Characterization of High Density Polyethylene (HDPE) Reinforced with Banana Peel Fibers

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Lignocellulosic fibers from banana peels were washed with water in order to increase their compatibility with a polymeric matrix, and their properties were compared with unwashed fibers. Washed banana fibers were mixed with high density polyethylene (HDPE) and placed in an injector chamber to produce specimens for tensile tests. Samples of washed banana fibers/HDPE composites were characterized by tensile tests and thermal analysis. The chemical composition of unwashed and washed banana fibers was analyzed by thermogravimetric analysis, scanning electron microscopy, and X-ray diffraction. The treatment with water was effective at removing extractives and increasing the surface roughness, thereby increasing the thermal stability of the fibers. However, results showed that the addition of washed banana fibers decreased the thermal stability of composites, while increasing the melting and crystallization temperatures of composites. The addition of 5 wt% fibers also provided an improvement in mechanical properties of composites in comparison with pure HDPE.

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INTRODUCTION

A recent global trend is to use natural, renewable, alternative resources that are beneficial in developing new materials. Researchers are looking forward to developing new materials that address socioeconomic needs as well as environmental problems (Oliveira *et al.* 2012). For this reason, studies and applications of lignocellulosic composites are being further pursued as a good alternative resource (Ibrahim *et al.* 2010).

Natural fibers as reinforcement for polymeric matrices have been studied due to their numerous advantages and ideal environmental performance when compared to inorganic fibers, such as glass or carbon fibers (Xie *et al.* 2010; Awal *et al.* 2011; Venkateshwaran *et al.* 2013). The main advantages presented by these fibers are: abundance and therefore low cost, biodegradability, flammability, flexibility during processing, low density, relatively high tensile and flexural modulus, and non-toxicity (Xie *et al.* 2010; Qin *et al.* 2011; Antich *et al.* 2006; Kim and Netravali 2010; Pereira *et al.* 2010, 2011; Maschio *et al.* 2012).

In tropical countries, such as Brazil, fibrous plants are available in abundance. Among these plants, the banana tree is highlighted as being typically from tropical and subtropical regions. Therefore, it takes constant heat and high humidity in order for the tree to exhibit proper growth and production, such as the climate in regions near the

Brazilian coast and in southern Asian countries, especially in India (CEPEA 2011; Venkateshwaran *et al.* 2012).

According to the data obtained from Embrapa (2011), Brazil is the fifth highest banana-producing country in the world, with a production of 6.783.480 tons from 479.614 hectares in 2009 (Table 1). World consumption of banana fruit is about 10.43 kg/inhabitant/year. The largest consumers are South American inhabitants, consuming approximately 21.23 kg/inhabitant/year. In second place comes Central America, with 13.90 kg/inhabitant/year, and Oceania in third with 11.26 kg/capita/year (Vieira 2010). Despite the large consumption of bananas and hence the large amount of peels that are thrown away, there are few industrial uses for this residue which represents 30 to 40% of the weight of the fruit (Albarelli *et al.* 2011).

Table 1. Production of Banana Fruit per Country in 2009*

Country	Area (hc)	Production (ton)	Yield (ton/hc)
India	748.100	26.996.600	36.09
Philippines	446.400	9.013.190	20.19
China	350.224	9.006.450	25.72
Ecuador	216.115	7.637.320	35.34
Brazil	479.614	6.783.480	14.1
Others	2.683.131	37.941.232	14.14
World	4.923.584	97.378.272	19.78

*(Embrapa 2011)

Some attempts to apply this residue were carried out for production of protein methane, ethanol, pectins, and enzyme extracts (Albarelli *et al.* 2011). It has also been used as livestock feed and heavy metal bio-absorbents, dyes, and to remove phenolic compounds (Albarelli *et al.* 2011; Achak *et al.* 2009; Anwar *et al.* 2010; Comim *et al.* 2010). However, nothing was found in the literature on the use of fibers of banana peel as reinforcement in polymer composites.

Several authors, such as Guimarães *et al.* (2010), Ibrahim *et al.* (2010), Oliveira *et al.* (2012), and Venkateshwaran *et al.* (2013) studied the use of fiber extracted from banana pseudostem as reinforcement in polymer composites. Ibrahim *et al.* (2010), for example, studied the banana fiber and its microfibrils as reinforcement in polymer composites of HDPE, using alkaline treatments and different amounts of fiber inserted in the matrix, and concluded there was an improved fiber/matrix adherence, resulting in improved mechanical properties.

