

Effects of Organic Peroxide and Polymer Chain Structure on Mechanical and Dynamic Mechanical Properties of Sisal Fiber Reinforced Polyethylene Composites

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Received 9 September 2011; accepted 3 November 2011

DOI 10.1002/app.36434

Published online 20 January 2012 in Wiley Online Library (wileyonlinelibrary.com).

ABSTRACT: Three types of polyethylene (PE), low-density PE (LDPE), linear low-density PE (LLDPE), and high-density PE (HDPE) were used as polymer matrices to prepare untreated as well as dicumyl peroxide (DCP) treated sisal fiber composites. The effect of polymer chain structure, addition of DCP, and sisal fiber loadings on the mechanical and dynamic mechanical properties of the composite was investigated in this study. It was found that the extent of improvement in tensile properties of the composite samples varied with respect to the polymer molecular characteristics. The elongation at break for all the composites decreased significantly. Young's modulus and the tensile strength of the treated LDPE and LLDPE composites increased significantly compared with the untreated composites, whereas Young's modulus of the

treated HDPE samples decreased observably compared with the untreated samples. DCP treatment, however, did not change the tensile strength of HDPE and its composites. The storage modulus results for all the PE composites correlate well with the tensile testing results. In the case of the LDPE and LLDPE samples, the curves of the mechanical loss factor ($\tan \delta$) show a clear relaxation around -18°C , which shifted to higher temperature in the treated composites, whereas for HDPE this transition was not seen. © 2012 Wiley Periodicals, Inc. *J Appl Polym Sci* 125: 2216–2222, 2012

Key words: polyethylenes; sisal fiber; composites; crosslinking; grafting; mechanical properties; dynamic mechanical properties

INTRODUCTION

During the last decade, the use of natural fibers as reinforcement in plastics has received considerable attention. Natural fibers have many advantages over their synthetic counterparts. They are cheap, widely available, renewable, recyclable, biodegradable, and have high specific strength. These properties are in agreement with the recent tendency and legislation toward using materials that are renewable and environmentally friendly. Therefore, many studies have been carried out on natural fibers reinforced polymer composites as alternative to synthetic reinforcing materials.^{1–5} Among natural fibers, sisal fiber has been widely used in polymer/natural fiber composites because of its wide availability, short renewable time, ease of cultivation, low cost, and excellent physical and mechanical characteristics.^{1,6}

However, there are some shortcomings of natural fibers, which affect their reinforcing capabilities. Natural fibers have a tendency to absorb moisture

due to their hydrophilic nature as well as to form aggregates during processing. In addition, their thermal stability (start degrading above 200°C) limits their usage in certain polymer matrices. On top of that, because of their hydrophilic nature, natural fibers are incompatible with hydrophobic polymers, which leads to weak interfacial bonding between the two components. Consequently, this will deteriorate the mechanical properties and performance of the resulting composites.^{1,7,8} Hence, modification of the fiber and/or the polymer is required to improve the compatibility and consequently the performance of the composites.

Pretreatments of natural fibers can clean the fiber surface, chemically modify the surface, increase the surface roughness, and reduce the moisture absorption process. Modifications of the polymer matrix and the fiber during processing, and of the polymer matrix alone, have been reported to improve the interfacial bonding. These various strategies were reviewed by a number of researchers.^{9–13} Among the various treatments, peroxide treatment of cellulose fiber, as well as the addition of peroxide to the molten polymer-fiber mixture during processing, has attracted the attention of various researchers due to easy processability and the improvement in the mechanical properties. Addition of dicumyl peroxide (DCP) to the composite mixture during melt mixing

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Contract grant sponsors: National Research Foundation in South Africa, University of the Free State.

