

Research Article

Influence of Incorporation of Natural Fibers on the Physical, Mechanical, and Thermal Properties of Composites LDPE-Al Reinforced with Fique Fibers

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This study shows the effect of the incorporation of natural fique fibers in a matrix formed by low-density polyethylene and aluminum (LDPE-Al) obtained in the recycling process of long-life Tetra Pak packaging. The reinforcement content was 10, 20, and 30% fibers, manufactured by hot-press compression molding of composite boards (LDPE-Al/fique). From the thermogravimetric analysis (TGA) it was determined that the proportions of the LDPE-Al were 75 : 25 w/w. Likewise, it was found that the aluminum particles increased the rigidity of the LDPE-Al, reducing the impact strength compared to LDPE recycled from Tetra Pak without aluminum; besides this, the crystallinity in the LDPE-Al increased with the presence of aluminum, which was observed by differential scanning calorimetry (DSC). The maximum strength and Young's modulus to tensile and flexural properties increased with the incorporation of the fibers, this increase being a direct function of the amount of reinforcement contained in the material. Finally, a reduction in the density of the compound by the generation of voids at the interface between the LDPE-Al and fique fibers was identified, and there was also a greater water absorption due to weak interphase fiber-matrix and the hydrophilic fibers contained in the material.

1. Introduction

In industry, composite materials have attracted great interest due to their good specific properties. These are evaluated according to the materials' mechanical performance, which depends on the characteristics of each of their components, the arrangement of the reinforcement in the matrix, and interfacial quality. In recent years, interest has grown worldwide in the incorporation of cellulosic fibers such as sisal, fique, jute, henequen, and wood pulp, among others, in composites, because present studies show that for their good mechanical properties the fibers can act as reinforcing polymeric matrices [1–6].

Among natural fibers, it can be highlighted that fique has an average density of 1.47 g/cm³, a diameter (equivalent to the area) of around 0.24 μm, an ultimate strength of approximately 132.40 MPa, and Young's modulus between 8.20 and 9.10 GPa. This fiber is produced in different departments of

Colombia, mainly Cauca, Nariño, Santander, and Antioquia, which produce 98% of the fiber in the country [7–11].

Moreover, the trend in the state of the art tends to point toward polymeric matrices coming from renewable sources or recycling processes involving high turnover products. Amongst the latter class of materials are mixtures of low-density polyethylene and aluminum (LDPE-Al) obtained from the recycling of long-life Tetra Pak packaging, which are composed of 5% aluminum, 20% polyethylene, and 75% paperboard. Neves in 1999 reported the basic process to the industrial recycling of the Tetra Pak packaging, mainly based on the separation of the paperboard layer of the aluminum and polyethylene layers by hydropulping process for 35 min, where cellulosic fiber was obtained suitable for use in different papermaking process, while the LDPE-Al's residue could be used in power generation (through incineration) or in the manufacture of plastics elements, such as chipboards; also, it is possible to recover the aluminum by pyrolysis [9, 12].

Mourad et al., 2008 [13], analyzed the influence of recycling of the Tetra Pak packaging on the reduction of Global Warming Potential (GWP); they determined that an increase in recycling from 2% (2000) to 22% (2004) reduces the GWP in more than 14%, and a 48% could be reached if the recycling of Tetra Pak packaging increases to 70%.

Due to the global interest, particularly by the Tetra Pak company, in the recovery and reuse of LDPE-Al, in this work we studied the influence of incorporating fique fibers in a matrix based on a binary mixture of LDPE and aluminum (LDPE-Al) on some physical, thermal, and mechanical properties of composite LDPE-Al/fique. The material is manufactured by the hot-plate press molding process with fique contents of 10, 20, and 30% by volume; these values of contents were determined from previous works, where it was observed that using the same process implemented in this work it is difficult to achieve a good distribution of the fibers in the composite and complete wetting of the fibers with the matrix when content of fiber is above 30% [9]. The characterization focused primarily on the tensile, flexural, and impact mechanical performance of the material. Also, we evaluated the influence of the fibers on the heat stability, crystallinity, density, and water absorption of the material.

