and Van Ness¹⁰ investigated the effect of the addition of postindustrial polypropylene containing 35 wt % glass fiber [PP-GF(35)] to recycled HDPE to obtain a suitable composition to be used for crossties. Table I presents the variation of the tensile modulus and strength of the composites with the weight percentage of PP-GF(35). The addition of PP-GF(35) enhances the mechanical performance of recycled HDPE significantly, although a discontinuity can be observed for the 35 wt % PP-GF(35) composition, which has not been assigned any technical explanation. To obtain a light-weight crosstie with suitable processability, a composition with a minimum content of glass fibers that satisfies the required mechanical properties is preferred.¹⁰ To achieve such a composition based on recycled materials, we need additional information about the constituents. For instance, because recycled HDPEs may be obtained from different sources that may be exposed to different storage and reprocessing conditions, they may show different performances depending on their degradation level. Therefore, the effect of the degradation level of recycled HDPE on the final product needs to be identified. To the best of our knowledge, such an analysis has not been addressed in the literature so far.

To make the use of recycled composite crossties in the railroad industry appealing, its final price should be kept as low as possible to compete with other

materials such as wood. Consequently, the primary concern is to design a composition with a minimum cost/performance ratio. To achieve this, we can use appropriate constituents with reasonable costs. An important constituent that has been claimed to be used in the composition of recycled plastic crosstie is GTR.⁹ However, there is no detailed information concerning its effectiveness on the mechanical performance of the final product.

Generally, GTR can be used as either treated¹¹⁻¹⁴ or untreated.¹⁵ Treated GTR can provide promoted interfacial adhesion, which may lead to better mechanical performance, but its large-scale utilization is normally hindered by the high cost. Of course, some simple methods have been addressed in the literature to improve the interfacial adhesion of polymers and GTR, but none of them have improved the performance of the obtained materials significantly. For instance, Radhesh Kumar et al.¹⁶ treated GTR with processing oil, but it did not show a significant improvement in the mechanical properties of a low-density-polyethylene-based thermoplastic elastomer in comparison with an untreated one. More recently, Scaffaro et al.¹⁵ observed that mixing GTR with recycled polyethylene at an elevated temperature (300°C) allowed them to enhance the compatibility between the phases. However, because of the degradation of polyethylene at such a high temperature, the final mechanical properties do not exhibit a significant improvement.

This investigation is an attempt to provide detailed information concerning the role of constituents in the performance of structural composites based on recycled HDPE, as claimed in patents,⁷⁻⁹ to be used as railroad crossties. Such details can enable us to find a suitable composition with a minimum cost/performance ratio. To do so, the effectiveness of fine particles of GTR in HDPE filled with glass fibers has been examined. This is supported by past studies that show that the incorporation of GTR into polyolefins has the smallest negative influence on the mechanical properties, particularly with polyethylenes.¹⁷ For economical considerations, untreated GTR has been used.

EXPERIMENTAL

Materials

The HDPE was a blow-molding grade (Poliran HB0035; melting temperature = 130°C) from BIP Co. (Bandar Imam, Iran). The HDPE supplied from the company was a virgin polymer that was degraded thermomechanically with a twin-screw extruder under controlled conditions. The polypropylene filled with 30 wt % glass fiber [PP-GF(30); received from local companies] was used as a reinforcing component of

the composition. This composite was added to the HDPE as received from the company. The GTR used in this study was donated by Isatiss Co. (a subsidiary of Yazd Tire Co., Iran) and was being provided via simple grinding with an extruder machine without any treatment; it had an average particle diameter of less than 0.4 mm. The GTR particles were mixed with polyethylene and PP-GF(30) in the extruder as received from the producer without further modification.

Melt flow index (MFI)

The MFI of pure HDPE (virgin and degraded) was determined with an MFI testing machine (GT-700-MI, Gotech Testing Machinery, Taiwan) according to ASTM D 1238-73. The tests were performed at 190°C under a load of 2.16 kg, and the results are reported in terms of grams per 10 min.

