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Mechanical and thermal properties of biocomposites from nonwoven industrial Fique fiber mats with Epoxy Resin and Linear Low Density Polyethylene

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ABSTRACT

In this work Linear Low Density Polyethylene-nonwoven industrial Fique fiber mat (LLDPE-Fique) and Epoxy Resin-nonwoven industrial Fique fiber mat (EP-Fique) biocomposites were prepared using thermo-compression and resin film infusion processes. Neat polymeric matrices and its biocomposites were tested following ASTM standards in order to evaluate tensile and flexural mechanical properties. Also, thermal behavior of these materials has been studied by differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). Tensile and flexural test revealed that nonwoven Fique reinforced composites exhibited higher modulus and strength but lower deformation capability as compared with LLDPE and EP neat matrices. TG thermograms showed that nonwoven Fique fibers incorporation has an effect on the thermal stability of the composites. On the other hand, Fique fibers did not change the crystallization and melting processes of the LLDPE matrix but restricts the motion of EP macromolecules chains thus increases the Tg of the EP-Fique composite. Finally, this work opens the possibility of considering non-woven Fique fibers as a reinforcement material with a high potential for the manufacture of biocomposites for automotive applications. In addition to the processing test specimens, it was also possible to manufacture a part of LLDPE-Fique, and one part of EP-Fique.

Introduction

Nowadays polymer matrix composite materials reinforced with fibers such as glass or carbon are increasingly being used in sectors such as aeronautics, automotive, naval and sporting goods. This due mainly to the advantages they present in terms of specific mechanical strength, low weight and the possibility of making pieces of different sizes with custom designs [1,2].

However, they present specific problems, their high dependence on oil and the waste management after its end of life. As a result, the scientific community has focused on the development of composites materials reinforced with natural fibers such as jute, kenaf, Fique, flax or hemp [3–6].

Some characteristics that make natural fibers attractive are low cost, their high specific stiffness and impact resistance, reduced energy consumption, non-irritation to the skin, renewability, recyclability and biodegradability. They also provide thermal and acoustic insulation, structural lightness, which results in a reduction in weight and therefore, lower fuel consumption and lower emissions for specific applications. Consequently, during the last decade, polymer matrix composites reinforced with natural fibers, were increasingly being introduced in sectors such as automotive and furniture [3,5].

In the South American Andean region Fique is one of the most abundant fibers, which it uses in the manufacture of sacks, shoes, bags, and handicrafts. However this fiber can also be used as a structural reinforcement of polymeric matrix composites [7–11].

Thermoplastics polymers are the most commonly used matrix materials for natural fiber composites materials [4,6]. Indeed, Polyolefins such as Polypropylene (PP) and Polyethylene (PE) are the two most adopted thermoplastics for this purpose. Among these materials, Linear Low Density Polyethylene (LLDPE) is remarkable due its mechanical properties related to Low Density Polyethylene (LDPE). The physical properties of LLDPE make it a good candidate for several product applications as food packaging, grocery bags, agricultural films, pipes and rotomoulded pieces [12]. The most thermosets matrices used for natural fiber composites materials are unsaturated polyester (UP), epoxy resin (EP), phenol formaldehyde (PF) and vinil ester resins (VE) [4,13]. However, several research works regarding natural fiber composites have shown

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that the best mechanical performance is obtained by using EP as matrix [4,14].

During the last years the research group GITEM has been developing several studies focused on the production of materials and products based on natural fiber composites materials through different transformation processes such as thermocompression, injection moulding, resin film infusion, additive manufacturing, among others [9–11,15].

The purpose of this work is trying to study the influence of the incorporation of nonwoven industrial Fique fiber mats on the thermal and mechanical properties of two polymer matrices with a different thermal behavior (LLDPE and a flexible EP). This would allow appreciating the real possibilities of this natural fiber to be used as reinforcement for polymer matrix composites.