2. Materials

The LDPE-Al without cellulose used in this research as composite matrix, supplied by INNOPACK LTDA, was processed in order to reduce its particle size. The material has a LDPE/aluminum ratio of 89.75/10.25 v/v by volume and a density of 1.10 g/cm^3 . Fique fibers belonged to the (white) eagle claw variety of the *Furcraea* genus and were supplied by Compañia de Empaques de Medellin; these fibers were used without any treatment and were arranged in the compound as a two-dimensional mat with a random arrangement, as shown in Figure 1. The fique employed had an average length of $51.70 \pm 33 \text{ mm}$, maximum tensile strength of 263.65 MPa, average Young's modulus of $8.64 \pm 0.71 \text{ GPa}$ (measured with fiber lengths of 50 mm), and true density of approximately 1.102 g/cm^3 [11, 14].

3. Experimental Procedures

3.1. Preparation of Composite and Specimens. The composite material formed with the binary mixture LDPE-Al as a matrix and fique as reinforcing fibers (LDPE-Al/fique) was obtained as rectangular boards with dimensions of $290 \times 290 \times 2 \text{ mm}$, using for this purpose one hot-plate press and a forced water circulation cooling system (LabPro 400, Fontijne Presses). To shape the specimens, stainless steel close-up type molds were used, where layers of LDPE-Al and fique fiber mats (to reach the desired percentages of fibers of 10, 20, and 30% fiber volume) were alternately placed. The molding was conducted at a temperature of 170°C and a pressure of 3 MPa; the complete process was 15 min, comprising 11 min of heating and 4 min of cooling under pressure (Figure 2). Finally the boards were demolded and adjusted to the required dimensions in various

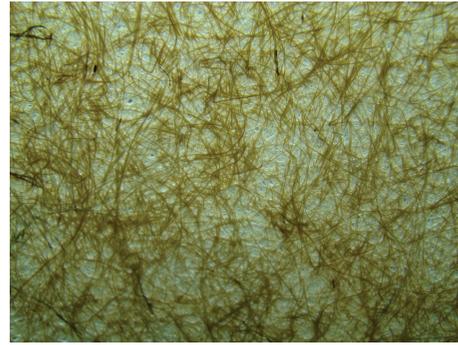


FIGURE 1: Dimensional randomization of fique mat used in the reinforcement of the LDPE-Al.

mechanical (tensile, flexural, and impact), physical (density, water absorption), and thermal (thermogravimetry and differential scanning calorimetry, DSC) tests using a numerical control router.

3.2. Mechanical Tests. Tensile mechanical properties were determined on samples of LDPE-Al matrix and the three LDPE-Al/fique composites with 10, 20, and 30% reinforcement. In all cases this characterization was performed after 48 hours of conditioning at a temperature of 25°C and relative humidity of 50%. The tests were conducted on an Instron 3366 mechanical universal testing machine fitted with a 10 kN load cell. Type I specimens were used and tested with a speed of displacement between clamps of 5 mm/min following the ASTM standard D638. All measurements were carried out on five test specimens and the analysis took into account the average of these ratings.

Through the flexural test, the mechanical flexural properties of LDPE-Al with and without reinforcement were measured. The materials were subjected to 48 hours of conditioning at a relative humidity of 50% and a temperature of 25°C . The tests were carried out on an Instron 3366 universal mechanical testing machine equipped with a 10 kN load cell. The specimens used had dimensions of $12.70 \text{ mm} \times 100 \text{ mm} \times \text{thickness}$ (thickness varied depending on the compound) and were evaluated at a speed within the range of 0.79–1.01 mm/min (the rate was determined based on the dimensions of the specimen). The distance between the supports was 16 times the thickness of the specimen and the test was conducted up to 5% strain, following the ASTM D790 standard. All measurements were carried out on five test specimens and the analysis took into account the average of these ratings.

The impact strength of LDPE-Al and LDPE-Al/fique for the three compounds was determined. Prior to the test, the materials were subjected to conditioning for 48 hours at 50% relative humidity and a temperature of 25°C . Trials were performed on an Izod Tinius Olsen impact pendulum equipped with a 4.53 N pendulum. The specimens were made following the standard ASTM D256, and the starting angle of the test was 55.80° . All measurements were carried out on five test specimens and the analysis took into account the average of these ratings.

