Manufacturing and Characterization of High Impact Polystyrene (HIPS) Reinforced with Treated Sugarcane Bagasse

Kelly Cristina Coelho de Carvalho Benini*, Herman Jacobus Cornelis Voorwald and Maria Odila Hilário Cioffi

Fatigue and Aeronautical Materials Research Group, Department of Materials and Technology, UNESP - Univ. Estadual Paulista, 12516-410 Guaratinguetá, São Paulo, Brazil

Abstract: Natural fibers obtained from sugarcane bagasse were used as reinforcement for high impact polystyrene (HIPS) composites. Fibers were chemically treated with an alkaline solution and then bleached with sodium chlorite and acetic acid, in order to remove amorphous constituents and improve adhesion with polimeric matrix.The alkali-treated and bleached fibers over a range of 10-30 wt% were mixed with HIPS and placed in an injector chamber in order to obtain tensile and flexural test specimens. Chemical treatment effects on composites properties were evaluated through mechanical tests and thermal and microscopy analysis. Experimental results show that composites with 30 wt% of alkali-treated fibers present an improvement in the tensile strength (17%), tensile modulus (96%) and flexural modulus (34%) with a consequent decrease in the ductility and in the thermal properties in comparisson to pure HIPS. An huge increase of 191% in the flexural modulus for composites with 30 wt% of bleached fibers was obtained compared to pure HIPS.

Keywords: High impact polystyrene, Mechanical properties, Thermal properties, Sugarcane Bagasse fibers.

1. INTRODUCTION

Fibers from natural resources as reinforcement for polymeric matrixes have been studied for decades due to many gained advantages and environmental appeal when compared to inorganic fibers, such as glass and carbon fibers [1-3]. The main advantages presented by these fibers are: abundance and therefore low cost, biodegradability, flammability, processing flexibility, low density, relatively high tensile and flexural modulus and non-toxicity [1, 4].

As reinforcement of thermoplastic polymers, natural fibers provide an improvement in mechanical properties, the use of a lower amount of pure polymer and reduce the cost and the density of composite materials [3]. On the other hand, their potential use as reinforcement is greatly reduced because of their incompatible with the hydrophobic and non-polar polymeric matrix [5-8]. This incompatibility may cause a weak interfacial adhesion and, as a consequence, the stress transfer efficiency from matrix to fibers is reduced [1, 7, 9].

Adhesion between fiber and matrix, an important parameter that affects the composites mechanical properties, results either from a physical origin or a chemical cross-linking [10-12].

In order to optimize interfacial adhesion and moisture absorption of natural fibers composites, various chemical treatments can be applied on fibers surface [10, 13, 14]. Some of them, such as alkali-treatment and bleaching with sodium hypochlorite (NaClO) or chlorite ($NaClO_2$), are applied by several researches [12, 15-17] increasing the compatibility with polymeric matrix [10].

Treatments with alkaline solutions promote the removal of partially amorphous constituents such as hemicellulose, lignin, waxes, and oils soluble in alkaline solution, and therefore reduce fiber diameter and the level of fiber aggregation. A better fiber/matrix adhesion is obtained with a rougher surface [10, 18, 19].

Another chemical treatment used is the bleaching of fibers with sodium chlorite ($NaClO_2$), acidified in acid solution, leading to the formation of choleric acid ($HClO_2$) and chlorine dioxide (ClO_2), which reacts with lignin constituents and with hydrophilic hydroxyl groups of hemicellulose removing lignin from the fibers [10].

Sugarcane bagasse is an agro-industrial residue generated after the process of extraction from the sugarcane industry in the manufacturing of sugar and alcohol. Due to its high cellulose content, this fiber is considered as one of the largest natural fiber available resource. As a consequence, used for several applications [20] such as reinforcement for polymeric composites [21-24], adsorbing materials [25, 26] and components for construction industries [27, 28].

Vegetal fibers are basically composed of cellulose, hemicellulose and lignin and, as these fibers are from a

*Address correspondence to this author at the Fatigue and Aeronautical Materials Research Group, Department of Materials and Technology, UNESP - Univ. Estadual Paulista, 12516-410 Guaratinguetá, São Paulo, Brazil; Tel: +55-12-31232865; Fax: +55-12-31232852; E-mail: kcccarvalho@hotmail.com

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natural source, the morphological and chemical composition will vary depending on growth, extraction, soil and weather conditions [29]. This variability of natural fibers properties and some parameters such as fibers dispersion quality in the polymeric matrix, and final fibers dimension causes an effect on the composites properties and therefore, need to be controlled in the manufacturing of these materials [31-33].

In the present research, alkali-treated and bleached sugarcane bagasse fibers were used as reinforcement in HIPS matrix in order to analyze the influence of the chemical treatment and the percentage of fibers on the mechanical, thermal and physical properties of composites.