Mixing procedure and preparation of the test specimens

To investigate the effects of the constituents on the performance of the final products, mixtures of HDPE with PP-GF(30) and GTR were prepared, with various compositions, with a counter-rotating twin-screw extruder (SHJ-20, Nanjing Giant, China; length/diameter = 32, diameter = 21.7 mm) at a screw speed of 50 rpm. The melting-zone temperatures of various compounds were different, ranging from 190 to 210°C, depending on the glass-fiber content of the compounds. For compounds containing higher contents of glass fibers, the melting-zone temperature was kept higher to get a lower melt viscosity. The extrudates were quenched inside the water bath and then were cut into small granules with a pelletizer. The compounds were molded with an injection-molding machine into dumbbell-shaped and notched specimens.

Characterization of the mechanical properties

Tensile tests were carried out at room temperature with a tensile testing machine (H10KS, Hounsfield Co., England). The test was performed at a cross-head speed of 10 mm/min on dumbbell-shaped specimens according to ASTM D 638-76. The elastic modulus was determined as the stress increase between 0.05 and 0.25% strain and was measured with an incremental extensometer. In addition, the yield stress and elongation at yield were reported as engineering properties of the samples. The surface hardness of the samples was also measured on the surface of the tensile test specimens with a Shore D hardness tester according to DIN 53505-87. Notched impact specimens with dimensions of 126 mm × 12 mm × 6 mm were used to perform the impact

tests. The impact strength was measured at room temperature with an impact tester machine (Antam, Iran) according to ASTM D 256-73 in the Charpy mode. All experimental results were the averages of at least three experiments.

Differential scanning calorimetry (DSC)

The thermal characterization of the polyethylenes was investigated with a PerkinElmer differential scanning calorimeter in a nonisothermal mode within the temperature range of 0–200°C at a heating rate of 10°C/min. The heat of fusion of virgin and degraded polyethylenes was obtained from the melting peak of the DSC diagrams.

Morphology characterization

An XL30 scanning electron microscope (Philips, The Netherlands) was used to examine the morphology of the various composite systems. To do so effectively, the fractured surfaces were etched with a solvent to remove the contamination for scanning electron microscopy (SEM) observations.

RESULTS AND DISCUSSION

Thermomechanical degradation of HDPE

Recycled polymers show reduced mechanical performance because of exposure to different environments before recycling and reprocessing during recycling. Consequently, composite materials based on recycled polymers have low quality with respect to those based on virgin materials. To address the issue of the degradation of a recyclable polymer matrix and its effect on the final properties of a product, HDPE was degraded thermomechanically by re-extrusion of the virgin polymer under controlled conditions, and then the mechanical properties of the degraded polymer were examined. To do this, HDPE was extruded with a twin-screw extruder at a rotor speed of 100 rpm and 190°C. The virgin HDPE was designated PE(0), and PE(1) and PE(2) represent HDPEs extruded once and twice, respectively.

Table II presents the MFI values of the virgin and extruded HDPEs. The MFI of the virgin HDPE was 0.3, but it increased with the extrusion and re-extrusion of HDPE, suggesting a decrease in the melt viscosity of HDPE and correspondingly its molecular weight. This behavior was expected as a result of the polymer chain degradation due to a severe thermal and stress cycle exerted during the extrusion process leading to a molecular weight reduction. For PE(1), MFI showed a 30% increase in comparison with the virgin polymer, but PE(2) presented a significant change in the MFI value, that is, more than a 300%

TABLE II
MFI Values of Virgin and Extruded Polyethylenes

Polyethylene	MFI (g/10 min)
PE(0)	0.3
PE(1)	0.4
PE(2)	1

increase. Therefore, extruding and re-extruding HDPE resulted in two more HDPEs with different degrees of molecular weight reduction or degradation levels.