With the use of reinforced natural fibers in polymeric composites, the main identified problem is the poor compatibility between the fibers and the matrix. In order to optimize this fiber/matrix interface and to promote adhesion, various chemical treatments can be applied on the surface of the fibers, such as alkaline treatment, bleaching, acetylation, and steaming (Venkateshwaran *et al.* 2013; Rosa *et al.* 2009; Bessadok *et al.* 2009). Among these treatments, the most commonly applied ones are the chemical treatments and have, as the main objective, the removal of amorphous materials such as lignin, waxes, and extractives. Such materials tend to make the fiber incompatible with polymeric matrices (Carvalho *et al.* 2010). However, according to Oliveira *et al.* (2012), the application of chemical surface treatments has been limited in industrial applications

due to problems related to the high cost of the treatment and the disposal of the used chemical products.

In the present work, the usual chemical treatments were replaced with simple washing with water in order to simplify and lower the cost, and also to evaluate whether washing with no chemical reagents is sufficient to improve the compatibility between the fibers and the matrix. Fibers obtained from banana peels and fibers washed with water were characterized according to their chemical, physical, thermal, and morphological properties. The fibers were then incorporated into the HDPE through processing in a thermokinetic mixer and injection molding to produce composite materials. HDPE, as well as the composites, were characterized by thermal analysis, and a tensile test to evaluate the effects of the fibers addition on the composites thermal and mechanical properties.

EXPERIMENTAL

Materials and Methods

Materials

Banana fibers were obtained from the peel of banana fruit (*Musa*, genotype AAB). The peels were separated, dried at 50 °C for 48 h, after being crushed in a common processor, and finally sieved to obtain a sample that passed through a 50 mesh (opening 300 mm/µm). High density polyethylene (HDPE), designated as HI-60070, was supplied by Riopol and used as the composites matrix.

Fiber treatment

The banana peel fibers were washed with distilled water (50 g crushed fiber/500 mL water) for 10 min at room temperature. Once the washing was complete, the solution was filtered in a vacuum filter, and the fibers were dried in an oven at 50 °C (122 °F) for 48 h. Figure 1(a) and (b) show images of the banana fibers before and after washing.

(a)   (b)

Fig. 1. (a) Banana fibers (BF) and (b) washed banana fibers (WBF)

HDPE/banana fibers composites preparation

The washed banana fibers (WBF) were mixed with the high density polyethylene (HDPE) in a thermokinetic mixer, model MH-50H, with the speed rate kept at 5250 rpm. Fibers are made up of 5 and 10 wt% of the composition. After mixing, the composites were dried and ground in a mill, model RONE. Next, HDPE/banana fibers composites

were placed in an injector chamber at 165 °C and heated at the rate of 2 °C·min^{-1}. The melted material was injected into the required dimensions in a pre-warmed mold in order to obtain tensile specimens. Pure HDPE specimens were submitted to the same process for comparison.

Characterization of Raw Materials and Composites

Chemical composition

Fibers were chemically characterized to evaluate the effect of washing with distilled water on their fiber chemical composition by quantifying the amount of cellulose, hemicellulose, lignin, and ash. The characterization was performed according to the analytical methodology described by Rocha (2000) and validated by Gouveia *et al.* (2009).

X-ray diffraction (XRD)

The crystallinity index of BF and WBF was evaluated by X-ray patterns obtained with a Shimadzu diffractometer, model XRD6000, under the following conditions: CuKα radiation with graphite mono-chromator, 40 kV, and 40 mA. The patterns were obtained in 10 to 70° 2θ angular interval, scanning 0.05 (2θ/5s) for values of 2θ between 10° and 70°.

The crystallinity index (CI) was calculated using Equation 1, where I_{002} is the maximum intensity of the I_{002} lattice reflection, and I_{am} is the minimum intensity of X-ray scattering broad band due to the amorphous region of the sample. This method was developed by Segal *et al.* (1959), and it has been widely used for the study of natural fibers.

$$CI(\%) = [(I_{002} - I_{am}) / I_{002}] \times 100 \tag{1}$$

Thermal analysis (TG/DTG)

The thermogravimetric analysis (TGA/DTG) was performed in a study on the thermal characteristics of banana fibers and HDPE composites. The thermal behavior was determined using a Shimadzu TGA-50 model, in an open platinum crucible with a heating rate of 20 °C/min in 50 mL/min flow of nitrogen, in a temperature ranging between 25 °C and 900 °C.

