Utilization of Lignocellulosic Fibers in Molded Polyester Composites

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ABSTRACT: Using rice straw, bagasse, and cotton stalk fibers as reinforcing fillers in polyester composite was studied. The effects of fiber loading and fiber size on the modulus of rupture (MOR), modulus of elasticity (MOE), tensile strength, water absorption, and thickness swelling were investigated as well. The effect of esterification using maleic anhydride on the aforementioned properties also was studied. Ultraviolet light resistance and thermal behavior of the produced composites were also investigated. © 2002 Wiley Periodicals, Inc. J Appl Polym Sci 87: 653–660, 2003

Key words: rice straw; cotton stalks; bagasse; polyester; composite; esterification; maleic anhydride

INTRODUCTION

Thermosets are the most commonly employed matrices in the production of fiber-reinforced composites. This is because of the easy processing and manufacture of long-fiber composites, as in general these resins are provided as liquids or molding powders. Polyester is one of the thermoset matrices most frequently employed in the production of fiber-reinforced composites, as it is provided as liquid, is easily processed and cured, is low in cost, is easily available, and has good mechanical properties when reinforced. Therefore, polyesters are suitable for a variety of applications and are adaptable to the fabrication of large structures. Reinforced unsaturated polyesters represent about 70% of all matrix polymers in composites, with the remaining 30% being evenly shared by thermoplastics and other reinforced thermosets.[1] Many lignocellulosic fibers have been studied as reinforcing fillers in polyester composites such as jute fiber,[2–6] coconut,[7] pineapple leaf,[8] sisal,[9–11] sunhemp,[12] sponge gourd,[13] banana fibers,[14] Curaua fiber,[15] jute/cotton woven fabric.[16] Nabi and Jog[17] reviewed the use of natural fibers in composites, with special reference to the type of fibers, matrix polymers, treatment of fibers and fiber-matrix interface. Substitution of natural fibers (flax, sisal, hemp, and jute) for the hitherto used glass fiber in polyester based automobile parts has been highlighted by Asche.[18]

The main objective of the present study was to evaluate the mechanical properties and dimensional stability of polyester composites containing rice straw, bagasse, and cotton stalk fibers, in addition to the esterified derivatives of these fibers. The research also investigated the UV resistance and thermal behavior of the produced composites.

EXPERIMENTAL

Lignocellulosic fibers preparation

Edfu Co. for Pulp (Edfu, Egypt) kindly supplied bagasse in the form of 2- to 3-cm-long fibers. Cotton stalks and rice straw were collected from local fields and cut into lengths about 2–3 cm long before milling. Cotton stalks, bagasse, and rice straw were milled using 0.55-, 0.35-, 0.2-, and 0.15-cm screens. The fibers were then oven-dried at 105°C for 6 h before making the composites. The chemical composition of the different fibers is shown in Table I.

Polyester

The matrix polymer was a maleic polyester (PE) resin, Siropol 8340-TP (Saudi Industrial Resins Limited, Jeddah, Saudi Arabia), containing 40% styrene monomer and 2% calcium naphthenate as catalyst. Two percent (based on the weight of the polyester) of methyl ethyl ketone peroxide was used as an initiator.

PE composite making

Different ratios (15%–30%) of oven-dry fibers were mixed with the polyester resin, to which the initiator was premixed, and the resulting mixture was pressed in a mold 30 cm × 10 cm in size under a light pressure of 1 MPa for 40 min to ensure complete curing of the

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TABLE I
Chemical Composition of Lignocellulosic Fibers

	Ethanol–Benzene extractives (%)	Lignin (%)	Hemicellulose (%)	α Cellulose (%)	Ash (%)
Rice straw	1.6	18.9	19.3	45.0	14.7
Bagasse	2.1	22.7	23.6	48.4	1.3
Cotton stalks	2.8	24.9	22.1	46.8	4.1

polyester resin. The thickness of the produced composites was 0.4–0.6 cm.

Esterification of lignocellulosic fibers by maleic anhydride

To 100 g of oven-dry fibers, 100% (based on the weight of the fibers) maleic anhydride (MA) was applied to the fibers. Maleic anhydride was first dissolved in the least possible amount of acetone. The mixture was thoroughly mixed and kept in a fume hood to allow acetone evaporation, then put in a preheated oven for 2 h at 100°C. The excess nonreacted anhydride was removed from the fibers by acetone by soxhlet extraction. The fibers were air-dried, then oven-dried at 105°C for 6 h. The weight percent gain (WPG) from the esterification was calculated. WPGs of 12.6, 14.3, and 15.7 were obtained for bagasse, rice straw, and cotton stalks, respectively.