Table III lists the mechanical properties of the virgin and degraded polyethylenes. The tensile modulus and yield stress of the degraded polymers were reduced, whereas the surface hardness and elongation at yield showed slight variations. It is recognized that the amount of the reduction depends on the degradation level. The tensile modulus of the polyethylene decreased from 1.4 GPa for the virgin material, PE(0), to 1 GPa for PE(2), showing an almost 30% reduction. The same trend was observed for the yield stress, which decreased from 25.2 to 21.5 MPa. Similarly to the MFI values of various polyethylenes reported in Table II, the observed behavior for the tensile properties could be relevant to the molecular weight reduction of the polyethylene due to the degradation of the polymer. However, the amounts of change in the tensile properties and MFI of the polyethylenes did not exhibit the same level. For instance, the maximum change in the yield stress for degraded polyethylene was 15%, whereas it was 300% for MFI. The difference may be explained by different dependences of the melt viscosity and the mechanical properties on the molecular weight of the polymers.

Using the available theoretical models, we can provide a rough estimation concerning molecular weight reduction and obtain insight into the degradation level. The melt viscosity (η) of a polymer is related to the molecular weight according to the following expression:¹⁸

$$\eta = kM_w^a \quad (1)$$

where M_w is the weight-average molecular weight, k is a constant that depends on the structure of the

TABLE III
Mechanical Properties of Virgin and Extruded Polyethylenes

Polyethylene	Tensile modulus (GPa)	Yield stress (MPa)	Elongation at yield (%)	Hardness (Shore A)	Impact strength (kJ/m ²)
PE(0)	1.4	25.2	9.4	66	690
PE(1)	1.22	23.5	11	66	500
PE(2)	1.01	21.5	9.5	68	490

polymer and temperature, and a is a temperature-independent constant whose value is 3.5 for molecular weights above the critical value. Keeping constant the value of k for virgin and degraded polyethylenes and assuming a linear relationship between the MFI value and melt viscosity, we can estimate the ratio of the molecular weight of the virgin polymer to the molecular weight of the degraded polymer on the basis of eq. (1) as follows:

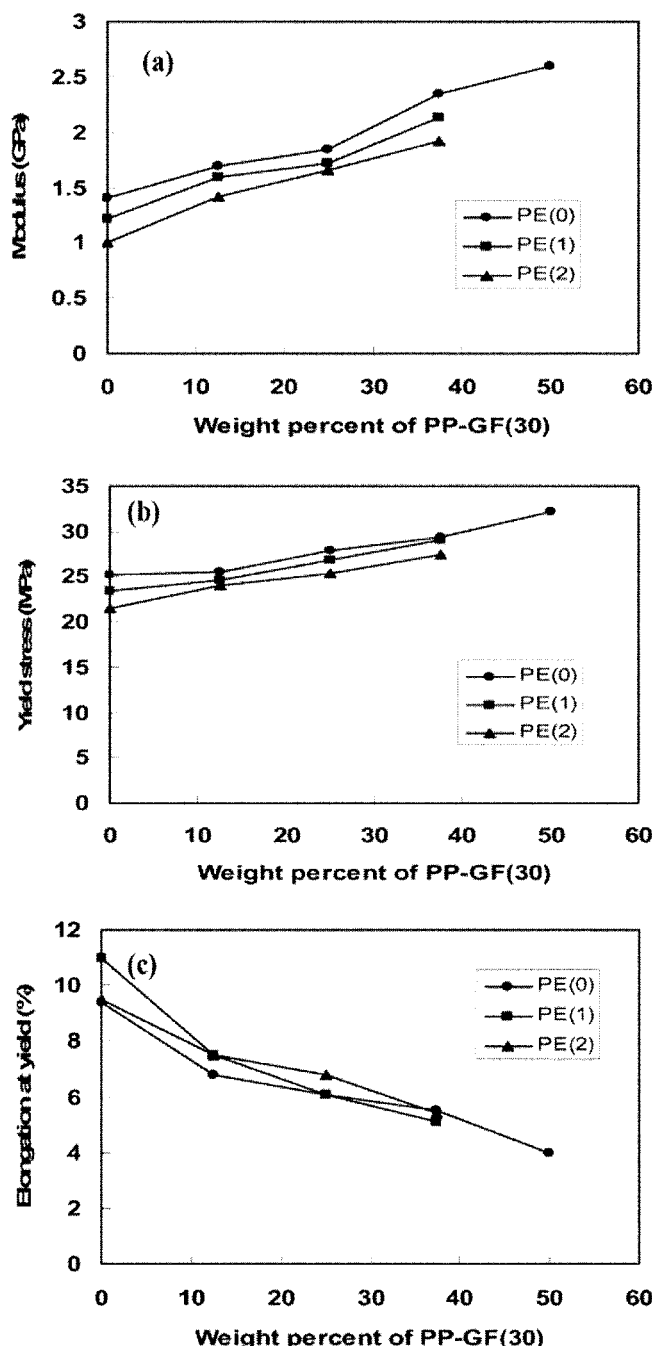


Figure 1 Variation of the tensile properties with the PP-GF(30) content for the HDPE/PP-GF(30) composites: (a) tensile modulus, (b) yield stress, and (c) elongation at yield.

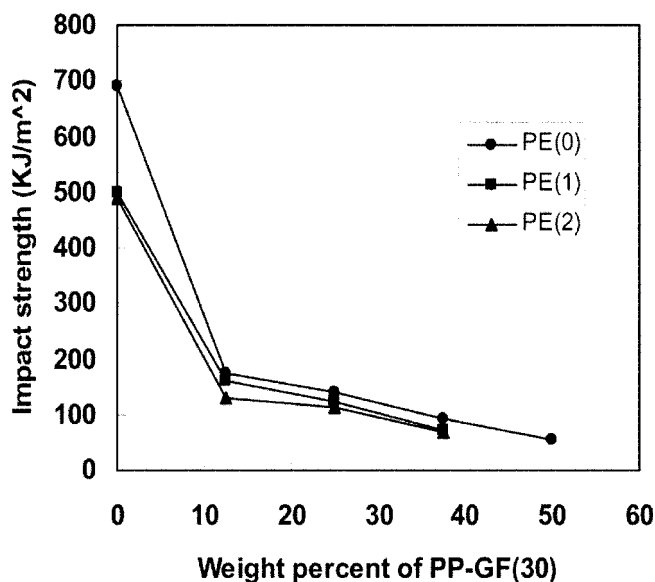


Figure 2 Variation of the impact strength with the PP-GF(30) content for the HDPE/PP-GF(30) composites.

$$\frac{(M_w)_d}{(M_w)_v} = \left(\frac{(\text{MFI})_v}{(\text{MFI})_d} \right)^{1/3.5} \quad (2)$$

where subscripts d and v represent the degraded and virgin polymers, respectively. On the other hand, the tensile strength (σ) with the molecular weight is as follows:¹⁸

$$\sigma = \sigma_0 \left(1 - \frac{M_e}{M_n} \right) \quad (3)$$

where σ_0 is the limiting tensile strength for a very high molecular weight, M_e is the critical molecular weight, and M_n is the number-average molecular weight of the polymer. According to eq. (2) and the MFI values of the virgin and degraded polyethylenes, PE(2) exhibited an almost 30% reduction in the molecular weight with respect to PE(0). Using this value for the molecular weight reduction, assuming that the reduction percentage in M_n was

the same as that for M_w , and performing some simple mathematical operations based on eq. (3), we found that the reduction percentage in the yield stress of polyethylene, that is, 15%, was completely consistent with the molecular weight reduction predicted on the basis of eq. (2). A somewhat higher reduction in the elastic modulus with respect to the yield stress may be related to the change in the structure and morphology of the crystal and extent of crystallinity with a reduction of the molecular weight. The heat of fusion of PE(0), determined by DSC, was 130 J/g, whereas for PE(2), it was 123 J/g. The result indicated a slight decrease in the extent of crystallinity of degraded polyethylene. For HDPE, which has a simple chemical structure with a regular molecular chain, reducing the molecular weight may lead to an increase in the chain ends, which act as imperfections and change the crystallinity. Of course, the opposite behavior has been reported for degraded poly(ethylene terephthalate), in which the crystallinity increases with recycling, resulting in higher stiffness.¹⁹