The differential scanning calorimetric analyses (DSC) of the composites were performed in a DSC calorimeter (Seiko) under nitrogen atmosphere. The samples were heated up to 300 °C then cooled down to 25 °C, and again heated up to 300 °C, with a heating rate of 10 °C/min, to eliminate previous thermal history.

The glass transition temperatures were determined using ASTM D 3418-08. The method used was extrapolation, by drawing two parallel lines to curves before and after increasing the flow of heat at temperatures between 60 °C and 70 °C.

Tensile tests of composites

Five specimens of each composite with different compositions and pure HDPE were tested in an Instron universal testing machine (Instron model 8801). Tensile tests were carried out according to ASTM D638 (Type I) at a crosshead speed of 10 mm/min using a 100 kN load cell without an extensometer.

Tensile strength, maximum strength, and tensile modulus values were automatically calculated by the Wave Maker Runtime software provided by Instron.

Scanning electron microscopy (SEM)

The fibers, as well as the composites fracture surface, were analyzed with a JEOL JSM5310 scanning electron microscope with a tungsten filament operating at 10 kV, using secondary electrons and samples covered with a thin layer of gold.

RESULTS AND DISCUSSION

Fibers Chemical Composition

Table 2 presents the fiber chemical composition from banana peels (BF and WBF) and banana fibers from pseudostem (obtained from literature) in order to compare chemical compositions of fibers obtained from different parts of a same banana tree. By analyzing the data, it is possible to observe that the percentage amount of insoluble lignin and ash increased to about 77% and 163%, respectively, after washing. According to Jústiz-Smith *et al.* (2008), the amount of ash gives an indication of the carbon content, so that a higher amount of ash represents a lower amount of carbon.

After washing, the percentage amount of other components as soluble lignin, hemicellulose, and moisture decreased, as expected, since the soluble lignin and hemicellulose are water-soluble components.

As the chemical analysis is in relation to the percentage amount of each component in the fiber, the decrease of a given component consequently represents the percentage increase of other components.

In this way, the decrease of about 37% in cellulose content is not related to the absolute amount of cellulose, once it is insoluble in water, but to its percentage quantity in the fibers in relation to other components.

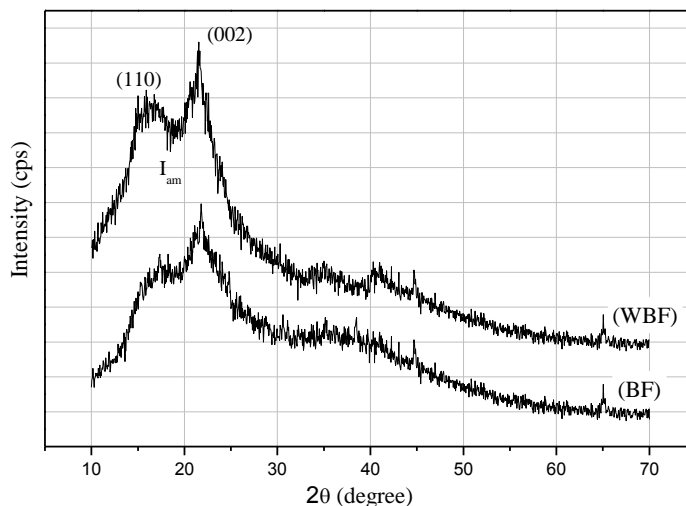**Table 2.** Chemical Composition and Crystallinity Index of Banana Fibers

Component (%)	BF	WBF	From pseudostem*
Cellulose	27.07± 0.15	19.71 ±0.02	50.15 ±1.09
Soluble lignin	0.78± 0.01	0.13 ±0.00	17.44 ±0.19
Insoluble lignin	27.73 ±0.07	49.16 ± 0.05	
Hemicellulose	19.71± 0.10	15.76± 0.13	0.77 ±0.58
Ash	0.41 ± 0.41	1.10± 0.02	4.14 ±0.92
Moisture	14.45 ± 0.41	13.49 ± 0.68	8.57 ±0.19
Cristallinity index (%)	23.78	17.45	39.00

*Guimarães *et al.* 2009

The reduction in lignin solution was approximately 50%, which is relevant when the fiber is used as reinforcement in composites, because the removal of amorphous components, such as lignin, contributes to a better adhesion between the fiber and the matrix (Carvalho *et al.* 2010). Emaga *et al.* (2008) in the study of banana peels (*Musa*, genotype AAB) determined there was 6.4% cellulose, 0.6% hemicellulose, and 15.4% lignin. These values were lower from those obtained for the fibers analyzed in this work, although the fibers were obtained from banana fruits of the same species. Comparing the values obtained from banana fibers from different sources, it is possible to observe that fibers from pseudostem have higher cellulose and ash contents, but have a lower content of hemicellulose and moisture than the fibers obtained from banana peels.