Fourier transform infrared analysis

Infrared spectra of the lignocellulosic fibers and maleic anhydride–esterified fibers were obtained by using JASCO FTIR 800 E spectrometer. The samples were measured using KBr disc technique.

Thermogravimetric analysis

A Perkin-Elmer thermogravimetric analyzer was used to study the thermal properties of the lignocellulosic fibers and lignocellulosic fibers–polyester composites. The heating rate was set at 10°C/min over a temperature range of 50°C–500°C. Measurements were carried out in a nitrogen atmosphere with a rate of flow of 50 cm^3/min.

UV light resistance

The resistance of the different lignocellulosic–PE composites to UV light was measured using a Fade-Ometer instrument. The temperature and humidity were set at 30°C and 50%, respectively, for 50 h. The wavelength range of emitted light was from 350 to 430 nm.

Testing of composites

Static bending [modulus of rupture (MOR) and modulus of elasticity (MOE)], tensile strength, and water absorption and thickness swelling tests of the produced composites were determined according to the ASTM D790-90,[19] ASTM D638-90,[20] and ASTM D570-81, respectively.[21] For the water absorption and thickness swelling test, the samples were immersed in water for 7 days at room temperature. For flexural properties testing a three-point loading system was used, and the span-to-depth ratio was 16:1. All samples were conditioned at 30% relative humidity before mechanical testing. Five specimens of each sample were tested and the results averaged.

RESULTS AND DISCUSSION

Effect of fiber loading and fiber size on the properties of the produced composites

Properties of composites depend on the interaction between the matrix polymer and the filler used. Polyester resin contains polar groups that can form hydrogen bonds with hydroxyl groups on the surface of the lignocellulosic fibers. So it is important to get the maximum possible interaction between the fibers and the matrix polymer without deteriorating the strength of the fibers. Because cotton stalks (a woody residue), rice straw, and bagasse have different morphology, it is important to determine the suitable conditions of milling and percentage of fibers in the composites of each of these lignocellulosic materials. Therefore, the fibers were milled using different screen sizes to reach the optimum fiber dimensions.

Figures 1–3 show the MOR, MOE, tensile strength, water absorption, and thickness swelling of bagasse–polyester (B–PE), rice straw–polyester (RS–PE), and cotton stalks–polyester (CS–PE) composites containing different ratios of fibers that were milled using different screen sizes. From the figures it is clear that the properties of the composites depended on both the fiber size and loading, but in many cases at low fiber loading there was an irregular trend for the fiber loading and fiber size and the strength properties. This may be due to the nonhomogeneous distribution of the fibers at low fiber. When bagasse fibers were used
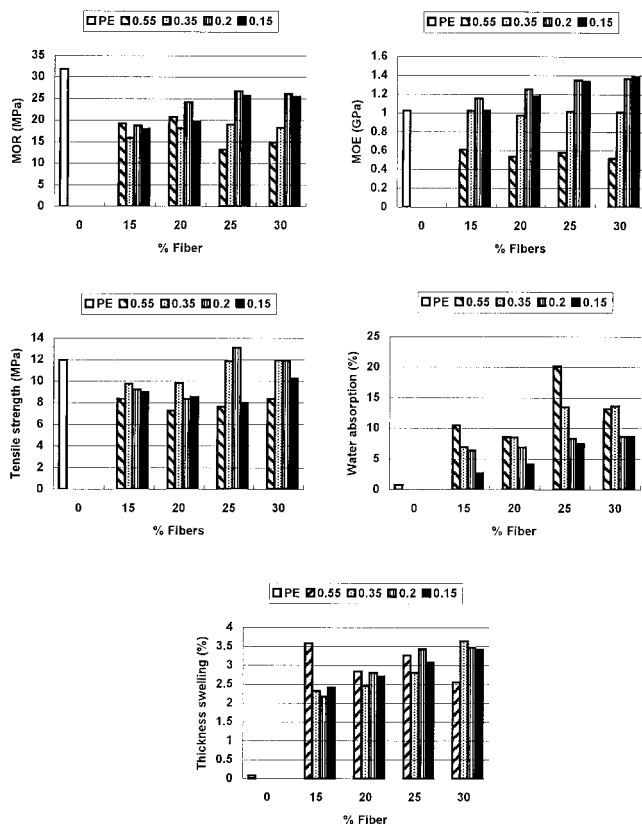
Figure 1 Effect of fiber loading and fiber size on properties of bagasse fiber–PE composites.