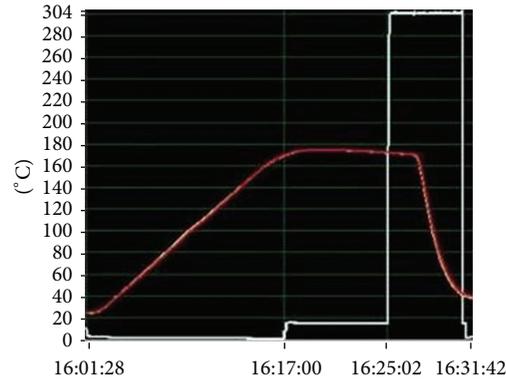


FIGURE 2: Manufacturing process of the boards produced from the LDPE-Al/fique compound.

3.3. Physical and Thermal Tests. The determination of the LDPE-Al and the three different compounds' (LDPE-Al/fique) density was performed according to the Archimedes method specified in ASTM D792. Due to the hydrophilic character of the fibers present in fique, the conventional density test consisting of immersion in distilled water was discarded; canola oil was used as the immersion liquid, which has a density of 0.911 g/cm^3 .

The water absorption test determined the ability of the LDPE-Al and the three compounds (LDPE-Al/fique) to absorb water at a set time. First, the materials were conditioned in an oven for 24 h at 50°C ; after this time had expired, conditioned specimens were weighed (conditioned weight) before being immersed in distilled water at a temperature of 25°C for 24 h. Finally, the water was removed, the specimens were surface-dried and weighed (wet weight), and the determination of the percentage of water absorbed was performed according to the model specified in the following:

$$\begin{aligned} &\text{Weight increment, \%} \\ &= \frac{\text{Wet Weight} - \text{Conditioned Weight}}{\text{Conditioned Weight}} * 100. \end{aligned} \quad (1)$$

The thermogravimetric analysis test (TGA) was carried out on TA Instruments TGA Q500 with a protective nitrogen atmosphere; the test was run at a heating rate of $10^\circ\text{C}/\text{min}$ in a temperature range between 25 and 550°C . The average weight of the samples tested was 6 mg.

Differential Scanning Calorimetry (DSC). The percentage crystallinity and the melting temperature of the LDPE-Al matrix, as well as the influence of the incorporation of fibers in the LDPE-Al/fique composite, were determined using a Q2000 DSC equipment from TA Instruments under a protective atmosphere of nitrogen, with a heating and cooling speed of $10^\circ\text{C}/\text{min}$. Tests were carried out in three steps: (1) first heating: -90°C to 150°C with an isotherm at 150°C for 4 min; (2) cooling: 150°C to -90°C with an isotherm at -90°C for 3 min; (3) second heating: -90°C to 150°C . The percentage

crystallinity was estimated using the model presented in the following:

$$X = \frac{\Delta H_f}{W_{pe} \Delta H_f^0}, \quad (2)$$

where X is the percentage crystallinity, ΔH_f is the compound's fusion enthalpy, ΔH_f^0 is the 100% crystalline polyethylene's fusion enthalpy (288.83 J/gr), and W_{pe} is the weight fraction of the polyethylene phase in the compound [15, 16].

4. Results and Discussion

4.1. Tensile Test. Figure 3 shows the maximum strength, Young's modulus, and maximum strain values for each material tested. Here it can be demonstrated that increasing the fiber content in the compound led to increases in the maximum strength and Young's modulus and a decrease in the strain of the material, revealing that the material undergoes a phenomenon of stiffening due to the inclusion of natural fibers. This behavior might be generated by the high aspect ratio of the embedded fibers (average of 247), which causes the fique properties to prevail over those of LDPE-Al. Accordingly, it is worth noting that Hidalgo et al. [9], in a similar work about a polyethylene-aluminum matrix reinforced with short fiber of fique, suggested the possible presence of mechanical anchors between the fibers and the matrix, which would restrict the strain capacity of LDPE-Al. Moreover, if one considers that the fibers of fique mats, which are used in this work, have a random distribution and could be tangled with each other, the possible mechanical anchorage between the fibers and the matrix could be even more important.