X-ray Diffraction

Figure 2 shows the X-ray diffractograms of the banana fibers (BF and WBF). Two peaks were observed for both samples at $2\theta = 16°$ and $2\theta = 22°$, representing the cellulose crystallographic planes I_{101} and I_{002}, respectively. The superposition of the X-ray diagrams shows that the signal characteristics of fibers in different conditions are similar.

Fig. 2. X-ray diffraction patterns of banana fiber (BF) and washed banana fiber (WBF)

The crystallinity index (I_c), calculated according to Equation 1, can be observed in Table 1. According to the obtained results of fiber chemical compositions, as the percentage amount of cellulose, a semi-crystalline component, decreased, while the percentage amount of amorphous soluble lignin increased. Therefore, the fibers crystallinity index after washing decreased to about 26.6%.

Fibers Thermal Analysis

Thermogravimetric analyses were performed with the objective of determining the thermal stability and the influence of washing on the fibers thermal properties. TGA and DTG curves are shown in Fig. 3.

It can be seen that curves in Fig. 3(a) and (b) show different behavior, with four main weight loss steps for the BF and two main weight loss steps for the WBF. In the first step (114 °C and 72 °C for BF and WBF, respectively), a weight loss related to desorption of water of the polysaccharide structure was observed. Although fibers were dried before the analysis, according to Guimarães *et al.* (2009), the total elimination of water is quite difficult due to their hydrophilic nature. For instance, water is present even as structurally bound water molecules.

For the BF, the second step (216 °C) can be attributed to the decomposition of the hemicellulose contents. The third one (324 °C) is attributed to the residual lignin decomposition reaction degradation, and the fourth one (397 °C) is due to the breaking of the protolignin bonds (Guimarães *et al.* 2009).

For the WBF, the second and main step at 378 °C is related to the decomposition of cellulose and lignin.

The presence of only two peaks at DTG curves of the WBF is an indicator of a more homogeneous composition than BF fibers, due to the removal of some water soluble components. According to the results presented in Table 3, the BF exhibited thermal stability until reaching 153°C, while, after washing, the thermal stability increased, suggesting that WBF can be safely used up to the maximum temperature of 200 °C. This temperature is similar to that observed by Guimarães *et al.* (2009) and Kumar *et al.* (2008).

(a)

(b)
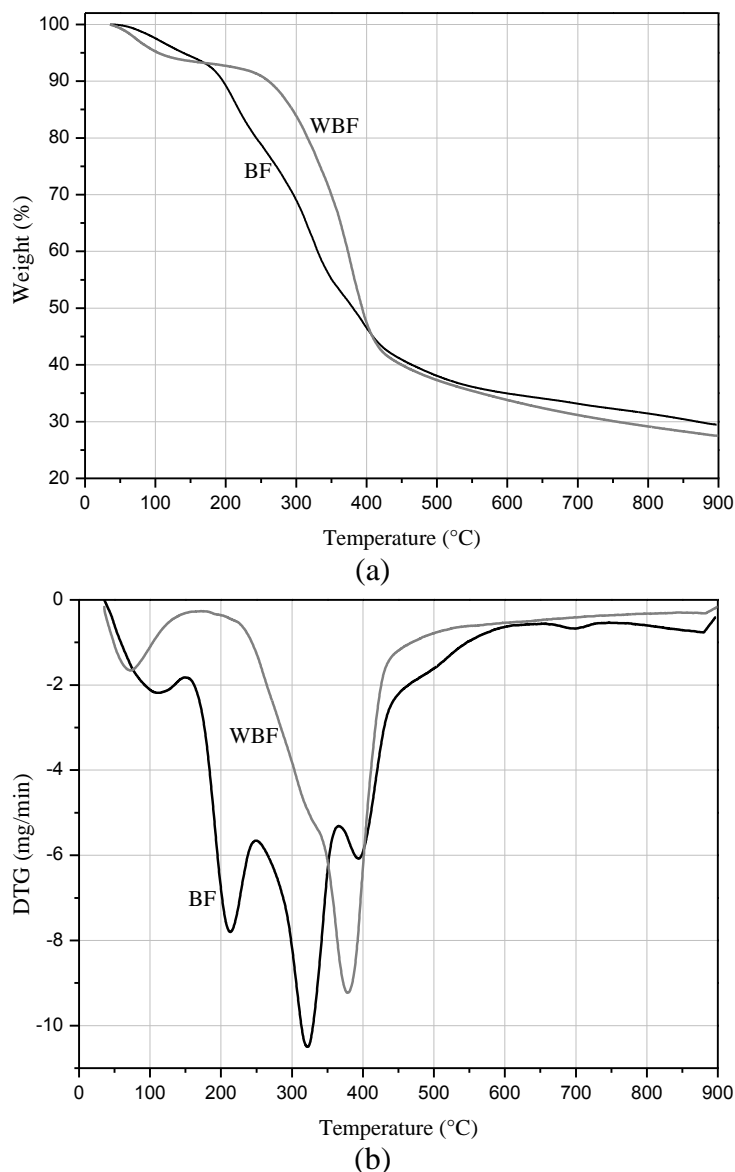
Fig. 3. (a) TGA and (b) DTG curves of banana fibers (BF) and washed banana fibers (WBF)