TABLE I
Densities, Melt Flow Indices, and Molar Masses of the Neat Polyethylenes

Sample	ρ (g cm ⁻³)	Melt flow indices ^a	M_n (g mol ⁻¹)	M_w (g mol ⁻¹)
LDPE	0.922	2	30,302	206,797
LLDPE	0.924	1	68,409	290,056
HDPE	0.956	2	27,575	539,389

^a Melt flow indices = g/10 min at 190°C/2.16 kg for LDPE and LLDPE and g/10 min at 190°C/5 kg for HDPE.

can give rise to crosslinking of the polymer chains and grafting between the polymer chains and the natural fibers. However, because of the presence of three reactive hydroxy groups on each cellulose unit, the grafting of the cellulose fibers to the polymer chains should dominate because of the higher free radical reactivity of the hydroxyl groups.^{14–16}

Polyethylenes (PEs) have been widely used as matrix materials in natural fiber reinforced composites due to their excellent value (cost and performance), relatively modest physical properties, ease of processing, recyclability, and adequate mechanical properties. There are three major classes of PEs, high-density PE (HDPE), low-density PE (LDPE), and linear low-density PE (LLDPE). The basic difference between these three types of PEs lies in the degree and regularity of branching. Although HDPE has very few branches, LDPE is characterized by significant branching with long, irregular branches at irregular intervals. LLDPE, on the other hand, is characterized by short branches of regular length at regular intervals.^{17–19} The molecular structure of a PE is an important characteristic influencing the physical and chemical properties. It has been reported that the structural parameters such as number average molecular weight, the molecular weight distribution, the presence of unsaturated functional groups (vinyl groups), and the content of branches as well as their length had important effects in the crosslinking/grafting behavior of PEs.^{20–23}

The objective of this study is to investigate the effects of PE molecular characteristics, the addition of DCP, and the sisal fiber loadings on the mechanical and the dynamic mechanical properties of sisal fiber reinforced LDPE, LLDPE, and HDPE composites. DCP was used to improve the interfacial adhesion between the PE matrices and sisal fiber through initiation of both crosslinking of PEs chains and grafting of the chains onto the sisal fiber surfaces.

EXPERIMENTAL

Materials

Sisal fiber was obtained from the National Sisal Marketing Committee in Pietermaritzburg, South Africa.

LDPE and LLDPE were supplied in pellet form by Sasol Polymers, Johannesburg, South Africa, whereas HDPE was supplied by Safripol, South Africa. The densities (ρ), melt flow indices, and molar masses of the neat PEs are summarized in Table I.

DCP (bis(α,α -dimethylbenzyl)peroxide) was supplied by Merck, South Africa. It has a minimum assay of 98%. Petroleum ether was also supplied by Merck.

Treatment of sisal fibers

Sisal fibers were cut into an average length of between 5 and 10 mm, soaked in distilled water for 24 h to remove any surface impurities, filtered, and washed thoroughly with distilled water before being allowed to dry in an oven at 80°C for 48 h. The dried fibers were washed with petroleum ether for 5 h at 50°C to remove waxy materials and natural oils, followed by oven drying at 80°C.

Preparation of polyethylene composites

The PE-sisal fiber composites were prepared via melt mixing in a 55 mL mixing chamber of a Plastograph-W50EHT (Brabender® OHG, Germany) at 150°C, 30 rpm and 10 min. DCP treated samples were prepared by addition of 1 phr DCP to the mixture 1 min before the end of the mixing. The melt pressing of the prepared samples was performed at 175°C and 50 bar for 10 min. Table II shows the compositions of the PE composite samples used in this study.

Characterization methods

The tensile properties of the neat polymers and the composites were determined using a Hounsfield H5KS tensile tester at a crosshead speed of 50 mm min⁻¹ at ambient temperature. Dumbbell-shaped test specimens of 75 mm total length, 13 mm width at the two ends, 5-mm neck width, 20-mm gauge

TABLE II
Compositions of the Composite Samples Used in This Study

LDPE/sisal/ DCP (w/w)	LLDPE/sisal/ DCP (w/w)	HDPE/sisal/ DCP (w/w)
100/0/0	100/0/0	100/0/0
100/0/1	100/0/1	100/0/1
90/10/0	90/10/0	90/10/0
80/20/0	80/20/0	80/20/0
70/30/0	70/30/0	70/30/0
90/10/1	90/10/1	90/10/1
80/20/1	80/20/1	80/20/1
70/30/1	70/30/1	70/30/1

length, and 1.5-mm thickness were used. The stress and the elongation at yield, Young's modulus, as well as the tensile strength and elongation at break were determined from the stress–strain curves. At least eight samples were tested for each composition, and the mean values are reported.