Table III also presents the impact strength of virgin and degraded HDPEs. The degradation of the polyethylene made it brittle. The decrease in the molecular weight and change in the crystalline state can be explained as the main causes of the polymer brittleness.¹⁸

Effect of the glass fiber

To investigate the role of the glass-fiber content in the final properties of the composites, PP-GF(30) was mixed with virgin and degraded polyethylenes with different compositions, that is, up to 50 wt % PP-GF(30), with a twin-screw extruder. Figure 1 illustrates the variations of the tensile properties of the final product with the weight percentage of PP-GF(30). The tensile modulus and yield stress were enhanced continuously with the PP-GF(30) content for both the virgin and degraded polyethylenes. For instance, the tensile modulus of PE(2) filled with 37.5% PP-GF(30) increased from 1 to 1.9 GPa, showing a significant

TABLE IV
Composition of Composites Containing HDPE, GTR, and PP-GF(30)

Code	Composition by weight ^a
[PE(1)/GTR(0)]/[PP-GF(30)](12.5)	12.5% PP-GF(30) and 87.5% HDPE/GTR(0) with once extruded polyethylene
[PE(1)/GTR(12.5)]/[PP-GF(30)](12.5)	12.5% PP-GF(30) and 87.5% HDPE/GTR(12.5) with once extruded polyethylene
[PE(1)/GTR(25)]/[PP-GF(30)](12.5)	12.5% PP-GF(30) and 87.5% HDPE/GTR(25) with once extruded polyethylene
[PE(1)/GTR(0)]/[PP-GF(30)](37.5)	37.5% PP-GF(30) and 62.5% HDPE/GTR(0) with once extruded polyethylene
[PE(1)/GTR(12.5)]/[PP-GF(30)](37.5)	37.5% PP-GF(30) and 62.5% HDPE/GTR(12.5) with once extruded polyethylene
[PE(1)/GTR(25)]/[PP-GF(30)](37.5)	37.5% PP-GF(30) and 62.5% HDPE/GTR(25) with once extruded polyethylene
[PE(2)/GTR(25)]/[PP-GF(30)](37.5)	37.5% PP-GF(30) and 62.5% HDPE/GTR(25) with twice extruded polyethylene
[PE(0)/GTR(25)]/[PP-GF(30)](37.5)	37.5% PP-GF(30) and 62.5% HDPE/GTR(25) with virgin polyethylene

^a The numbers in parentheses following "HDPE/GTR" represent the weight percentages of GTR in the HDPE/GTR compositions.

improvement in the stiffness. However, at a specified PP-GF(30) content, the tensile modulus decreased as the degradation level of the HDPE matrix increased by a factor that was almost equal to that of the pure virgin and degraded polyethylenes. The same trend was observed for the yield stress. These results emphasize that the final mechanical performance of the composites is dominated by both the glass-fiber content and mechanical properties of pure polyethylene. In other words, the minimum glass-fiber content required to achieve the desirable mechanical properties for a composite cross tie depends on the mechanical properties of the recycled HDPE obtained from plastic waste.

Figure 2 presents the impact properties of HDPE filled with PP-GF(30). A significant reduction in the impact strength can be observed for the HDPE/PP-GF(30) composites even at relatively low fiber contents, and it decreased further with increasing fiber content. It is well known that a rigid filler such as glass fiber produces stress concentration in composites, resulting in reduced impact properties. In addition, the impact strength of the HDPE/PP-GF(30) composites based on the degradation level of HDPE followed this order: PE(0) > PE(1) > PE(2).