The best tensile mechanical performance was obtained by the 30% fique fiber compound, resulting in a maximum strength and Young's modulus of 21.22 MPa and 1.56 GPa, respectively. This implied increases of 97.80% in the maximum strength and 180.78% in Young's modulus relative to the material without reinforcement (LDPE-Al). It is important to remark that the maximum tensile strength was higher than that reported by Hidalgo et al. [9], who achieved a maximum tensile strength of 15.56 MPa for a composite of LDPE-Al matrix reinforced with 30% short fique fibers. However, the

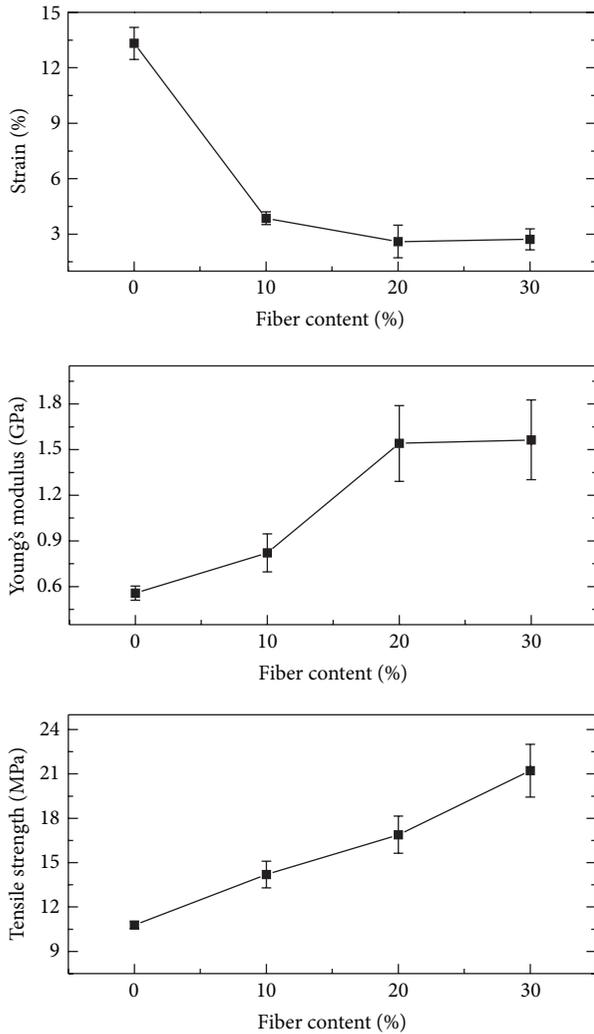


FIGURE 3: Tensile properties of the materials tested.

strain of this compound showed a decrease of 389.41% compared to the LDPE-Al.

4.2. Flexural Test. The bending behavior of the materials studied is shown in Figure 4. Here it can be observed that the properties increased when the incorporation of fique fibers increased in the LDPE-Al matrix, corroborating the stiffness that was previously evidenced in the tensile tests.

Just as in the tensile tests, the best performance was observed in the material reinforced with 30% fique fibers, which reached values of 26.97 MPa to maximum strength and 1.37 GPa to Young's modulus, improving the properties of LDPE-Al matrix by 117.81 and 204.28%, respectively.

4.3. Impact Test. Figure 5 shows an impact strength of 10.47 kJ/m² (103.43 J/m) for the LDPE-Al; this value is less than that reported by Desiderá [17] for a LDPE recycled with Tetra Pak without aluminum (315 J/m). This made it clear that the 10.50% aluminum particulate content in the polyethylene used as a matrix in this investigation weakens the material,