The dynamic mechanical properties (storage and loss modulus as well as $\tan \delta$) of the samples were determined in a Perkin Elmer Diamond dynamic mechanical analyzer (DMA). Rectangular bar specimens with dimensions of $50 \times 10 \times 1.5 \text{ mm}^3$ were used for this study, there was no pretreatment after composite preparation, and the samples were stored in the dark under ambient conditions. The measurements were carried out in the dual cantilever bending mode and the corresponding viscoelastic properties were determined as a function of temperature. The samples were heated under nitrogen flow (30 mm min^{-1}) from -100°C to 100°C at a heating rate of 5°C min^{-1} , and at a frequency of 1 Hz.

RESULTS AND DISCUSSION

Tensile properties

The tensile properties of the PE/sisal fiber composites as a function of sisal fiber content are shown in Figures 1–3. As can be seen from Figure 1, the elongation at break of the treated and the untreated LDPE composites decreased significantly with incorporation of the sisal fiber, and it further decreased with increasing filler content. No significant differences in the elongation at break between the treated and the untreated samples could be seen. This can be explained by the sisal fibers imparting rigidity and brittleness to the LDPE matrix.^{24,25} The tensile modulus of the composites (Fig. 2) was found to

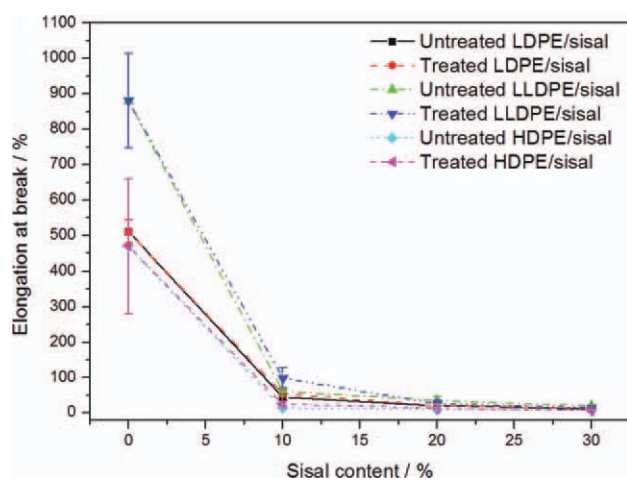


Figure 1 Elongation at break as function of sisal fiber content for untreated and DCP treated polyethylenes composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

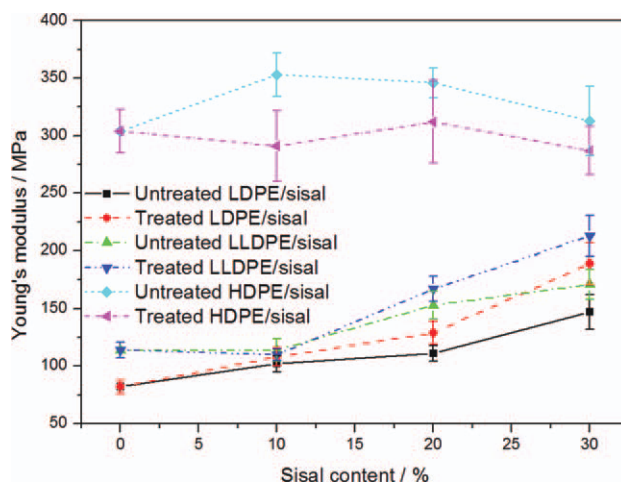


Figure 2 Tensile modulus as function of sisal fiber content for untreated and DCP treated polyethylenes composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

increase significantly with the presence of sisal fiber as well as with the addition of DCP. The treated composites showed higher values compared with the untreated ones for all fiber loadings. The increase in the modulus upon fiber addition is ascribed to the higher modulus of the lignocellulosic fibers compared with LDPE, whereas the treatment of the composites with DCP resulted in better interfacial adhesion between the fiber and the polymer matrix,^{24,25} as well as in crosslinking of the polymer that will contribute to the increase in stiffness. The improved interfacial adhesion leads to good stress transfer from the matrix to the fiber and the mechanical properties of the fiber are fully used. This result is in agreement with the morphological observations and gel contents that were described in another