2. EXPERIMENTAL

2.1. Materials

Sugarcane bagasse fibers were kindly supplied by Edras Ecossistemas. Both fibers were dried at 100°C for an hour, ground in a mill and afterwards, sieved to obtain a sample that passed through a 45 mesh (opening 354 μm).

High Impact Polystyrene (HIPS 825) produced by FINA Technology and kindly supplied by Videolar was used as matrix.

2.2. Chemical Treatment of Fibers

2.2.1. Alkaline Treatment

Untreated sugarcane bagasse fibers (UBF) (100g) were pre-treated with 1 L of an alkaline solution containing 10 g sodium hydroxide (1% w/v), for an hour under constant stirring at room temperature. Once the time of treatment was reached, the solution was filtered in a vacuum filter and the fibers were washed with distilled water until a neutral pH was attained. Then, the fibers were dried in an oven at 50°C for 24 hours.

2.2.2. Bleaching

The alkali-treated fibers (ABF) (24 g) were bleached with 200 mL of a solution containing 1 mL of glacial acetic acid and 3 g of sodium chlorite (80%). This solution was stirred for 2 hours at 70°C, then bleached fibers were mixed with a solution of 250 mL of distilled water and 250 mL of NaOH (1.5 M) during 20 minutes at 50°C. After each treatment the fibers were washed by vacuum filtration with distilled water until a neutral pH was reached. Finally, the bleached fibers (BBF) were dried in an oven at 50 ºC for 24 hours.

2.3. Composites Preparation

Treated fibers (ABF and BBF) were mixed with the polymeric matrix (HIPS) using a thermokinetic mixer model MH-50H, with follow conditions: 5250 rpm in composition of 10, 20 and 30 wt% of the fibers. After mixing, the composites were dried and ground in a mill, model RONE, placed in an injector chamber at 200°C and heated at a 2ºC/min rate. The melted material was injected in a pre-warm mold (210°C) in order to obtain tensile and flexural specimens.

2.4. Thermogravimetric Analysis (TGA)

In the thermogravimetric analysis, samples were analyzed with a Seiko (TGA-50) thermal balance with a heating rate of 10 °C min-1, in a nitrogen flow of 60 mL/min, ranging from 50 to 600 °C, using approximately 10 mg of each sample. The analysis was applied to the fibers in their raw state (UBF) and after treatments (ABF and BBF) and to the composites in order to determine the temperature of degradation and the thermal mass loss in each event. The initial degradation temperature was determined from the first inflection of the baseline in the DTG curve based on the ASTM E2550.

2.5. Optical Microscopy

For the characterization based on the quantitative microscopy evaluation, a tensile test specimen of each manufactured composite and pure HIPS was chosen. Four samples (15 mm x 10 mm) were cut from each specimen for distribution analysis and morphology of the fibers dispersed in the matrix.

Afterwards, samples were embedded in epoxy resin and then polished with a speed of 250 rpm for 10 minutes, with diamond pastes of 3 and 9 um, with suitable webs for each solution. To conclude five more minutes with a solution of colloidal silica (PB-U) velvet cloth.

To analyze the morphology of fibers, images were obtained in an optical microscope on a dark field illumination of the entire sample surface; captured at a magnification of 50x with a Spot Insight QE Camera, associated with a Nikon Epiphot Model 200 optical microscope.

The images were digitally processed using *NIH Image J software*, public domain, following steps to convert to grayscale. A filter via Fourier transform (*FFT/Bandpass filter*) was applied, with the use of

thresholding (*Threshold*), resulting in a binary image, together with the application of the filter *binary/fill holes* and fiber analysis of the particles through the *Set Measurements* tool.

To obtain values of the dimensions and the shape parameter of each analyzed particle, the *Measurement Set/Fit Ellipse* and the *Set Measurement/Shape Descriptors* features were applied to each binary image, which measures the diameters (maximum and minimum) and the circularity of each particle.

Circularity values were calculated according to Equation (1). Results range from 0-1; the closer to 1 the greater the particle circularity

$$\text{Circularity} = (4^* \pi * \text{area})/ (\text{perimeter})^2 \quad (1)$$

2.6. Mechanical Properties

Five specimens of each composites and pure HIPS were analyzed in a Shimadzu testing machine (model AG-X). Tensile tests were carried out according to ASTM D638 (Type I) standard with a crosshead speed of 5 mm/min, using 50 kN load cell without extensometer. Flexural tests were carried out in accordance with ASTM D790 standard and follow procedure A: ratio L/d (where L = distance between beam) equal to 16, test speed of 1.4 mm/min, and with load cell of 5 kN. Flexural strength and modulus were calculated.

Tensile strength, elongation, tensile modulus, flexural strength and flexural modulus were calculated by the software *Trapezium X*, provided by Shimadzu.