Although the washing could not remove all of the amorphous constituents present in the fiber, it increased the temperature at which the degradation of the fiber occurs; thus there was an improvement in the fiber's thermal stability.

Table 3. Results of the Thermogravimetric Analysis (TG)

Sample	ΔT (ºC)	Tpeak (°C)	Tonset (°C)	Mass loss (%)	Residue (%)
BF	35-160	114		5.48	
	160-260	216		16.94	29.47
	260-375	322	153	26.14	
	375-900	394		21.96	
WBF	35-180	72	200	6.94	27.50
	180-900	378		65.56	

Analyzing the data in Table 3, it is possible to observe that the major weight loss for the BF occurred along the third step (26.14%), and the weight loss occurred mainly in the second step between 180°C and 900 °C for the WBF. The percentage of residue in washed banana fibers (WBF) decreased with respect to the composition of the banana peel, as indicated in Table 3.

Scanning Electron Microscopy (SEM)

The effect of washing on fiber surface morphology was analyzed by scanning electron microscopy (SEM). Figure 4 shows the changes in the banana peel fibers morphology before and after the washing.
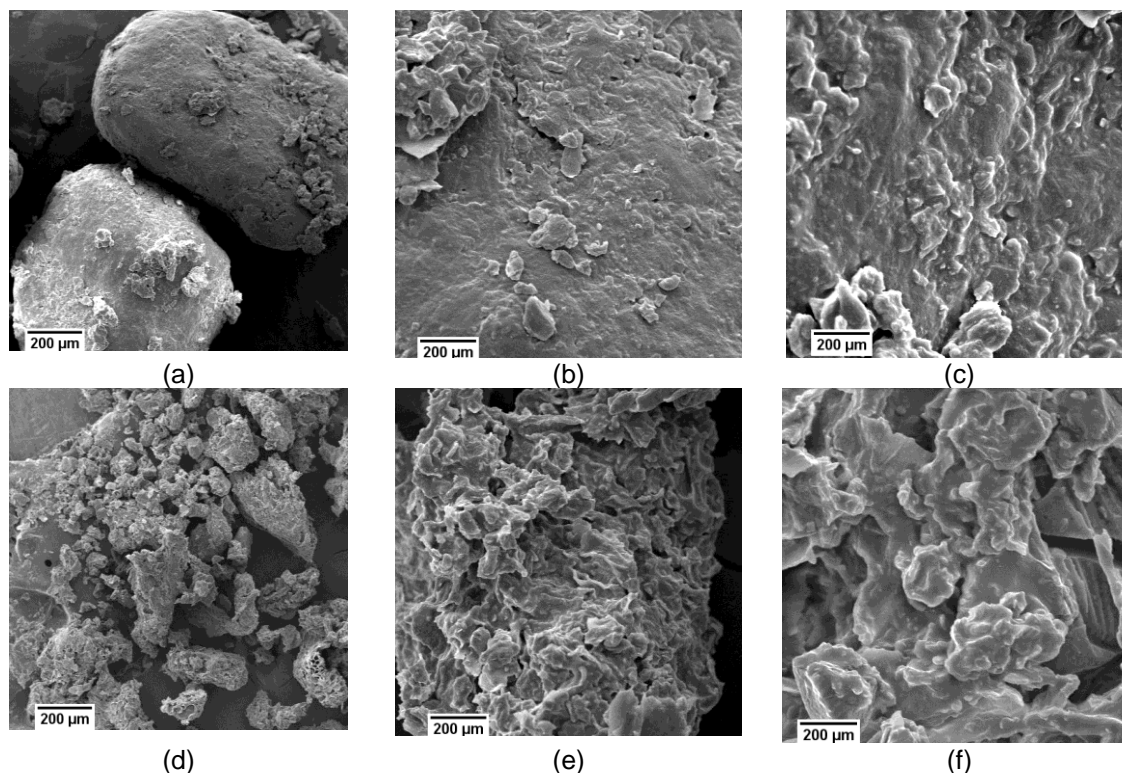
Fig. 4. SEM micrographs of fiber surface of: banana fiber (BF) (a), (b), and (c) and washed banana fibers (WBF) (d), (e), and (f)