Experimental

Materials

The Low Density Polyethylene (LLDPE) "R50035" was purchased as pellets from SABIC (Kingdom of Saudi Arabia) with a melt flow index of 5 g/10 min (2.16 kg/190 °C) and a density of 0.93 g/cm^3. The flexibilized Epoxy Resin (EP) was purchased from the company Sintepox (Colombia) and consist in a general purpose and low modulus epoxy system based on a low viscosity epoxy resin type Bisphenol A reference "R3610" and a modified cycloaliphatic amine hardener reference "E-1610". This epoxy system was prepared with a mixing ratio 50:50 (%wt/wt). Nonwoven industrial Fique fiber mats (Fique) were supplied by "Packaging Company of Medellin" (Colombia). The Fique fiber mats (Fig. 1) presents an average length of 51.7 mm, a tensile strength of 263 MPa and an average tensile modulus of 8.6 GPa [10,11].

Prior to biocomposites preparation, LLDPE was pulverized in order to increase the adhesion of the Fique fibers and to produce a better distribution in the matrix. The average particle size of the LLDPE powder was 500 μm. Also, the nonwoven industrial Fique mats were dried in an oven at 60 °C for 24 h.

Composites preparation

Linear Low Density Polyethylene-non woven Fique Fiber biocomposite (LLDPE-Fique) was obtained in panels with dimensions of 180 mm × 180 mm × 2 mm. A close up-type stainless steel mould was used and panels were manufactured by compression moulding in a hot plates press Carver model 973214A. To prepare the biocomposite, nonwoven Fique mats were placed between Teflon films along with the LLDPE powder, ensuring a complete coverage of the mat filling the mould. The sample was compression moulded at 170 °C and 10 MPa for 8 min. Based on a mass balance, the fiber weight fraction of the composite was predicted to be around 20% (% wt/wt).

Fig. 1. Nonwoven industrial Fique Fiber mats.

Epoxy Resin-non woven Fique Fiber Fiber biocomposite (EP-Fique) was manufactured in a Teflon mould (300 mm × 300 mm). In a first step, the mould surface was coated with a thin layer of EP and then the nonwoven Fique mat was placed on the mould. After that, the Fique mat was filled with more EP and was left to be absorbed. Then, the mould was placed inside a flexible film in which it was left to cure under vaccum for 24 h at room temperature. The EP-Fique weight fraction was 80/20 (% wt/wt). When the composites sheets were ready, they were removed from the moulds and being cut into different tensile and flexural test specimens using a numerical control router. For comparison purposes, neat LLDPE and EP specimens were prepared using the same procedures and processing conditions. Also, from the biocomposites studied, two products were manufactured in order to observe the functionality of these materials in two real manufacturing processes such as thermocompression and resin film infusion (Figs. 2 and 3 respectively).

Tensile properties

Tensile testing was performed with an INSTRON universal testing machine model 3366 according to the ASTM D 638-14 [16]. The tests were carried out at 23 °C, with a constant rate of 5 mm/min and type IV samples. All the results were taken as the average value of five samples.

Flexural properties

Three point bending flexural tests were performed with an INSTRON universal testing machine model 3366 according to the ASTM D 790-17 [17]. The tests were carried out on bars of rectangular cross section at 23 °C and at a rate of crosshead motion between 0.9 and 1.3 mm/min (the rate was determined based on the dimensions of the specimen). Also, the distance between the supports was 50 mm and the tests were conducted up to 5% strain. All the results were taken as the average value of five samples.

Differential scanning calorimetry (DSC)

DSC test were carried out using a TA Q2000 differential scanning calorimeter under nitrogen atmosphere at a scanning rate of 10 °C/min, with a sample of 10 mg in aluminium pans. The thermal history of the samples was erased by a preliminary heating cycle at 10 °C/min from 20 to 200 °C and maintaining it at that temperature for 10 min to melting residual crystals, cooling at 10 °C/min to 0 °C and finally, they were heated at °C/min from 0 to 200 °C. The glass transition temperatures (T_g), crystallization temperatures (T_c) and melting temperatures (T_m) were determined from cooling and second heating scans. The melting enthalpies values were normalized according to the proportion of the components in the samples. The crystallinity (χ_c) was determined from the Eq. (1):

$$\chi_c = \left(\frac{\Delta Hm}{\left[\Delta H_m^0 * (1 - w_{Fiber}) \right]} \right) * 100 \quad (1)$$

where w_{Fiber} is the Fique fiber mass fraction, ΔH is the melting enthalpy of the sample and ΔH_m^0 is the specific enthalpy of melting for 100% crystalline LLDPE. This value was reported in literature as 293 J/g [12].