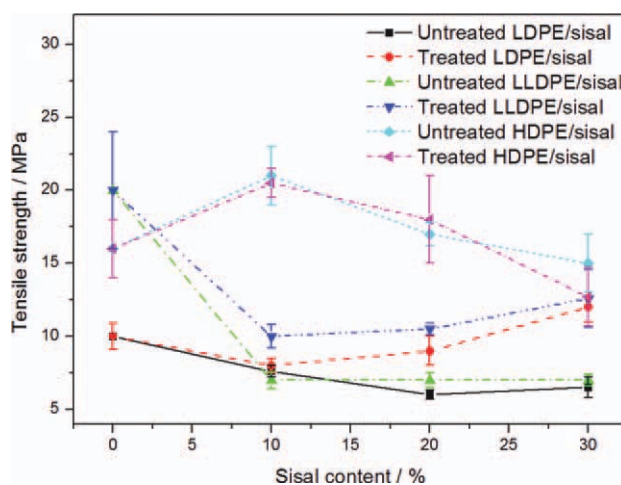


Figure 3 Tensile strength as function of sisal fiber content for untreated and DCP treated polyethylenes composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

paper.²⁶ Figure 3 shows the tensile strength as a function of fiber loading of the treated and the untreated LDPE composites. The tensile strength of the untreated composites is lower than that of the unfilled polymer matrix and the treated composites. The treated composites show a gradual increase in tensile strength with increasing fiber content. An almost 20% increase in tensile strength of the composite with 30 wt % fiber is observed when compared with the neat LDPE.^{25,27} This increase is a clear indication of the improved interfacial adhesion between the polymer matrix and the sisal fiber, which was described in our other paper.²⁶

The tensile results of LLDPE and its composites are summarized in Figures 1–3. As expected, the elongation at break of the LLDPE composites (Fig. 1) decreased significantly with the presence of the sisal fiber, and even further decreased with increasing fiber content. A considerable increase in the tensile modulus (Fig. 2) was observed with increasing fiber content. The tensile modulus of the treated composites was higher than those of the untreated ones, especially at higher fiber loadings. The decrease in the elongation at break as well as the increase in the tensile modulus could be explained in the same way as for LDPE. Figure 3 shows the dependence of the tensile strength of the untreated and the treated LLDPE composites on the fiber content. The tensile strength of the untreated and treated composites decreased noticeably with the incorporation of 10 wt % sisal fiber, and thereafter, the tensile strength of the untreated samples remained almost unchanged with increasing fiber loadings. The tensile strength of the treated composites was, however, found to increase slightly with an increase in fiber content. The tensile strength of the treated 70/30 w/w LLDPE/sisal composite was about 80% higher than that of the untreated composite. This is the result of a lack of interfacial adhesion between the fiber and the polymer in the case of the untreated samples, which resulted in poor stress transfer from the polymer to the reinforcing filler. The grafting in the treated composites resulted in good stress transfer from the LLDPE matrix to the fiber.