Effect of GTR

To investigate the influence of GTR on the final properties of the fiber-reinforced composites studied in the previous section, various compositions containing different contents of GTR were mixed with a twin-screw extruder. All GTR-filled compounds were mixed under the processing conditions mentioned in the Experimental section. Table IV lists the compositions of all the composites prepared in this study. The whole compound is divided into two parts: HDPE/GTR and PP-GF(30). This allows us to study the effect of the GTR content on the final mechanical properties at a given glass-fiber content. In addition, composites were made with both virgin and degraded polyethylenes.

The effect of the GTR content on the tensile properties of the final composite materials is illustrated in Figure 3. At a specified glass-fiber content, both the tensile modulus and yield stress decreased with the addition of GTR to the HDPE/PP-GF(30) composites. For instance, for compound HDPE/PP-GF(30) containing 37.5 wt % PP-GF(30), the addition of GTR up to 25 wt % reduced the tensile modulus and yield stress by about 20 and 30%, respectively. This behavior was expected because the GTR particles acted as soft fillers with low interactions with the matrix. The results also indicated that the elongation at yield increased slightly, particularly for lower contents of PP-GF(30), and this could be due to the low interaction between the matrix and GTR. Although

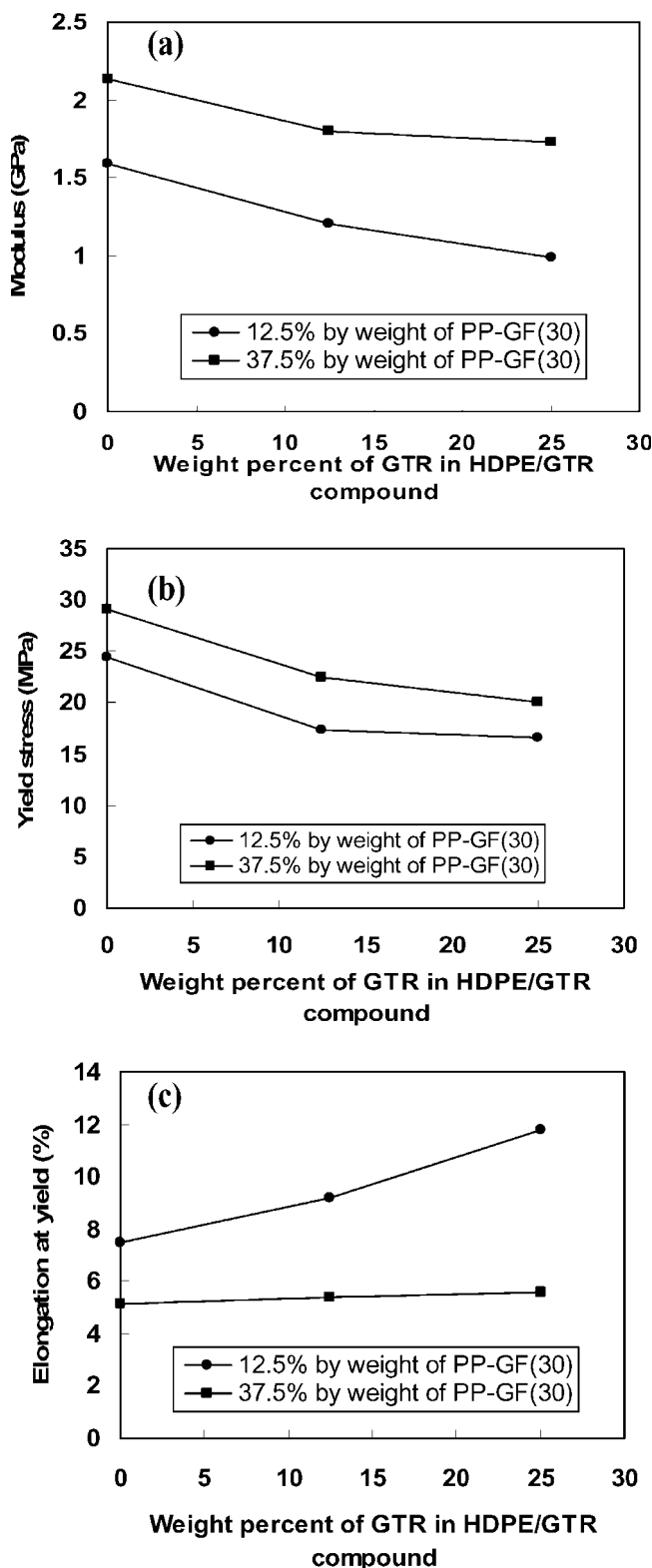


Figure 3 Variation of the tensile properties with the GTR content for the HDPE/GTR/PP-GF(30) composites: (a) tensile modulus, (b) yield stress, and (c) elongation at yield.

TABLE V
Mechanical Properties of HDPE/PP-GF(30)/GTR Systems Based on Virgin and Extruded Polyethylenes at Specified GTR and PP-GF(30) Concentrations