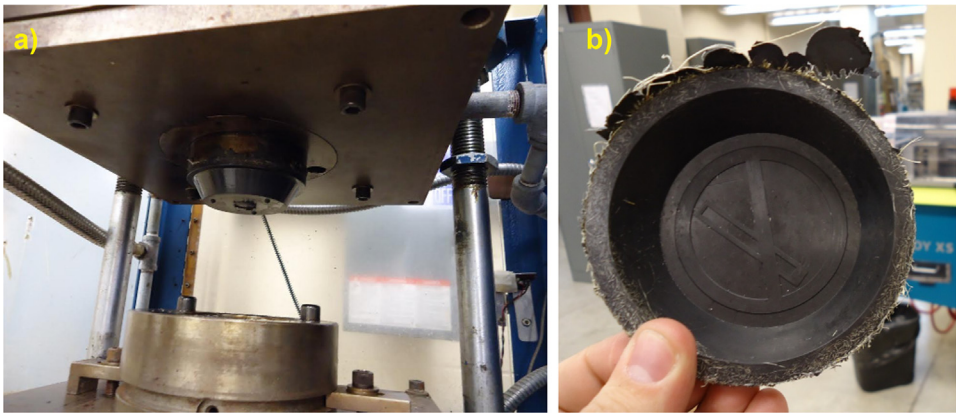

Fig. 2. (a) Thermocompression process and (b) LLDPE-Fique biocomposite part manufactured by thermocompression process. Laboratory of the Polymer Engineering Center (PEC)-University of Wisconsin.

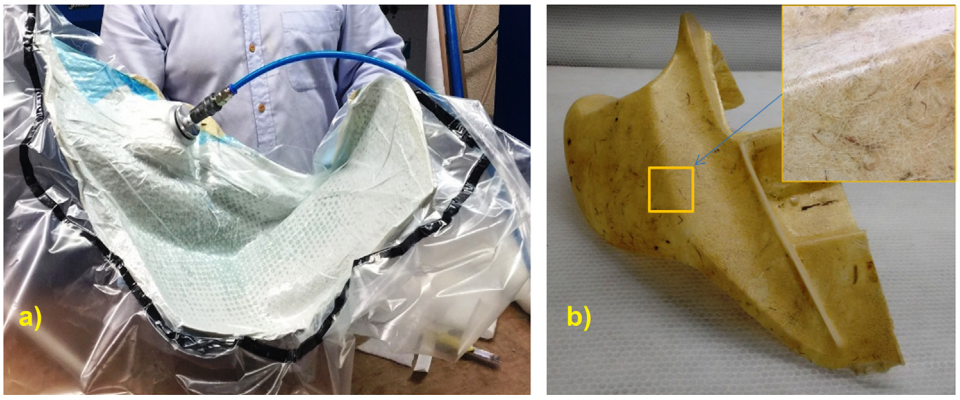

Fig. 3. (a) Resin film infusion process and (b) EP-Fique biocomposite automotive part manufactured by resin film infusion process.

Thermogravimetric analysis (TGA)

TGA was carried out on 10 mg samples using a TA Q500 thermogravimeter at 10 °C/min from 23 to 600 °C under nitrogen flow. The thermal degradation temperatures taken into account were the onset of inflection (T_o) and the temperature of maximum weight loss rate (T_{max}).

Statistical analysis

Tensile and flexural properties of the materials were subjected to analysis of variance (ANOVA), and the Tukey's test was applied at the 0.05 level of significance. All statistical analyses were performed using Minitab Statistical Software Release 12 (Pennsylvania, United States).

Results and discussion

Mechanical properties

The influence of Fique fibers addition on the LLDPE and EP tensile properties was evaluated. The tensile behavior of the materials is shown in Fig. 4. Results of the tensile properties are also presented in Table 1.