In the case of the HDPE composites (Figs. 1–3), the elongation at break of the treated and the untreated composites was found to decrease in a similar way as for the LDPE and LLDPE composites. The tensile modulus of the untreated HDPE composites increased by about 20% in the presence of 10 wt % fiber, but decreased slightly at higher fiber contents (Fig. 2). However, the tensile modulus for all the untreated HDPE composites was higher than that of neat HDPE because of the higher stiffness of the fibers. The tensile modulus of the treated composites did not change in the presence of fiber, and with increasing fiber content. This could have been

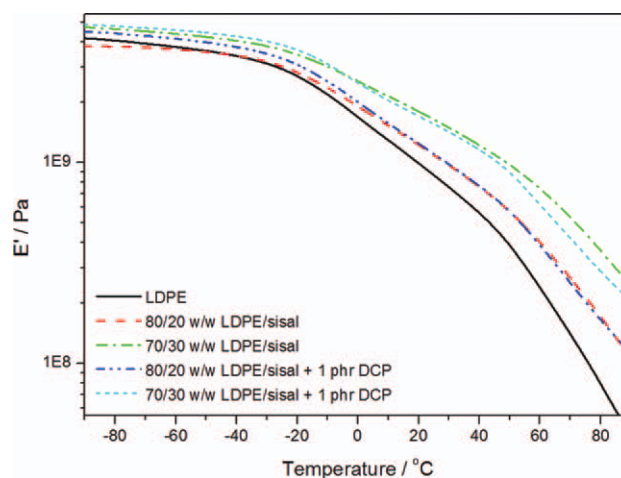


Figure 4 Storage modulus versus the temperature for neat LDPE as well as its untreated and DCP treated sisal composites. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://www.interscience.wiley.com).]

due to the reduction in the crystallinity of the HDPE matrix after addition of DCP, as was observed from DSC results.²⁶ The increase in modulus as a result of the presence of the stiffer fibers was obviously balanced by the decrease in modulus as a result of the decreased crystallinity brought about by crosslinking in the presence of DCP. It was reported that the tensile modulus of PE polymers depends strongly on its crystallinity. In general, the tensile modulus changes approximately linearly with the degree of crystallinity.²⁸ The tensile strength of the untreated and the treated HDPE/sisal composites increased at lower fiber loadings, and then decreased with an increase in fiber loading (Fig. 3). In general, no significant differences in tensile strength between the untreated and the treated composites were observed. The reason for this is that, because there was no grafting between the polymer chains and the fibers in the case of the DCP treated HDPE composites, the interaction (or lack thereof) between HDPE and the fibers was the same, whether the samples were prepared in the presence or absence of DCP.

Dynamic mechanical analysis

Dynamic mechanical measurements were performed on the neat PEs as well as the untreated and DCP treated sisal fiber composites to display the effect of the fiber loading, molecular characteristics of the polymer, and the DCP treatments on the viscoelastic properties of the composites. The storage modulus and $\tan \delta$ of the composite samples as function of temperature are shown in Figures 4–9. As can be seen in Figure 4, the storage modulus increases with increasing amount of sisal fiber, and all the composites show higher values than the neat LDPE,

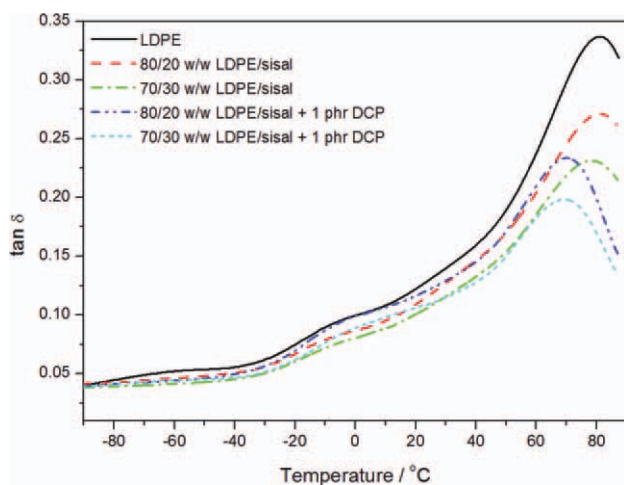


Figure 5 $\tan \delta$ versus the temperature for neat LDPE as well as its untreated and DCP treated sisal composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

especially at temperatures above the glass transition. The treated composites also show slightly higher storage modulus values than their untreated counterparts at temperatures below the glass transition. However, at higher temperatures, the curves of the treated and the untreated composites were almost overlapped. The increase in the storage modulus upon fiber addition is ascribed to the higher modulus of the lignocellulosic fibers compared with that of LDPE, whereas the DCP treatment of the composites resulted in better interfacial adhesion between the fiber and the polymer,^{24,25} as well as in cross-linking of the polymer, that would have contributed to the increase in stiffness.