(Fig. 1), MOR generally increased on increasing fiber loading up to 25%. MOR of the neat PE composite was significantly higher than the different B–PE composites. At high fiber loading (25%–30%), MOR increased on decreasing the fiber size up to 0.2 cm. The MOE of B–PE composites that were made using 15%–30% of the fibers milled using a 0.15- to 0.2-cm screen was higher than that of the neat PE composite. The maximum MOE was recorded for the composites made from 25%–30% of the bagasse fibers milled using a 0.15- to 0.2-cm screen. Tensile strength of the composites also decreased at the lower fiber loading (15%–20%). Critical content of fiber is required before the strength of the composites become greater than that of the matrix polymer (neat PE). The maximum tensile strength was recorded for the composite containing 25% of the bagasse fibers milled using a 0.2-cm screen. This composite had a slightly higher tensile strength than the neat PE composite. As expected, water absorption and thickness swelling of the different B–PE composites were a lot higher than the neat PE composite because of the hydrophilic nature and porous structure of bagasse fibers and. Water absorption was higher using fibers that were milled using bigger screens (0.55 and 0.35 cm) and higher fiber loading. Irregular trend was found for the effect of fiber size and loading on thickness swelling. This may be be-

cause of unhomogeneous distribution of the fibers in the matrix polymer.

When using rice straw in the composites (Fig. 2), MOR generally increased with increasing fiber loading and with decreasing fiber size up to the 0.35-cm screen; the MOR of some composites was close to or slightly higher than that of the neat PE composite. MOE also was lower on using a larger fiber size and increased on increasing fiber loading up to 20%. The MOE of some rice straw–PE composites significantly exceeded that of the neat PE composite. Tensile strength also increased on increasing the fiber loading. The fiber size of rice straw had no significant effect on tensile strength, except at high fiber loading (30%), when maximum tensile strength was obtained using rice straw fibers milled using a 0.35-cm screen. Tensile strength was slightly to significantly higher than that of the neat PE composite at different fiber sizes using 25%–30% fiber loading. Water absorption and thickness swelling generally decreased with decreasing fiber size and fiber loading. When cotton stalks were used (Fig. 3), a similar trend for the effect of fiber loading and size on MOR and MOE was found. The maximum MOR was recorded for the composite containing 25% of cotton stalk fibers milled using a 0.2-cm screen. Tensile strength of the composites exceeded that of neat PE only on using 20%–25% of cotton stalk
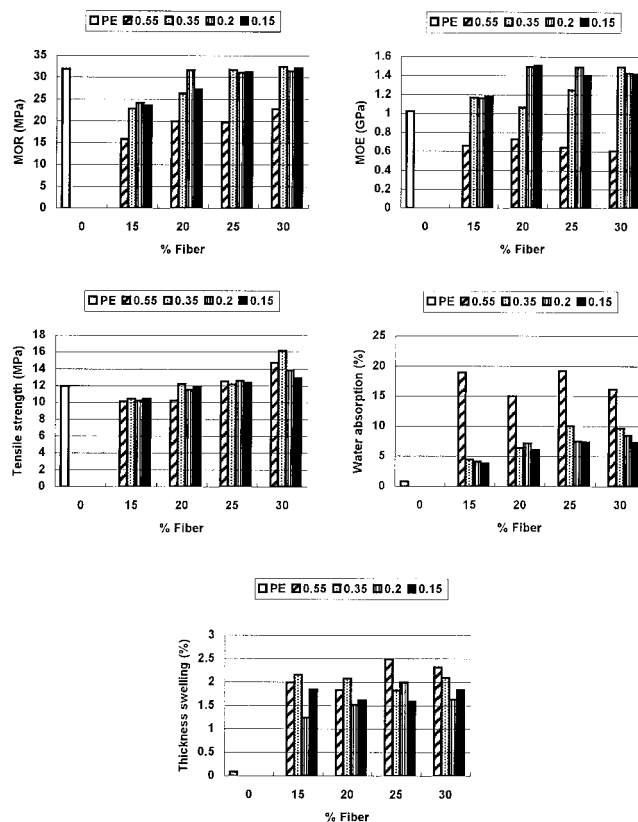