The results show that Fique fibers incorporation induces a significant improvement of mechanical tensile properties of LLDPE and EP. For LLDPE-Fique biocomposite tensile modulus (TM) increased 166% and tensile strength (TS) reached an improvement of 36% compared to neat LLDPE. On the other hand, for EP-Fique,

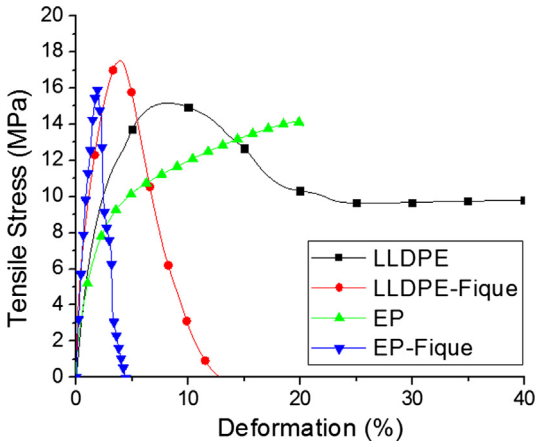

Fig. 4. Average Tensile Stress vs deformation of LLDPE, EP and their biocomposites.

TM and TS were enhanced 700% and 66% respectively regarding to neat EP. Nevertheless, both composites reveal a drastic drop on the deformation capability achieving reductions around 92 and 87% for LLDPE-Fique and EP-Fique respectively. This demonstrates that Fique fiber addition induces a stiffening process on the material.

In this sense, Khan et al. [18] studied the morphology and mechanical properties of short Jute and short E-Glass Fiber Reinforced PP-based composites. The authors report that composites presents higher TM and TS values in comparison with neat PP. In

Table 1
Tensile and flexural properties of LLDPE and LLDPE-Fique composite.

Material	Tensile and Flexural Properties*				
	Tensile properties			Flexural properties	
	Modulus (MPa)	Strength (MPa)	Deformation at break (%)	Modulus (MPa)	Strength (MPa)
LLDPE	514 ± 44[a]	14.4 ± 1.7[a]	215.6 ± 106[a]	576 ± 49[a]	8.2 ± 1.1[a]
LLDPE-Fique	1370 ± 389[b]	19.6 ± 3.2[b]	17.2 ± 3.0[b]	686 ± 28[b]	16.2 ± 0.2[b]

a–b) Different letters in the same column indicate significative differences ($p < .05$).
* Mean of five replications ± standard deviation.

the same way, they also revealed that deformation of the composites is reduced considerably due to the low deformation capability of the Jute and E-Glass fibers in comparison to PP.

Also, Väisänen et al. [6] discusses that the main reason for the poor mechanical properties of natural fiber/polymer composites is the low fiber-matrix interaction which is caused by the different chemical nature of the matrix and the fiber. This incompatibility of the components creates an insufficient stress transfer from the matrix to fibers. Moreover, Hidalgo-Salazar et al. [10], in a study of Fique fiber reinforced LDPE-Al composites suggested a mechanical anchorage on the interface of the materials which would restrain the deformation capability of LDPE-Al-Fique composite.

On the other hand, Fig. 5 shows the flexural behavior of the materials while Table 2 presents the values of their flexural modulus and flexural strength.

According to these results, in the biocomposites both flexural strength (FS) and modulus (FM) were also improved. In fact, Fique fibers addition leads to an increase in the FS (98 and 200%) and FM (20 and 300%) for LLDPE-Fique and EP-Fique respectively when compared with neat LLDPE and EP. From these results, it is clear that LLDPE-Fique and EP-Fique composites gained interesting mechanical properties over the neat matrices.

Differential scanning calorimetry (DSC)

DSC curves for neat matrices and their biocomposites with Fique fibers are shown in Figs. 6 and 7 for LLDPE and EP respectively. Numerical values of the thermal events are shown in Table 3.

The DSC cooling curve of LLDPE (Fig. 6a) shows a main exothermic peak located around 108 °C corresponding to the crystallization of LLDPE chains and another small peak (marked with the arrow in the Fig. 6a) at about 68 °C. This small peak was already observed for Krupa et al. [12] in LLDPE/Wax blends and can be attributed to the crystallization process of the small branches introduced into linear LLDPE by using co-monomers (such as butane or octene). Those crystallization peaks were also observed at similar temperatures for the LLDPE-Fique biocomposite.