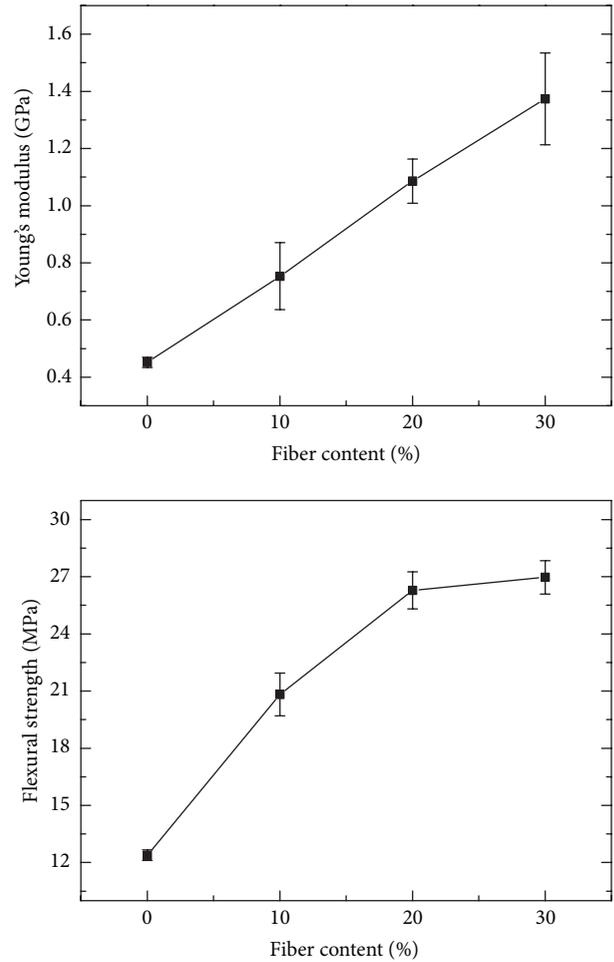


FIGURE 4: Flexural properties of the materials evaluated.

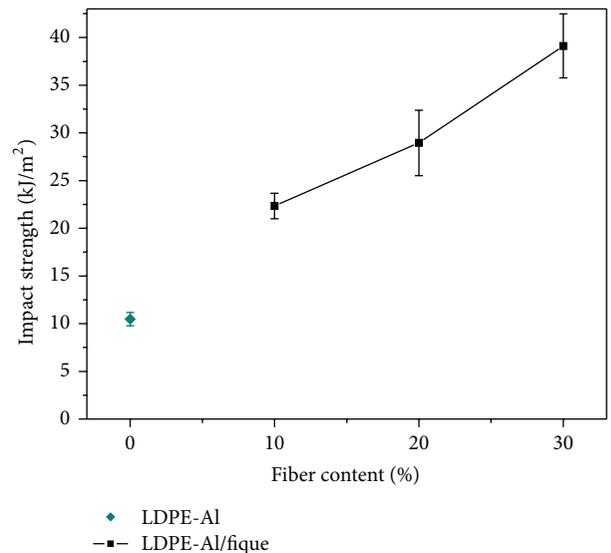


FIGURE 5: Impact strength of the composite depending on the fiber content.

generating a decrease in the ability of LDPE-Al to absorb energy; some authors that incorporated metal particles in

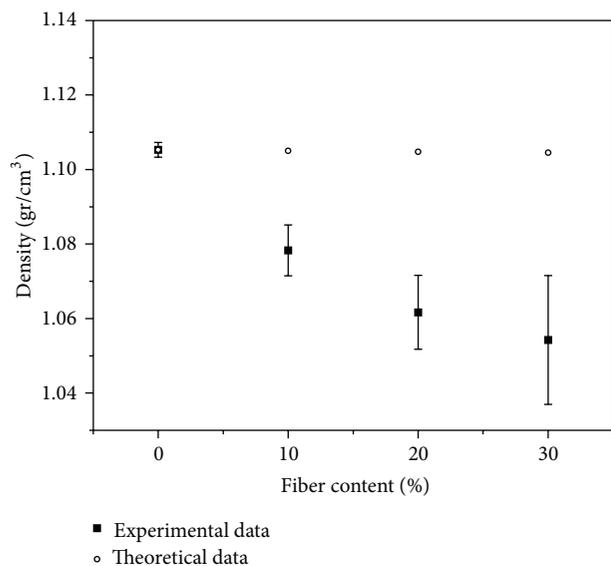


FIGURE 6: Density of LDPE-Al and 10, 20, and 30% fique fiber compounds.

polymeric matrices observed the same behaviour on impact strength and attributed it to weak interfacial adhesion in the composite, which generates that metallic particles behave as stress concentration points, facilitating the propagation of cracks in the material [17–19].

Moreover, it was found that, by incorporating fique fibers in the matrix, the material's impact strength increased. This phenomenon could be due to a possible energy absorption promoted by fracture mechanisms, which involve detachment, slippage, and fragmentation of the fiber and are not part of the unreinforced material fracture [20].

4.4. Density Test. Figure 6 shows the results obtained from the density tests of LDPE-Al and materials reinforced with 10, 20, and 30% fique fiber, and these results were compared with the density values estimated by the rule of mixtures.