Figure 2 Effect of fiber loading and fiber size on properties of rice straw fiber–PE composites.
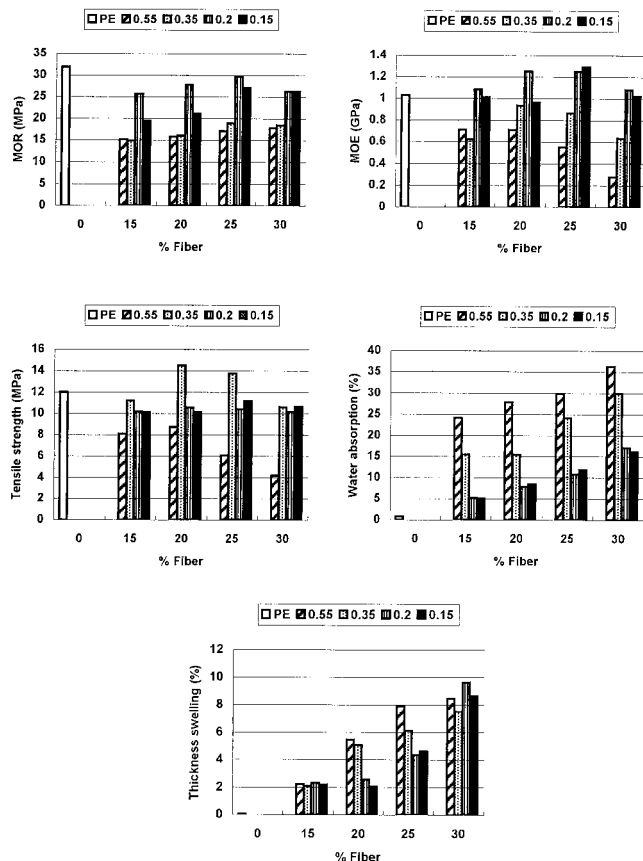
Figure 3 Effect of fiber loading and fiber size on properties of cotton stalk fiber–PE composites.

fibers milled using a 0.35-cm screen. The effect of fiber loading and size on water absorption and thickness swelling followed a trend similar to that of the RS–PE and B–PE composites.

Figure 4 shows the properties of selected lignocellulosic composites that have high strength properties and their water absorption and thickness swelling. As shown in Figure 4, the RS–PE composite had the highest MOR, which exceeded that of the neat PE composite. Also, all the lignocellulosic fiber–PE composites had close MOE, which was higher than that of the neat PE. All lignocellulosic fiber–PE composites had much, much higher water absorption and thickness swelling than did the neat PE composites. The rice straw–PE composite had the lowest thickness swelling. This may be because of the high ash content (especially silica) of rice straw fibers.

Effect of esterification of lignocellulosic fibers on properties of lignocellulosic–PE composites

The properties of composites depend on adhesion between the reinforced fibers and the matrix. Chemical treatment of fibers or using of the coupling agent has been studied to improve the adhesion between the fibers and the matrix, and consequently the mechani-

cal properties are improved. For example, alkali treatment, using 5% NaOH, of coconut fibers improved MOR, MOE, and tensile strength of the polyester/fiber composites.[7] Surface modification of sisal fibers with polystyrene, benzoyl peroxide, and poly(vinyl alcohol) was carried out with the aim of improving the properties of the sisal–polyester composites. A silane coupling agent was also used.[14] Jute fibers were coated with unsaturated polyester and vinyl ester resins, as well as isopropyl triisostearoyl titanate, aminotrimethoxy silane, sebacoyl chloride, and tolylene diisocyanate.[22] Surface treatment of Hessian cloth with maleic anhydride (2%), isocyanate-capped polyol (5%–35%), urethane prepolymer (5%–35%), and poly(vinyl acetate) (10%–20%) was employed to improve the mechanical properties of the Hessian/polyester composites.[23,24] In a previous study[25] it was found that esterification of old newsprint (ONP) fibers by MA improves the MOR, MOE, and dimensional stability of ONP–PE at a fiber ester content about greater or equal to 18%. The improvement of the mechanical properties was attributed to the possible crosslinking of the esterified fibers to the polymer matrix.