The second heating runs of LLDPE and LLDPE-Fique biocomposite were shown in Fig. 6b. Both samples exhibit an endothermic peak between 126 and 127 °C corresponding to the melting of the LLDPE matrix. These results indicate that the addition of the Fique fibers does not disturb the crystallization and melting processes of the LLDPE matrix. Also, a decrease in the melting enthalpy from 144 to 118 J/g was observed for LLDPE and LLDPE-Fique respectively. However, the crystallinity degree of the LLDPE matrix remains the same when the melting enthalpy is corrected considering the weight fraction of Fique fibers in the Eq. (1). This behavior was also observed by Hidalgo-Salazar et al. [10] in LDPE-Al/Fique fibers biocomposites. Therefore, the main reason for the mechanical strength improvement in the LLDPE-Fique biocomposite was the reinforcement effect of Fique fibers in the LLDPE matrix more than crystallinity changes in the thermoplastic matrix.

On the other hand, the DSC data reported in Table 3 show that neat EP and EP-Fique biocomposite does not crystallize on cooling, as confirmed by the absence of crystallization exotherms (Fig. 7a). The heating scans (Fig. 7b) show the glass transition temperature (Tg) of the EP at about 31 °C while for the biocomposite EP-Fique this temperature is around 48 °C. Tg of a polymer is known to depend on the mobility of the chain segment of the macromolecules in the polymer matrix. In this case, the reinforcement effect of the Fique fibers restricts the motion of EP macromolecules chains thus increases the Tg of the biocomposite.

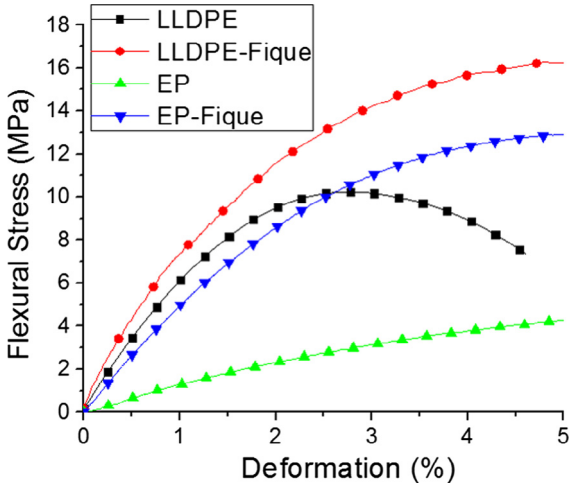

Fig. 5. Average Flexural Stress vs deformation of LLDPE, EP and their biocomposites.

Thermogravimetric analysis (TGA)

Regarding natural fibers, their low thermal stability is considered as one of the limiting factors for its application as reinforcement for composites compared to synthetic fibers [4,6]. In this sense, TG and DTG curves were used to determine the thermal stability of Fique fibers. The results are shown in Fig. 8. Also, main thermal parameters obtained from these curves are summarized in Table 4.

As shown in TG curve (Fig. 8a), Fique fibers presents three mass loss regions which are located around 60–100 °C, 250–350 °C and 350–600 °C. The first weight loss region below 100 °C can be attributed to the evaporation of superficial water present in the sample while the other regions might be associated with the decomposition of the fiber constituents. It can be appreciated in the DTG curve (Fig. 8b), that the first decomposition peak located at 296 °C corresponds to the temperature of maximum weight loss rate (T_{max}) of hemicellulose whereas the second peak located at 358 °C is related to the T_{max} of α-cellulose. The residual weight of Fique fibers has also been measured and is equal to 15.7 wt% at 600 °C. These results are in accordance with other studies of Fique fibers [7].

Table 2
Tensile and flexural properties of EP and EP-Fique composite.

Material	Tensile and Flexural Properties*				
	Tensile properties			Flexural properties	
	Modulus (MPa)	Strength (MPa)	Deformation at break (%)	Modulus (MPa)	Strength (MPa)
EP	134 ± 55[a]	9.9 ± 3.9[a]	23.5 ± 2.1[a]	90 ± 23[a]	4.3 ± 0.9[a]
EP-Fique	1074 ± 198[a]	16.6 ± 1.7[a]	3.8 ± 0.8[a]	390 ± 65[a]	12.9 ± 1.9[a]

a–b) Different letters in the same column indicate significative differences ($p < .05$).
* Mean of five replications ± standard deviation.