By incorporating fique fibers, it was found that composite density decreased as the amount of fiber in the material increased. This occurred even though it was expected that the density of the compound would not undergo large variations with respect to the density of the LDPE-Al due to the similarity between the densities of LDPE-Al (1.100 g/cm^3) and fique (1.102 g/cm^3). This effect of the content of fique fibers on the composite density may be caused by the formation of a weak interface between the phases of the compound, which would affect the generation of voids and/or cavities between the fiber and the matrix; in Figure 7 a SEM micrograph can be observed, where the cavities present in the different materials evaluated are shown; hence the greater the amount of fibers in the material, the greater the difference between the experimental values and the value estimated by the rule of mixtures, as shown in the graphs presented in Figure 6. It is important to say that the standard deviations for the experimental values are higher when increasing the fique fiber content in the

composite; this is associated with an increase in the rate of the weak fiber-matrix interface.

4.5. Water Absorption Test. Figure 8 shows the response of the materials evaluated to the water absorption after being immersed in distilled water for 24 h at 23°C . The LDPE-Al without reinforcement had a water absorption percentage of 0.05%, which increased with the fiber content incorporated. This is attributed to the possible water absorption by diffusion and percolation mechanisms; the latter phenomenon is generated by microspaces present between the fiber and the matrix, as seen in Figure 7, which allow the filtration of water on the material and it becomes more important when the fiber content on the matrix is increased. Another reason for the observed trend is the hydrophilic character of the cellulosic fibers, especially promoted by the hemicellulose content, which is considered as the fiber component with the highest water absorption capacity, because it has a disordered intermolecular structure which prevents its hydroxyl groups which can establish hydrogen bonds with each other, thus allowing the formation of bonds with water molecules [21–23].

4.6. Thermogravimetric Analysis (TGA). TGA was performed on both the LDPE-Al and the material reinforced with 30% by volume of fique. Figure 9 shows the thermograms obtained in the materials evaluated. Here we can see an initial mass loss at a temperature close to 65°C in the reinforcement material, highlighting that this fall was not important in the LDPE-Al matrix. According to Monteiro et al. [24], the mass loss could be attributed to the loss of water in fique fibers (due to their hydrophilic state) contained in the composite. This reduction in mass was about 2.61%.

Likewise, the thermogram of LDPE-Al (Figure 9) demonstrates that the onset of degradation of the LDPE occurred from 350°C and a faster loss of mass of the polyethylene was present around 470°C , as the peak of the derivative curve DTG shows. These values were similar to those reported by Tajeddin et al. [25], who worked with a LDPE/kenaf composite. Moreover, one can see that the degradation of LDPE-Al reached a mass loss of around 75%, which is only associated with the polyethylene phase because the aluminum phase does not suffer any loss of mass by volatilization at a temperature of 500°C [26], implying that the remaining 25% is aluminum. Given that the LDPE (with which the Tetra Pak packaging is manufactured) has a density of 0.918 g/cm^3 [26] and that the aluminum has a density of 2.70 g/cm^3 , it was possible to confirm that the ratio 75/25 w/w that was determinate in the test is equivalent to the 89.75/10.25 v/v for LDPE/Al, previously established in Section 2.

Regarding the material reinforced with 30% fique fibers, it can be observed in thermogram (Figure 9) that before the LDPE thermal degradation occurred, thermal events associated with fique fibers occurred, which resulted in a loss of mass around 305°C , which has been attributed by some researchers [14, 24] to the degradation of hemicellulose. Furthermore, another important mass loss can be observed in the range of 325 to 400°C , corresponding to the thermal

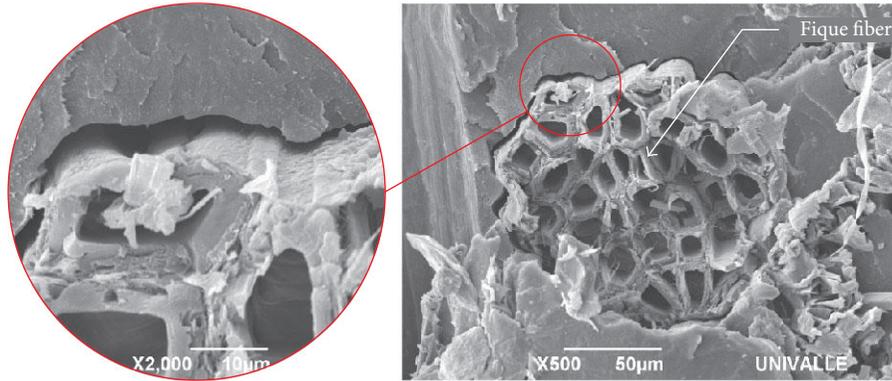


FIGURE 7: SEM micrograph. Cavities present in the fiber-matrix interface of the LDPE-Al/fique compound.