Composite	Tensile modulus (GPa)	Yield stress (MPa)	Elongation at yield (%)	Hardness (Shore A)	Impact strength (kJ/m ²)
[PE(0)/GTR(25)]/[PP-GF(30)](37.5)	1.8	20.3	4.9	68	70
[PE(1)/GTR(25)]/[PP-GF(30)](37.5)	1.74	19.9	5.6	68	67
[PE(2)/GTR(25)]/[PP-GF(30)](37.5)	1.72	20	5.7	67	60

the results showed a reduction in the mechanical properties with the addition of GTR, the final properties of the composite could be compensated by the addition of extra glass fiber to meet the required mechanical properties for applications such as railroad cross-ties.

Table V presents the role of the degradation of polyethylene in the mechanical properties of the HDPE/GTR/PP-GF(30) composites. Comparing the results of Table V with the mechanical properties of HDPE/PP-GF(30) presented in Figure 1, we found that the addition of GTR had the smallest negative effect on the mechanical performance of the composite materials based on degraded HDPE. For instance, incorporating 25 wt % GTR into [PE(0)]/[PP-GF(30)](37.5) reduced the tensile modulus from 2.3 to 1.8 GPa, whereas the same amount of GTR reduced the tensile modulus of [PE(2)]/[PP-GF(30)](37.5) from 1.95 to 1.72. This behavior could partly be attributed to the crystalline state of the polyethylene, which was further destroyed by the incorporation of GTR, but this effect for virgin polyethylene was much more pronounced.

In Figure 4, the effect of GTR on the impact strength of the final composite is shown. The impact

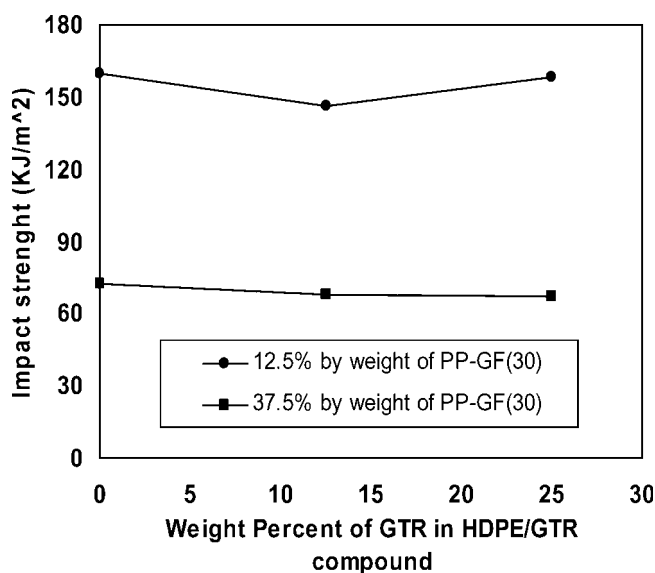


Figure 4 Variation of the impact strength with the GTR content for the HDPE/GTR/PP-GF(30) composites.

strength did not show improvements with the addition of rubber particles. The behavior may be associated with poor interfacial adhesion between GTR and the matrix, which could not completely transfer the load from the polyethylene matrix to the rubber particle.