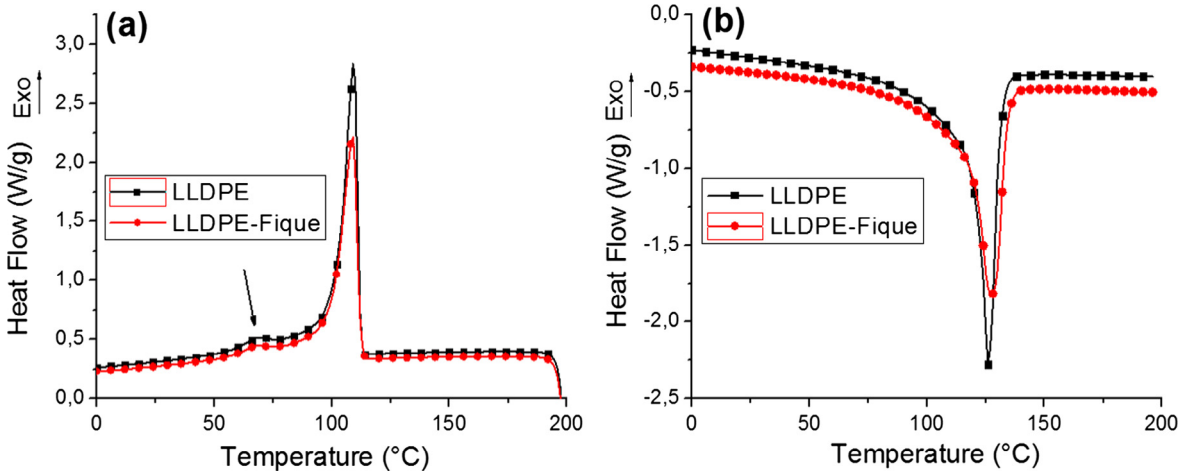

Fig. 6. Heating and cooling DSC curves for (a) neat LLDPE and (b) LLDPE-Fique biocomposite.

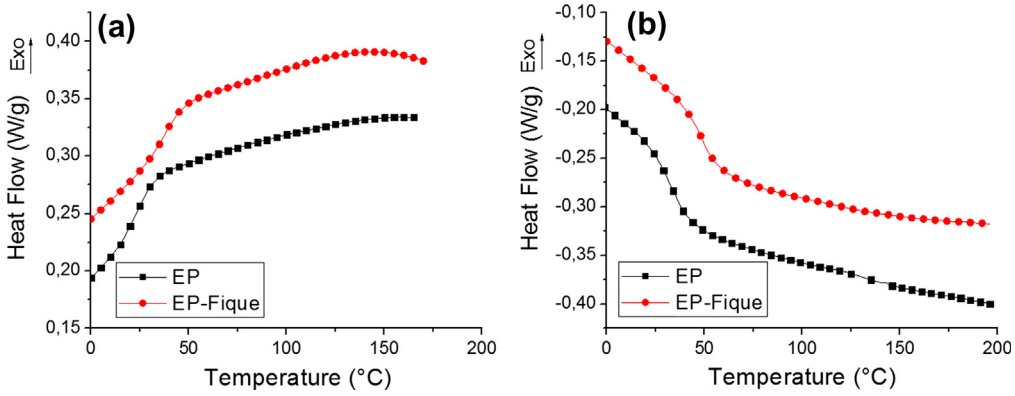

Fig. 7. Heating and cooling DSC curves for (a) neat EP and (b) EP-Fique biocomposite.

Table 3
Thermal properties on cooling and second heating DSC scans of the samples.

Sample	Cooling	Second Heating			
	Tc* (°C)	Tg ½ Cp** (°C)	Tm* (°C)	ΔHm (J/g)	χ (%)
LLDPE	67–109	–	126	144	49
LLDPE-Fique	67–108	–	127	118	50
EP	–	31	–	–	–
EP-Fique	–	49	–	–	–

* Tc and Tm were taken at the maximum peak of crystallization and melting peaks.
** Tg was determinate by the half step temperature.