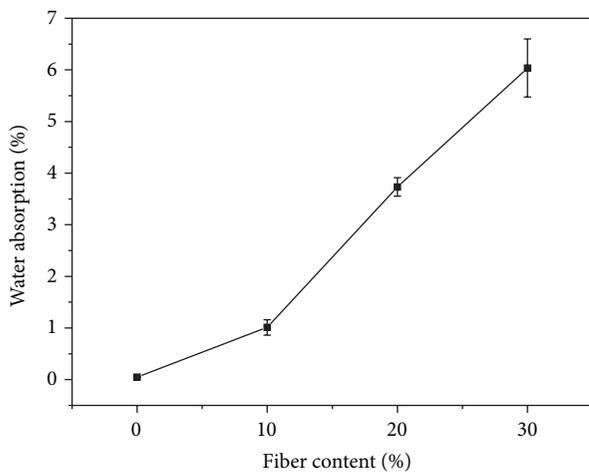


FIGURE 8: Water absorption by immersion of composite materials reinforced with fique fibers at 23°C.

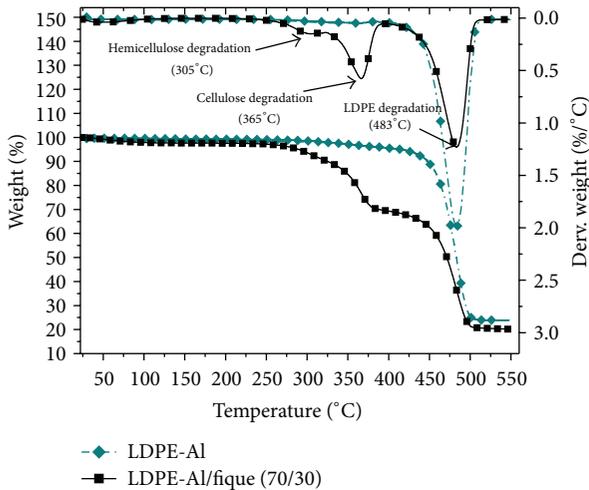


FIGURE 9: TGA thermogram for LDPE-Al compound and 30% fique fibers compound.

degradation of cellulose and thus the decomposition of the fiber. Moreover, the peak of the derivative associated with

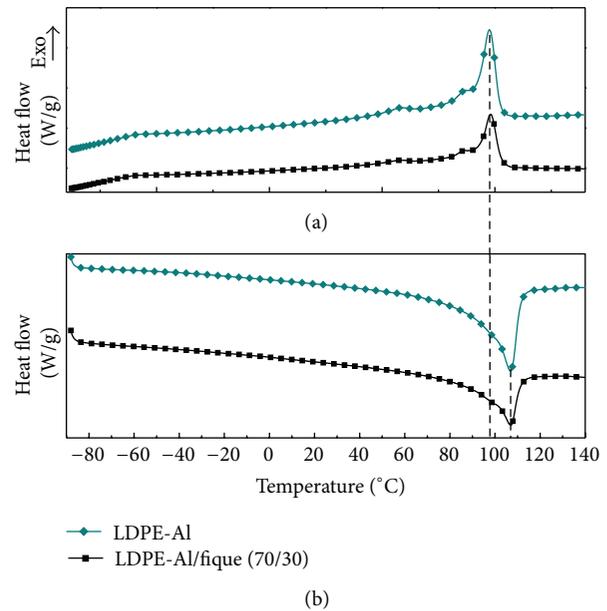


FIGURE 10: Heat flow versus temperature for LDPE-Al and 30% fique fiber compound: (a) cooling and (b) second heating.

the decomposition of LDPE shows a shift to about 483°C in the reinforcing material. The increase in the thermal stability of the compound has been previously reported in different studies [25, 27, 28], indicating that the incorporation of fibers in the material induces spherulite nucleation points, increasing the crystallinity of the polymer and, therefore, improving its thermal properties.