From Fig. 4(a), it is possible to observe the presence of oval particles, as Albarelli *et al.* (2011) also observed in the study of the effects of supercritical carbon dioxide on waste banana peels for heavy metal removal. Micrographs in Fig. 4(b) and (c) show a largely smooth area with some lumps on the surface due to the presence of a layer of

substances such as oils, waxes, and extractives, which are part of the natural constitution of lignocellulosic fibers.

After the washing, shown from Fig. 4(d) to (f), the WBF presented a rough surface due to the superficial layer removal, with an undefined format particles, as well as particles that are smaller than untreated fiber particles. The removal of the superficial layer, formed by amorphous constituents, and the subsequent improvement in the surface roughness is important because it provides a better mechanical anchorage with the polymeric matrix.

Composites Characterization

It was possible to establish the degradation temperatures and the effect of fiber addition on the thermal stability of composites by the characterization with thermogravimetric analysis. The TGA and DTG curves for HDPE and its composites with 5% and 10% of washed banana fibers are presented in Fig. 5.

(a)

(b)
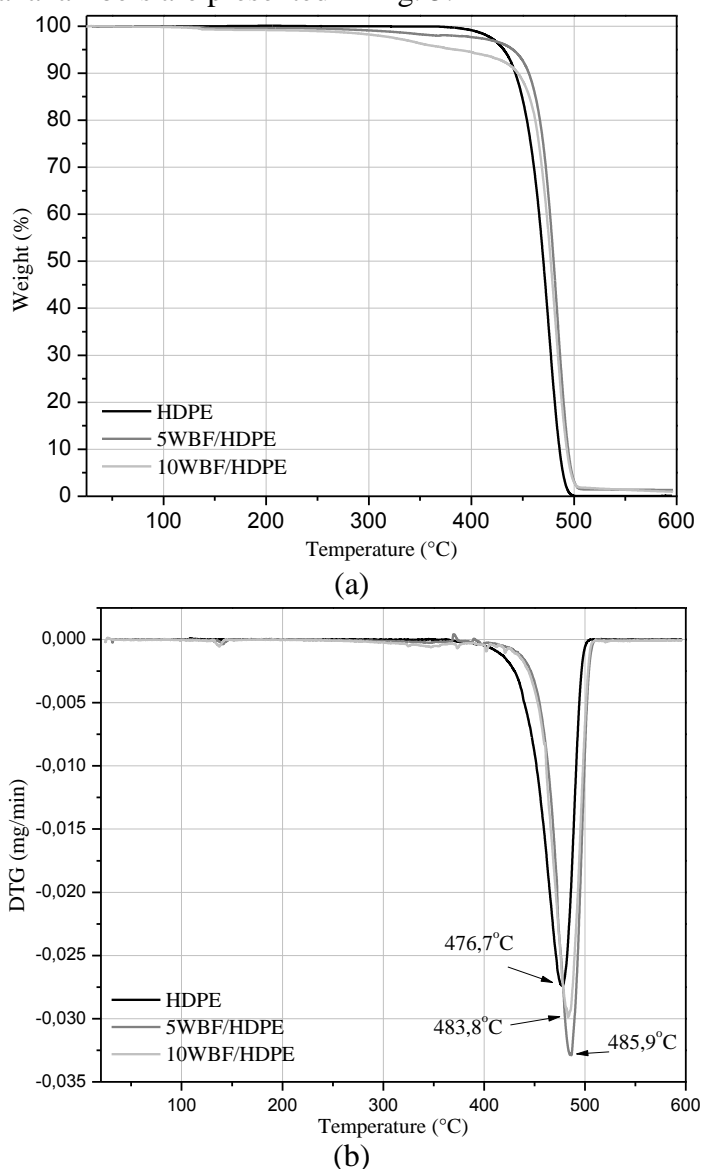
Fig. 5. Thermogravimetrical analysis for HDPE and WBF/HDPE composites (a) TGA and (b) DTG curves.