It has been reported that DMA measurements can give information on the interface between natural

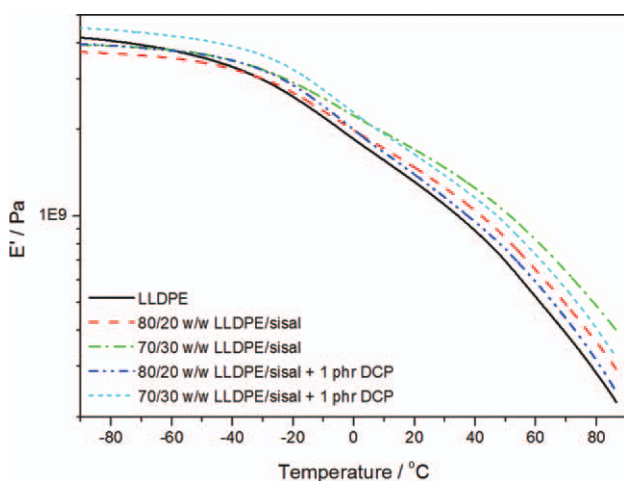


Figure 6 Storage modulus versus the temperature for neat LLDPE as well as its untreated and DCP treated sisal composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

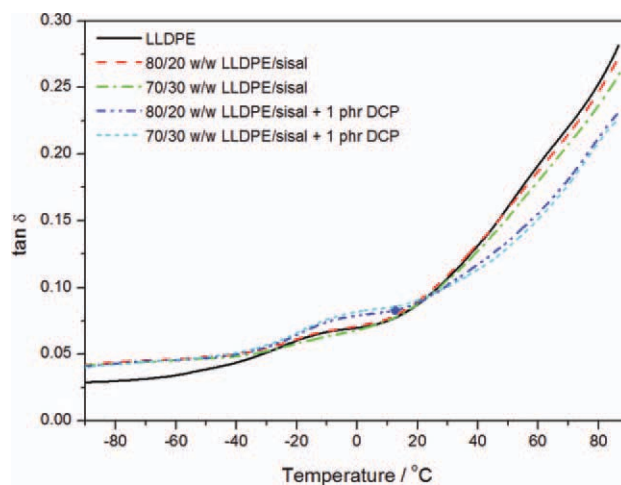


Figure 7 $\tan \delta$ versus the temperature for neat LLDPE as well as its untreated and DCP treated sisal composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

fibers and polymer matrices.^{29,30} In the case of good adhesion between the natural fibers and the polymer matrix, a shift in the glass transition toward higher temperatures is expected. Moreover, it was observed that good interfacial bonding has resulted in composite materials with lower energy dissipation than the poorly bonded or untreated composites. $\tan \delta$ as function of temperature for the LDPE composites is shown in Figure 5. The $\tan \delta$ curves of all the composites show lower values than the neat polymer matrix. The treated composites show slightly lower values than the untreated ones, especially at higher temperatures. Furthermore, the $\tan \delta$ curves show two relaxation processes located around -18°C (glass transition) and $69\text{--}80^\circ\text{C}$ (α -transition). The first relaxation process is called the β -relaxation, which is