4.7. Differential Scanning Calorimetry (DSC). Just as with the TGA, the materials evaluated with this technique were 30% fique and LDPE-Al without reinforcement. Figure 10 shows the curves of heat flow as a function of temperature (cooling and second heating) for the materials tested. Here it can be observed that in the heating curves (Figure 10(b)) there is an apparent decrease in the enthalpy of fusion from 90.01 to 63.22 J/g with increasing fraction of the material components (fique fibers and aluminum), which do not present melt at the

TABLE 1: Degree of crystallinity (X) estimated for the polyethylene phase for each of the materials tested.

| Material | Cooling | | | Second heating | | | $X_{\text{Phase LDPE}}$ |
|----------|-------------|--------------------|--------------|----------------|--------|--------------|-------------------------|
| | Onset point | T_c | ΔH_c | Onset point | T_f | ΔH_f | |
| | | $^{\circ}\text{C}$ | | | Peak | | |
| LDPE-Al | 102.36 | 97.58 | 84.61 | 94.88 | 106.91 | 90.01 | 44.53 |
| 30 | 102.98 | 98.21 | 61.54 | 94.95 | 106.88 | 63.22 | 44.70 |

conditions of the test and are not miscible with the polyethylene phase; however, although the melting enthalpy of LDPE-Al/fique appears to be smaller than that shown by the LDPE-Al and the LDPE (98–115 J/g) [16, 17], when the melting enthalpy is corrected taking into account only the content of LDPE, the crystallinity of the polyethylene phase does not decrease (Table 1). This behavior was also observed by Aht-Ong and Charoenkongthum [29] in a biocomposite of LDPE/banana starch.

Table 1 shows the degree of crystallinity (X) estimated for the polyethylene phase for each of the materials tested. It was found that the crystallinity of the polyethylene phase within the LDPE-Al was 44.53%, which was greater than the value of 34% reported in various studies [16, 30] for low-density polyethylene, which was also determined by DSC tests. Desiderá [17], who studied a mixture of polyamide and polyethylene recycled from multilayer recycled packaging, attributed the increase in crystallinity to the effect of the presence of aluminum particles and/or impurities present in the recycled material, which act as nucleation points promoting the formation of spherulites. Similarly, when comparing the degree of crystallinity obtained in the phase of the LDPE-Al to that achieved in the fique-fiber-reinforced material, no significant difference can be seen. This crystalline phase content was maintained despite the decreasing fraction of aluminum in the composite owing to the incorporation of the fibrous material. The above shows that the fique fibers also promote the formation of crystalline phases in the polyethylene present in the matrix of the composite. In some studies [14, 21, 31] of fiber-reinforced related compounds, it has also been found that fibers act effectively as nucleation sites for the formation of spherulites.

Furthermore, the melting of the materials presented a well-defined peak whose base was found around 107 $^{\circ}\text{C}$. It is important to note that in some studies [25, 32, 33] melting temperatures of 102 to 105 $^{\circ}\text{C}$ are reported for virgin and recycled LDPE, which could suggest further evidence of increased crystallinity in the polyethylene phase, since more energy is required to reach the melting point of the material, particularly regarding the fusion of the crystalline fraction.

5. Conclusions

In this work it was possible to develop a composite material comprising a matrix based on low-density polyethylene and aluminum (LDPE-Al) obtained from the recycling of post-consumer long-life Tetra Pak packaging and fique fibers as

reinforcement. Such material may be processed using a conventional processing method using hot compression molding.

With regard to the tensile and flexural properties, clear stiffening was observed with the addition of fique to LDPE-Al attributed to the good aspect ratio of the fibers used and their arrangement in the matrix. The impact strength of the material increased due to the generation of energy-absorbing mechanisms during fracture, such as detachment, slippage, and fiber fracture, which were absent in the material without reinforcement.

Thermal analysis techniques such as TGA and DSC, allowed determining that the aluminum particles (dispersed phase present in the LDPE-Al) and the incorporation of fique fiber on the composite induce nucleation points of spherulites which promote increased crystallinity on the polyethylene phase of the materials evaluated in 10.7% (with reference LDPE without fibers or aluminum), leading to improved thermal properties, as evidenced by the shifting of the decomposition temperature peak of the LDPE, which moved from 470 to 483 $^{\circ}\text{C}$ when fique fibers were incorporated.

Furthermore, the incorporation of fique fibers on the LDPE-Al caused a decrease in density and an increase of water absorption; this is due to the hydrophilic character of natural fibers that also generates the formation of a weak interface with the matrix, which influence the generation of voids and/or cavities in the material.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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