Analyzing the TGA curves, it can be seen that the HDPE was thermally stable up to a temperature of 350 °C. From this temperature, the HDPE process of thermal decomposition occurred at only one stage until 500 °C.

With the addition of washed banana fibers, it was verified that there was a slight reduction in thermal stability of the material, and that the degradation process occurred in two distinct stages: the first one occurred between 300 °C and 400 °C, related to decomposition of fibers and was almost imperceptible in DTG curves due to the small amount of reinforcement used (5 and 10%), and the second one occurred between 400 °C and 550 °C, with peaks at 476.7 °C, 485.9 °C, and 483.8 °C for HDPE, 5WBF/HDPE, and 10WBF/HDPE, respectively.

The initial degradation and the residue obtained at the end of the degradation process are summarized in Table 4. It is possible to observe that the addition of WBF in the pure polymer increased the amount of residue after the thermal degradation, due to the decomposition of some fiber components that had initial degradation temperatures that are over 600 °C.

Table 4. Weight Loss and Degradation Peaks for HDPE and Its Composites

Sample	Tpeak (°C)	Tonset (°C)	Residue (%) 600 °C
HDPE	476.7	380.1	0.1
HDPE/5WBF	485.9	272.9	1.3
HDPE/10WBF	483.8	238.3	1.0

The melting and crystallization behavior of the composites and the pure HDPE, obtained from DSC analysis, are shown in the curves of Figs. 6 and 7, and the quantitative results are summarized in Table 5.
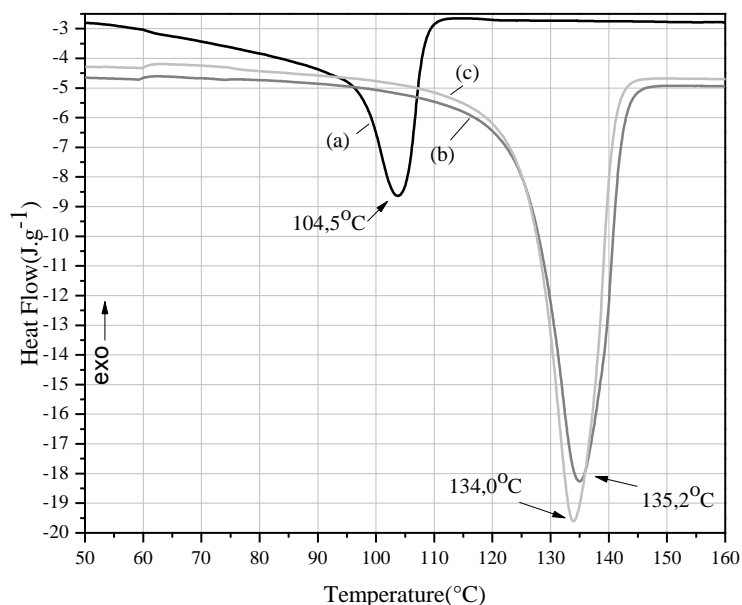

Fig. 6. DSC second heating run of (a) HDPE, (b) 5WBF/HDPE, and (c) 10WBF/HDPE

The crystallinity index (X_c) for HDPE and the composites were assessed from Equation 2,