In this study the different lignocellulosic fibers were esterified using maleic anhydride. It was found that WPG due to esterification at the optimum conditions was relatively low compared with esterification of ONP fibers.[25] WPGs of 12.6, 14.3, and 15.2 were obtained for rice straw, bagasse, and cotton stalks, respectively. This was expected because ONP fibers are mainly cellulose and hemicelloses, whereas bagasse, cotton stalks, and rice straw fibers also contain lignin, ash, and extractives, which have a lower reactivity toward the maleic anhydride compared with the reactivity of cellulose or hemicelluloses. Figure 5 shows the IR spectra of the esterified fibers of bagasse, rice
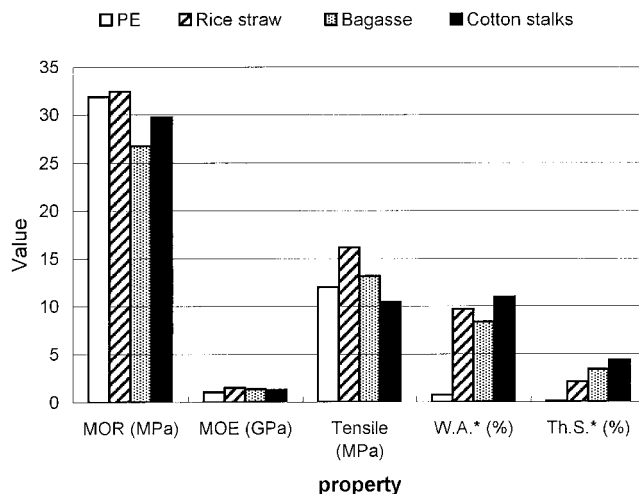

Figure 4 Optimum strength properties of the different lignocellulosic–PE composites and their water absorption and thickness swelling.
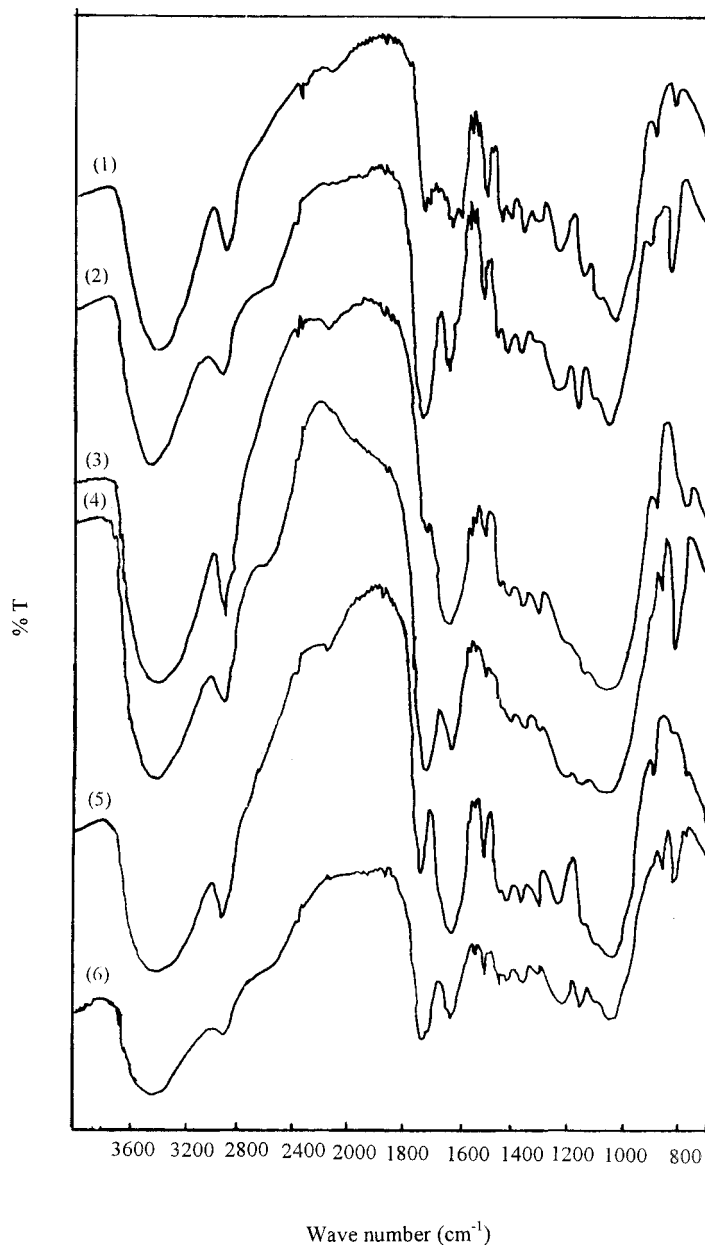
Figure 5 FTIR spctra of the lignocellulosic fibers and the maleic anhydride esterified derivatives: (1) bagasse; (2) esterified bagasse; (3) rice straw; (4) esterified rice straw; (5) cotton stalks; and (6) esterified cotton stalks.