TG and DTG curves for both LLDPE and LLDPE-Fique biocomposite as well as for both EP and EP-Fique biocomposite are shown in Figs. 9 and 10 respectively. Neat LLDPE degradation occurs in a single step process with an onset temperature (T_o) located at 439 °C and a T_{max} of 478 °C. The residue after final degradation was 4.1%. For the LLDPE-Fique biocomposite degradation occurs in a two steps process. The first degradation step is associated to the decomposition of fiber constituents with a T_o located at 266 °C

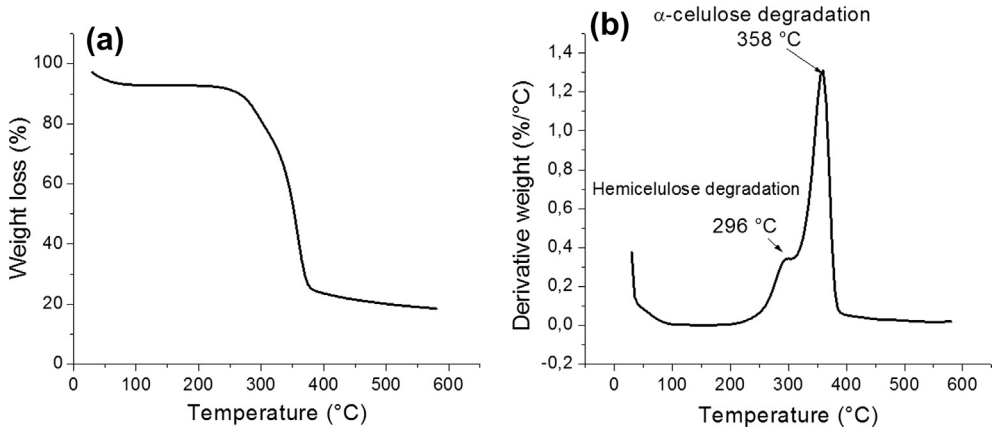

Fig. 8. (a) TG and (b) DTG curves of fique fibers at heating rates of 10 °C/min.

Table 4
Thermal degradation data of the samples at 10 °C/min in nitrogen atmosphere.

Sample	Degradation Stage	T_0 (°C)	T_{max}(°C)	Residual Char (%)
Fique	1	265	296	18.5
	2	335	358	
LLDPE	1	439	478	3.9
LLDPE-Fique	1	266	293	5.9
	2	336	358	
	3	437	470	
EP	1	96	167	6.1
	2	344	365	
EP-Fique	1	96	165	11.1
	2	352	373	

T_o: Onset of inflection of each stage in TG curves.
T_{max}: Peak of the maximum degradation rate in DTG curves.

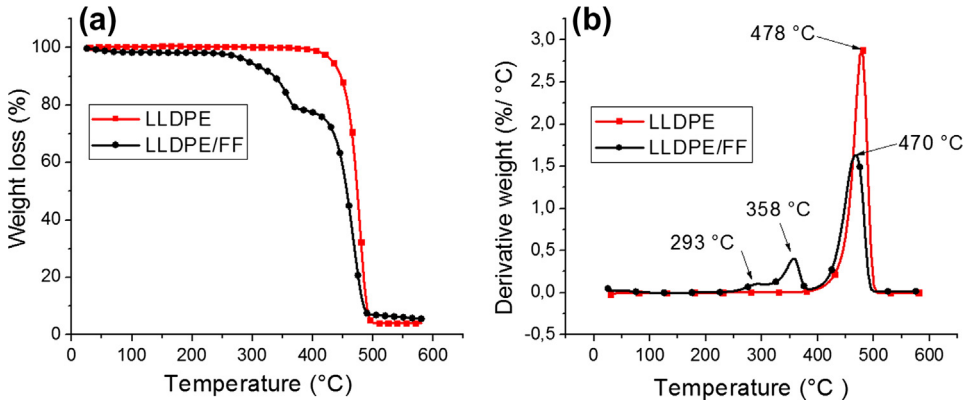

Fig. 9. (a) TG and (b) DTG curves of neat LLDPE and LLDPE-Fique biocomposite at a heating rate of 10 °C/min.