$$X_c = \frac{\Delta H_m * 100}{\Delta H_m^0 * w} \qquad (2)$$

where X_c is the crystallinity index, ΔH_m is the melting enthalpy of sample, ΔH_m^0 is the standard melting enthalpy, and w is the weight fraction of HDPE in the composite. The value of ΔH_m^0 obtained for HDPE was 293 J·g^{-1}.
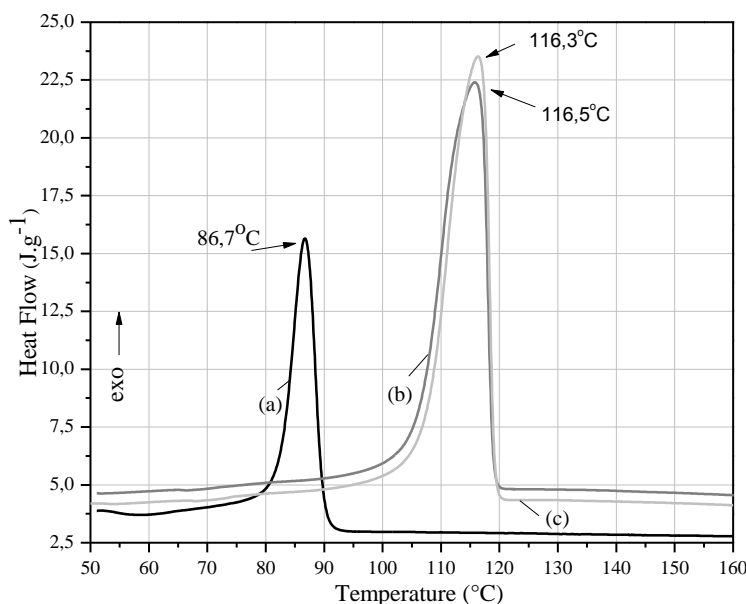
Fig. 7. DSC cooling run of (a) HDPE, (b) 5WBF/HDPE, and (c) 10WBF/HDPE

The fibers addition caused an increase in the melting and crystallization temperatures, as well as in the crystallinity index in relation to the pure HDPE, which is explained by the fact that the fiber provided higher ordering for the internal structure in the composite.

Table 5. Melting and Crystallization Parameters of HDPE and WBF/HDPE Obtained from DSC Measurements

Samples	T_c peak (°C)	T_m peak (°C)	ΔH_m (J.g^{-1})	X_c (%)
HDPE	86.7	104.5	188.3	64.3
5WBF/HDPE	116.5	135.2	183.1	65.8
10WBF/HDPE	116.3	134.0	183.9	69.7

T_c: crystallization temperature, and T_m: melting temperature

The crystallinity index value increase of the HDPE after the addition of fibers was also observed by Lei and Wu (2012), with the addition of 40% (w/w) of wood fibers in the polymeric matrix.

The melting peak temperatures (T_m) of the HDPE in composites presented a slight decrease with the increase of fiber content. According to Choudhury (2008), the

occurrence of a lower melting peak can be attributed to the fact that strong nucleation on fibers surfaces decreased the time of HDPE crystallization. As a consequence, smaller crystals were formed, creating a decrease in the melting temperature.

Mechanical properties such as elongation at break, tensile strength, and tensile modulus for HDPE and its composites with 5 wt% and 10 wt% WBF are presented in Table 6. The results showed that composites presented higher tensile strength compared to the pure HDPE. However, composites reinforced with 5 wt% fiber showed tensile strength about 7% higher than the composite that was reinforced with 10 wt%, which demonstrates that more than 5 wt% fiber as reinforcement was detrimental to mechanical properties.

Table 6. Mechanical Properties of WBF/HDPE Composites

Samples	Elongation (%)	Tensile strength (MPa)	Tensile modulus (MPa)
HDPE	2.1 ± 0.3	15.7 ± 1.1	732.5 ± 90.6
5WBF/HDPE	2.3 ± 0.1	19.1 ± 0.6	814.5 ± 8.5
10WBF/HDPE	2.4 ± 0.2	18.2 ± 0.4	760.5 ± 5.3

The values of tensile modulus for composites reinforced with 10 wt% of fibers in relation to those reinforced with 5 wt% means that the washing with water was not efficient in the improvement of fiber/matrix adhesion. A good mechanical and chemical anchorage between the fiber and the matrix is responsible for an improvement in the mechanical properties. As the treatment with water did not remove enough amorphous components, with the addition of 10% of fibers in the manufacture of composites, the fibers acted as a tensile concentrator rather than acting as reinforcement.

CONCLUSIONS

1. Washed banana fibers obtained from banana peel presented a considerable reduction in impurity level and a decrease in crystallinity when compared with an unwashed fiber. Also, after washing, the fiber had greater stability in temperatures up to 200 °C, and more roughness on its surface.

2. The addition of washed banana fibers in the HDPE decreased the thermal stability of the composite and increased the residue from its thermal degradation process. Melting and crystallization temperatures, as well as crystallinity, also increased with the addition of fiber in the polymeric matrix.

3. The addition of washed banana fibers increased the tensile strength and tensile modulus of composites. However, the amount of fiber in the composite should be 5 wt%, since a higher amount of reinforcement leads to lower values for mechanical properties.

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