straw, and cotton stalks. The intensity of the absorption peak at 1725 cm^{-1} was significantly increased due to the carbonyl group of the introduced carboxylic groups. The IR spectra of raw cotton stalks show a carbonyl absorption peak at 1725 cm^{-1}. This may be because of the presence of the bark, which contains waxes, resins, starches, and a high percentage of tannic acid. The intensity of this peak increased as a result of esterification.

Table II shows the properties of the esterified lignocellulosic fibers–PE composites containing 25% of the esterified fibers. As shown in the table, there was no improvement in MOR, MOE, and tensile strength of the composites of the different esterified fibers. A

slight decrease in MOR and MOE took place. This implies that the obtained esterification levels were not sufficient to cause a significant increase in the strength properties, as seen with ONP. It should be taken into consideration that the number of ester groups introduced into the fibers substitutes for the fibers in the composites. Thus, although lower strength properties were expected when using esterified fibers, the increase of the fiber–matrix interaction could compensate for the lower fiber content and, at sufficiently high ester content, could result in higher strength properties, as seen with the ONP–PE composites.[25] Water absorption increased, but thickness swelling decreased because of the introduction of the ester groups

TABLE II
Effect of Esterification of Lignocellulosic Fibers on Properties of the Produced Lignocellulosic Fibers–PE Composites

Type of fiber*	MOR (MPa)	MOE (GPa)	Tensile strength (MPa)	Water absorption (%)	Thickness swelling (%)
Rice straw	31.72 (3.16)	1.25 (0.07)	12.16 (1.92)	9.96	1.85
Esterified rice straw	29.14 (2.24)	1.23 (0.03)	12.48 (1.68)	12.26	1.01
Bagasse	26.76 (2.85)	1.35 (0.06)	13.14 (1.83)	8.34	3.42
Esterified bagasse	24.60 (2.18)	1.28 (0.05)	12.19 (1.37)	10.16	1.53
Cotton stalks	29.73 (3.59)	1.25 (0.04)	10.42 (1.87)	12.52	4.34
Esterified cotton stalks	25.30 (3.76)	1.16 (0.05)	9.44 (1.42)	15.21	2.25

Value in parentheses is the standard deviation.
* 25% (w/w) of fibers were used in the composites; fibers were milled using a 0.35-cm screen for rice straw and a 0.2-cm screen for the bagasse and cotton stalks.

into the cell wall polymers. This may be because esterification prevents any further swelling due to water absorption.

UV resistance of lignocellulosic–PE composites

Lignocellulosic materials exposed to outdoors undergo photochemical degradation caused by UV light. This degradation takes place primarily in the lignin component. The lignin acts as an adhesive holding the cellulose fibers together. Cellulose is much less susceptible than lignin to UV degradation. When rained on, the degraded fibers are washed off, and new lignin is exposed to degradation.[26] On the other hand, crosslinked polyester also undergoes photooxidation because of the presence of peroxide residue and the polystyrene chains, which are easily photolysed. Photooxidation of crosslinked polyester proceeds largely as a surface reaction because the polyester structure absorbs UV light. As a result, a thin layer of highly UV-absorbing groups is formed that protects the bulk

polymer in an auto-stabilizing process.[27] Table III shows the effect of UV light on the MOR, MOE, and tensile strength of lignocellulosic composites made from different lignocellulosic nonesterified and esterified fibers. As shown in Table III, all composites containing esterified fibers were affected to a higher extent by the UV light than composites that contain nonesterified fibers, and a significant loss of MOR and MOE occurred. This may be because of the deesterification and liberation of maleic acid, which may degrade the fiber constituents. A previous study on UV resistance of acetylated wood showed loss of 50% of the acetyl content of acetylated wood after 800 h of an accelerated weathering test.[26] The cotton stalk–PE composites showed the highest resistance toward UV light. This may be because of the presence of the bark in the cotton stalks used. The bark contains a high percentage of UV-absorbing groups. On the other hand, the MOR and MOE of the neat PE composite considerably increased because of exposure to UV. This may have resulted from the further crosslinking