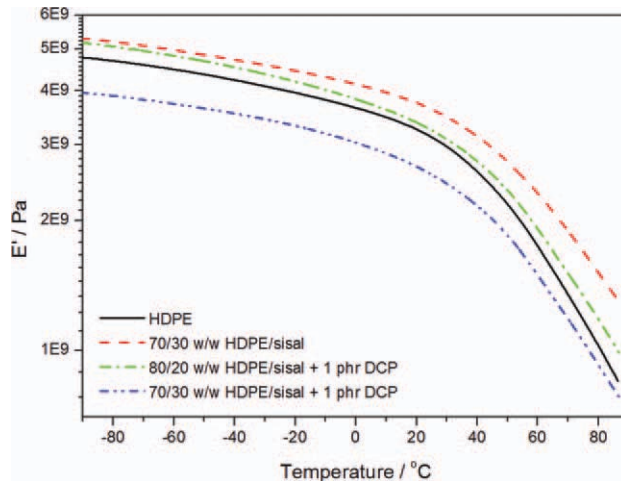


Figure 8 Storage modulus versus the temperature for neat HDPE as well as its untreated and DCP treated sisal composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

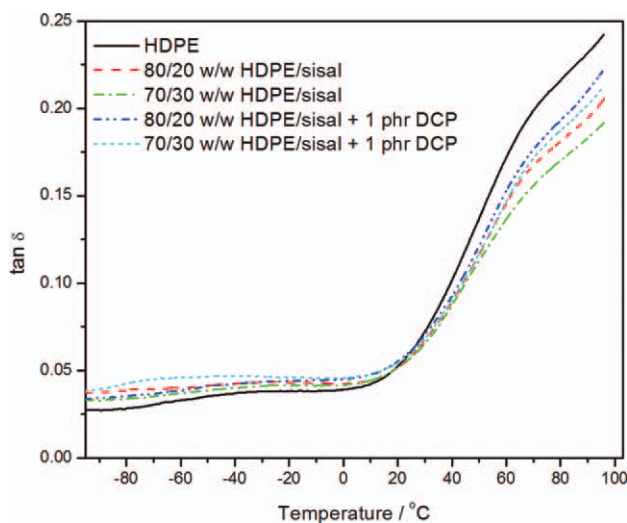


Figure 9 $\tan \delta$ versus the temperature for neat HDPE as well as its untreated and DCP treated sisal composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

generally accepted as being the glass transition in PEs.^{31,32} For the treated composites, there is a slight shift to higher temperatures (-13°C) of this transition, which is the result of the immobilization of the amorphous polymer chains through crosslinking/grafting. This is in agreement with the findings of the gel contents as well as the scanning electron microscopy (SEM) observations, which confirmed an enhanced interfacial adhesion as a result of grafting in the treated composites.²⁶ The relaxation process in the temperature range $69\text{--}80^{\circ}\text{C}$ is associated with the molecular motion within the crystalline phase. For the neat LDPE and the untreated composites, this relaxation occurred around 80°C , whereas for the treated composites it shifted to 69°C . This could be explained by the fact that crosslinking/grafting reduced the lamellar thickness of the polymer matrix and hence eased the polymer chain motions. Sirotkin and Brooks³³ studied three PE copolymers, which differed in short chain branch content, to establish the effect of the morphology on the α -, β -, and γ -relaxations. They found that the α -relaxation temperature increased with lamellar thickness, irrespective of the grade or crystallinity, and is associated with c-shear within the crystalline lamellae.

Figures 6 and 7 show the storage modulus and $\tan \delta$ of the LLDPE composites as function of temperature. It is evident from Figure 6 that the storage modulus of all the composite samples is higher than that of the neat LLDPE at the glass transition and higher temperatures. At temperatures above the glass transition, the storage modulus of the untreated samples was somewhat higher than that of the treated ones. This is probably due to the slight decrease in the crystallinity of the polymer matrix