and a mass loss of 21%. The DTG curve of this process display two T_{max} peaks at 293 and 358 °C which are related to the degradation of hemicellulose and α-cellulose as mentioned above. The second degradation step corresponds to the decomposition of LLDPE matrix. This process begins at 437 °C and display a T_{max} value of 470 °C. This result shows a slight decrease on the thermal stability of the polymeric matrix and could be related to an effect of the fiber degradation on the LLDPE during the thermocompression process at 170 °C. Also, the residual weight of LLDPE-Fique increased to 5.6%, due to the Fique fiber addition. On the other hand, neat EP and EP-Fique biocomposite decompose in a two-step weight loss process which indicates that they have a similar thermal degradation behavior. The first degradation step from 90 to 200 °C was attributed to the decomposition of small molecules of the EP [19]. For this stage the observed T_o were 96 and 114 °C while T_{max} were 167 and 165 °C for EP and EP-Fique respectively. Additionally, the second degradation step, observed on the 250–500 °C range, shows the decomposition of the main polymeric chain [19]. In this step, the observed T_o were 344 and 352 °C while T_{max} were 365 and 373 °C for EP and EP-Fique.

As mentioned above, TG curve of Fique fiber (Fig. 8a) show that degradation occurs at 296 °C. Meanwhile neat EP starts it decomposition at a temperature of 96 °, which is lower than that of the fiber. As consequence EP-Fique degrades after neat resin in both

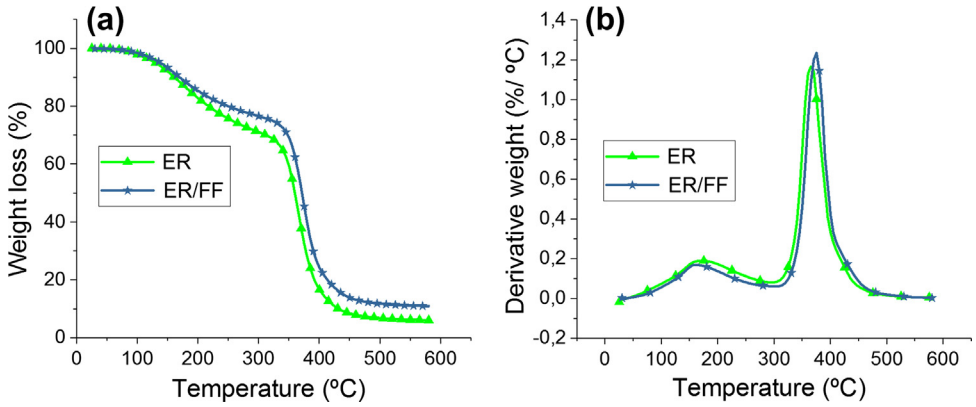

Fig. 10. (a) TG and (b) DTG curves of neat EP and EP-Fique biocomposite at a heating rate of 10 °C/min.

degradation steps. These results are consistent with previously published data on EP-*Phormium tenax* leaf fibers composites and can be due to improved fiber–matrix interaction [20]. The residues after final degradation were 6.1% for neat EP and 11.1% for EP-Fique biocomposite. This increase on the residual char of the composite can be due to the Fique fiber addition.

Conclusions

In this work LLDPE-Fique and EP-Fique biocomposites (20% fiber by weight) were successfully prepared using compression moulding and resin film infusion processing techniques respectively.

Tensile and flexural mechanical characterization showed that nonwoven Fique reinforced composites exhibited higher strength and modulus but lower deformation capability as compared with LLDPE and EP neat matrices.

TG thermograms showed that nonwoven Fique fibers incorporation has an effect on the thermal stability of the composites. On the other hand, Fique fibers did not change the crystallization and melting processes of the LLDPE matrix but restricts the motion of EP macromolecules chains thus increases the Tg of the EP-Fique biocomposite.

Finally, this work opens the possibility of considering nonwoven Fique fibers as a reinforcement material with a high potential for the manufacture of thermoplastic and thermoset biocomposites for several applications.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.rinp.2017.12.025.

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