TABLE III
Effect of Ultraviolet Light on Mechanical Properties of the Produced Lignocellulosic Fibers–PE composites*

	MOR (MPa)			MOE (GPa)			Tensile (MPa)		
	Befor UV	After UV	% change	Befor UV	After UV	% change	Befor UV	After UV	% change
100% PE	31.86 (2.80)	35.59 (2.16)	+ 11.7	1.02 (0.03)	1.18 (0.03)	+ 12.4	11.97 (0.81)	12.37 (1.13)	+ 3.4
Rice straw–PE	31.72 (3.16)	27.26 (2.74)	− 14.1	1.25 (0.07)	1.05 (0.04)	− 15.6	12.16 (1.92)	10.6 (1.66)	−15.1
Esterified rice straw–PE	29.14 (2.24)	20.05 (1.84)	− 31.2	1.23 (0.03)	0.88 (0.03)	− 28.4	12.48 (1.68)	10.34 (1.19)	−16.4
Bagasse–PE	26.76 (2.85)	22.79 (2.08)	− 14.8	1.35 (0.06)	1.06 (0.03)	− 21.3	13.14 (1.83)	11.63 (1.77)	−11.5
Esterified bagasse–PE	24.60 (2.18)	20.46 (2.40)	− 16.8	1.28 (0.05)	0.92 (0.07)	− 28.4	12.19 (1.37)	10.07 (1.43)	−17.4
Cotton stalks–PE	29.73 (3.59)	26.69 (2.16)	− 10.2	1.25 (0.04)	1.16 (0.03)	− 6.8	10.42 (1.87)	9.62 (1.08)	− 7.7
Esterified cotton stalks–PE	25.30 (3.76)	22.15 (2.47)	− 12.4	1.16 (0.05)	0.91 (0.06)	− 21.4	9.44 (1.42)	7.42 (0.68)	−22.5

Value in parentheses is the standard deviation.
* Temperature and humidity were set at 30°C and 50%, respectively; time of the test was 50 hs. 25% (w/w) of fibers were used in the composites; fibers were milled using a 0.35-mm mesh screen for rice straw and a 0.2-mm mesh screen for bagasse and cottons stalks.
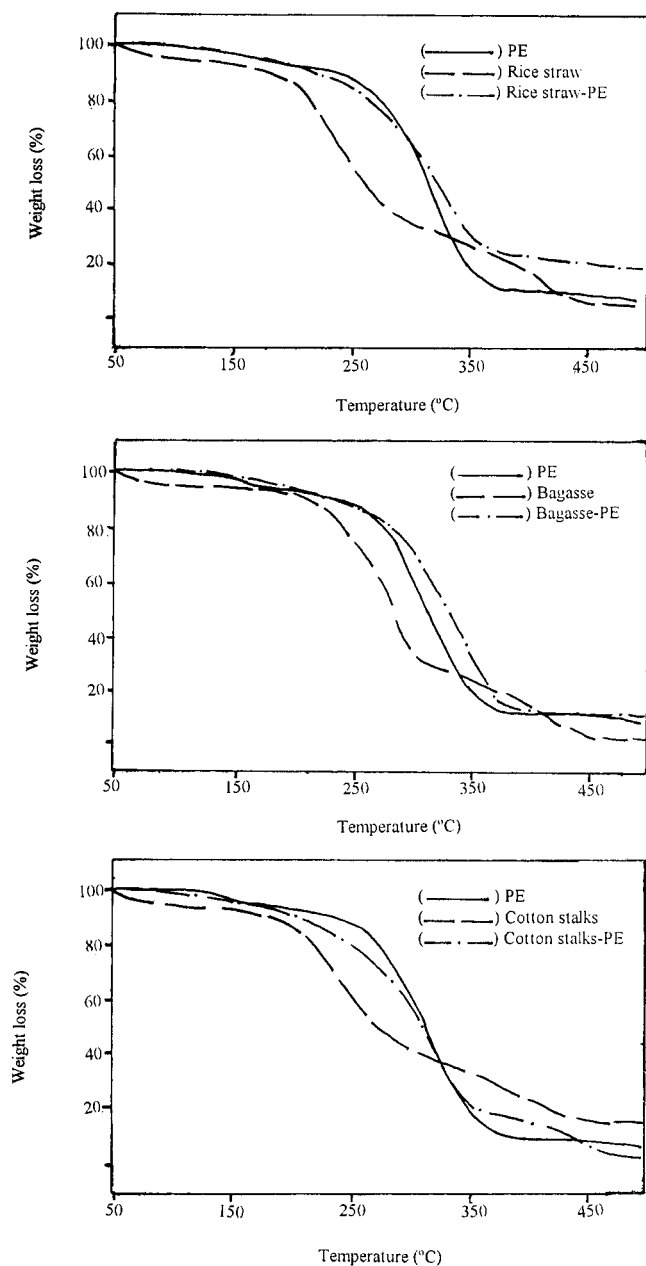
Figure 6 TGA curves of the lignocellulosic fibers and lignocellulosic–PE composites.