after treatment with DCP, which was apparent from the DSC results.²⁶ The $\tan \delta$ curves of the LLDPE composites as a function of temperature are shown in Figure 7. It is obvious that the intensity of the glass transition at -16°C of the treated samples increased, and that the transition shifted to higher temperatures, compared with both the untreated samples and the neat LLDPE matrix (-23°C). The increase in the intensity of the glass transition peak is due to the increase in the volume fraction of the amorphous phase as a result of the crosslinking/grafting processes, which restricted the polymer chains packing and hence reduced the crystallinity of the matrix.²⁶ The shift in the glass transition is probably due to the improved interfacial interactions between the polymer matrix and the sisal fiber, which restricts the segmental mobility of the LLDPE chains in the amorphous phase and hence shifts the β -transition to higher temperatures. The SEM and gel content results for the treated LLDPE composite samples showed good interfacial bonding between the polymer and sisal fiber.²⁶ The $\tan \delta$ values of the treated composites were also lower than those of the untreated composites and the neat LLDPE above the glass transition temperature, which indicates better interfacial interactions and less dissipation of energy.

The variation of the storage modulus of HDPE and its composites as a function of temperature is shown in Figure 8. The storage modulus values of the untreated samples increased with increasing fiber content over the whole temperature range, and they were higher than those of the treated samples, which decreased with increasing fiber content. These results show the same trend as the tensile modulus results, and can be explained in the same way. The dependence of $\tan \delta$ of HDPE and its treated and untreated composites on temperature is shown in Figure 9. The β -relaxation, which was seen in the $\tan \delta$ curves of the LDPE and LLDPE samples (Figs. 5 and 7), is not clearly seen in the case of HDPE and its composites. This could be ascribed to the higher crystallinity of the HDPE matrix. At higher temperatures, the $\tan \delta$ values of the untreated composites were lower than those of HDPE and the treated composites. This is probably due to the reduction in the crystallinity of the polymer matrix after treatment with DCP, which enhances the molecular motions of the amorphous phase and hence leads to higher dissipation of energy. It was expected that the reduction of the polymer's crystallinity due to DCP treatment would be counterbalanced by the enhanced interfacial bonding between the polymer matrix and the sisal fibers. However, the SEM and gel content results²⁶ both demonstrated poor interfacial bonding as well as less grafting in the case of the HDPE composites.²⁶ This can explain why the

DCP treated composites had higher dissipation of energy compared with their untreated counterparts.

CONCLUSIONS

In this study, the influence of the PE molecular characteristics, the sisal fiber loadings, and DCP treatment on the mechanical and the dynamic mechanical properties of PE-based sisal fiber composites were investigated. It was found²⁶ that the PE molecular characteristics play a crucial role in its crosslinking/grafting behavior. The degree of crosslinking/grafting was higher for the PEs with higher number average molecular weights, higher contents of vinyl groups, relatively lower molecular weight distributions, and higher branching contents. In our study,²⁶ it was found that the LLDPE had the highest crosslinking/grafting efficiency, followed by the LDPE and then the HDPE, for which no grafting was observed.

This crosslinking/grafting efficiency was clearly reflected in the mechanical and dynamic mechanical properties of the PE composites. The elongation at break was found to decrease significantly for all the PEs composites, and DCP treatment did not seem to make any difference. This is common for all particulate filled PEs, and crosslinking/grafting was not expected to make any difference. The grafting did, however, positively influence the tensile strength and modulus of the LDPE and LLDPE composites, because of the improved interfacial bonding between the fiber and the matrix. In the case of the HDPE composites, the DCP treatment had a marginal influence on the tensile strength, whereas the tensile modulus of the treated composites was smaller than that of the neat HDPE and the untreated composites. This is probably due to a combination of the reduction in the crystallinity of the polymer matrix as a result of DCP treatment, and the absence of grafting between the fiber and the matrix.

The DMA results were also in line with the effect of DCP treatment on the composite structures. For all three PEs, the storage moduli above T_g of the DCP treated samples were lower than those of the untreated samples. It seems as if the decrease in crystallinity as a result of crosslinking/grafting more strongly influenced the storage modulus values than the grafting between the matrices and the fibers. For both LDPE and LLDPE, the glass transition temperatures increased as a result of crosslinking/grafting due to the immobilization of the polymer chains, although the effect is more visible in the case of LLDPE. In the case of LDPE, the α -transition temperature observably decreased for the DCP treated sam-

ples, which is in line with the reduced lamellar thickness.

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