Phase morphology

Figure 5 shows an SEM image of fracture surface of HDPE containing 37.5 wt % PP-GF. The glass fiber was uniformly distributed within the matrix, and the fiber was mostly oriented along the flow direction during the injection molding. The uniform dispersion of the glass fiber showed good adhesion to the surrounding matrix, and this denoted the effectiveness of the PP-GF system in reinforcing the HDPE-based composites. The SEM micrograph also indicates that the fiber length ranged from 200 to 500 μm in the final product. Figure 6 illustrates the morphology of a sample containing both glass fiber and GTR, that is, sample [PE(2)/GTR(25)]/[PP-GF(30)](37.5). The picture shows the poor adhesion of the GTR particle to the matrix. In addition, the presence of GTR in the composite slightly disturbed the orientation of the

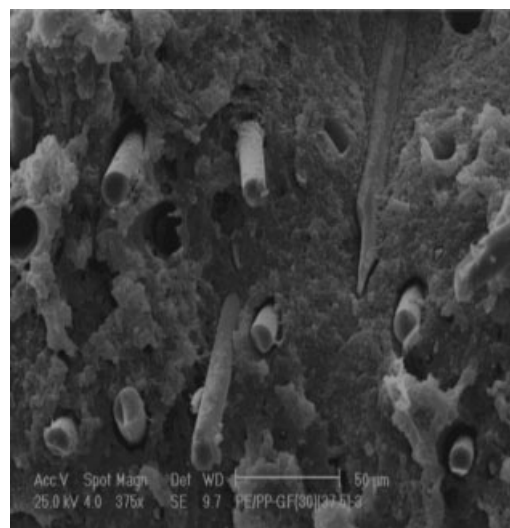


Figure 5 SEM micrograph of the fracture surface of HDPE filled with 37.5 wt % PP-GF.

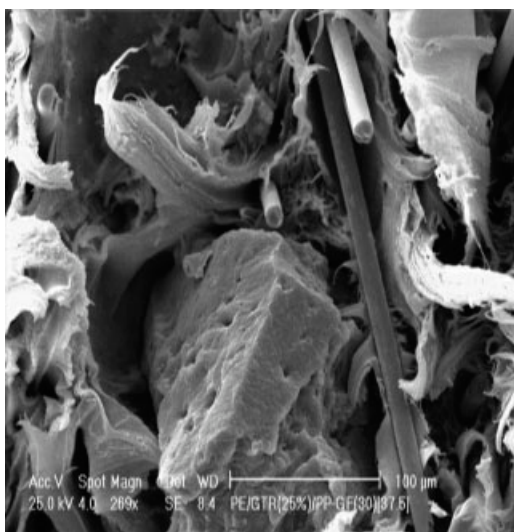


Figure 6 SEM micrograph of the fracture surface of [PE(2)/GTR(25)]/[PP-GF(30)](37.5).

glass fiber because of the melt flow, and this may have been partly responsible for the reduction of the stiffness of the composites containing GTR particles along the flow direction.

CONCLUSIONS

The mechanical performance of composite materials based on recycled HDPE, GTR, and PP-GF for structural applications such as railroad cross-ties was investigated. To control the final price of the product, untreated GTR was used, and the compounds were prepared without any additives. Besides, the effect of the degradation level of HDPE on the properties of the final composites was studied. Such composites seem to have potential for use in structural applications such as railroad cross-ties. The results

indicate that the addition of PP-GF increases the stiffness and strength of final composites. However, the final properties of a compound with a given glass-fiber content depend on the degradation level of HDPE. Moreover, the addition of untreated GTR reduces the tensile properties and does not produce any improvement in the impact strength because of the low interfacial adhesion between GTR and the matrix. Therefore, from the viewpoint of mechanical performance, the concentration of GTR in a final compound should be kept low, that is, less than 10 wt %. The results obtained in this study can help us obtain a suitable composition with minimum cost.

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