of the polyester with any residual styrene monomers by the UV light. Photodegradation of the of polyester is possible, as mentioned above, but because the exposure time to UV was not that long, the effect of postcuring of the polyester predominated.

Thermogravimetric analysis (TGA) of lignocellulosic–PE composites

The thermal behavior of the different lignocellulosic–PE composites was studied in the temperature range of 50°C–500°C using TGA. Figure 6 shows the TGA curves of neat PE, lignocellulosic fibers, and the different lignocellulosic fibers–PE composites. All composites contained 25% of the fibers that milled using a 0.2-cm screen. Table IV contains the data of the TGA curves. All lignocellulosic fibers showed three stages of weight loss. The first stage was due to water evaporation and continued up to about 120°C. The second stage started at about 200°C–208°C, this stage resulted from the generation of noncombustible gases such as CO_2 and formic and acetic acids. The third stage began at about 295°C–315°C. This occurred because of the pyrolysis and generation of combustible gases.[28] The PE showed two stages for weight loss. The first stage occurred because of water evaporation and continued up to about 120°C; the second one began at about 235°C. All lignocellulosic fibers–PE composites have two weight-loss stages instead of three, as is the case for the lignocellulosic fibers. As shown in Table IV, the neat PE composite had a significantly higher onset weight loss and maximum weight loss temperatures than did the lignocellulosic fibers; mixing the fibers with PE resulted in composites having a slightly lower onset degradation temperature than the neat PE. The different composites had maximum weight-loss temperatures close to that of the neat PE, except for the RS–PE composite, for which a higher maximum weight loss was found. All lignocellulosic–PE composites had onset weight-loss temperatures that were close to each other except for the CS-PE composite, which had a slightly lower onset weight loss temperature.

TABLE IV
TGA Data of the Different Lignocellulosic Fiber and Lignocellulosic Fibers–PE Composites

	Onset weight loss temperature (°C)	Maximum weight loss temperature (°C) of the 2nd stage*	Maximum weight loss temperature (°C) of the 3rd stage*
PE	235	320	
Bagasse	207	272	404
Rice straw	200	243	411
Cotton stalks	200	246	386
Bagasse–PE composite	230	318	
Rice straw–PE composite	230	332	
Cotton stalk–PE composite	212	321	

* Maximum weight-loss temperature was obtained from the differential TGA curves.

CONCLUSIONS

Studying bagasse, rice straw, and cotton stalks as reinforcing fillers in PE composites showed that rice straw fibers were found to be the best fibers in producing composites that had the highest strength properties and dimensional stability. The MOR, MOE, and tensile strength of a selected RS–PE composite were higher than those of the neat PE composite. Using any of the previously mentioned fibers may result in increasing MOE of the lignocellulosic fiber–PE composites compared with the neat PE composite. Esterification of the different lignocellulosic fibers under the conditions used in this work was not efficient in improving the strength properties of the different lignocellulosic fiber–PE composites because of the relatively low extent of esterification (WPG) obtained. The cotton stalk–PE composite showed the highest resistance to UV light. This is probable because of the bark, which contains a high percentage of UV-absorbing groups, in the cotton stalks used. All lignocellulosic fiber–PE composites were slightly thermally less stable than the neat PE composite.

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