2.7. Scanning Electron Microscopy (SEM)

Intact composites fracture surface, after tensile tests, were analyzed with a JEOL JSM5310 scanning electron microscope with a tungsten filament operating at 20 kV, using a low vacuum technique and working distance of 12 mm. All samples were coated with gold to make them conductive before the SEM analysis.

3. RESULTS AND DISCUSSION

3.1. Thermal Characterization

The TGA and DTG curves for sugarcane bagasse fibers, HIPS and its composites with ABF and BBF are presented in Figure **1**.

The decomposition of bagasse fibers, as described at Table **1**, is characterized by three distinct stages,

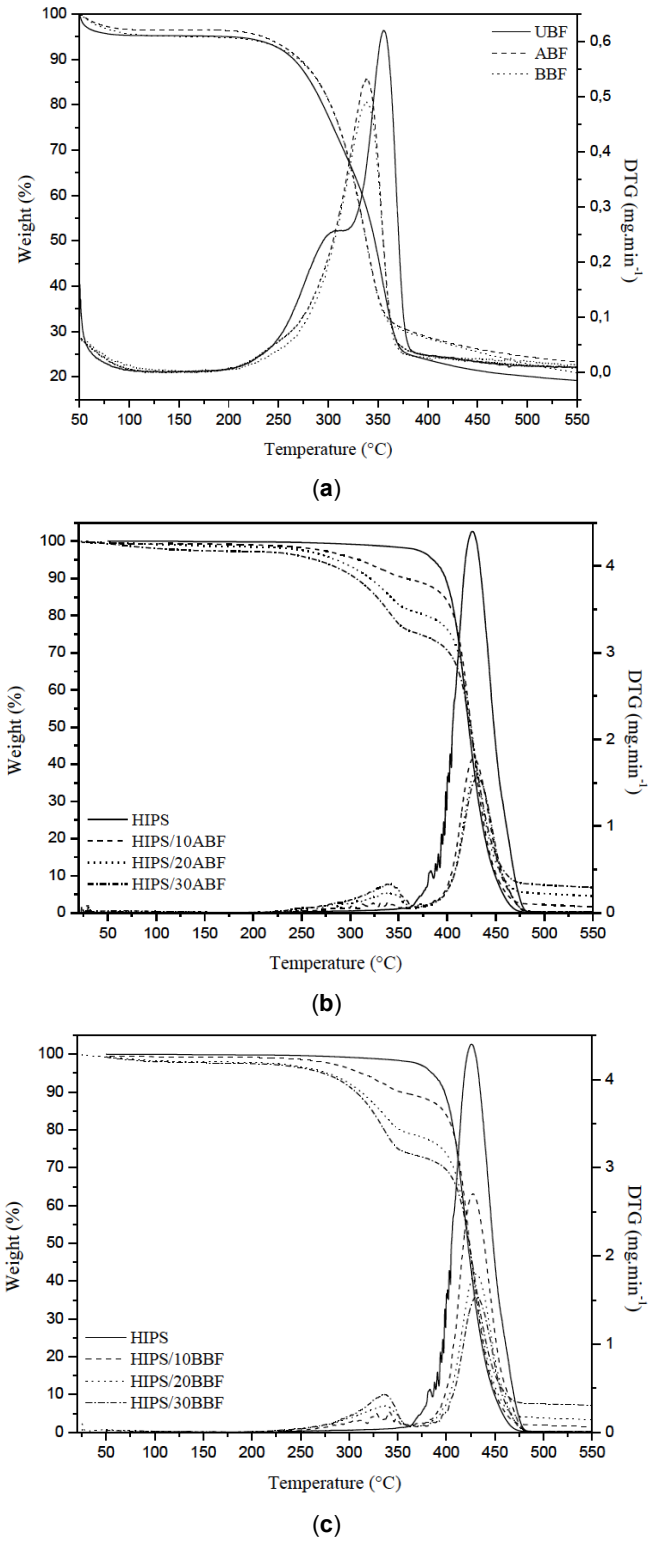

Figure 1: TGA and DTG curves for (**a**) Bagasse fibers (UBF, ABF and BBF), (**b**) HIPS and its composites with ABF and (**c**) HIPS and its composites with BBF.

which are observed in a better way from the DTG curves (Figure **1a**). The first stage, due to adsorbed moisture content, occurred below 100°C [33, 34]. The second one, a smal hump between 200°C and 325°C,

Table 1: **Thermogravimetric Results of HIPS and its Composites Reinforced with Sugarcane Bagasse Fibers**

Samples	ΔT °C	T_{peak} °C	T_{onset} °C	Weight loss (%)	Residue at 550 °C (%)
UBF	50-100	50	200	4.7	
	200-325	304		29.8	18.7
	325-400	355		41.5	
ABF	50-100	55	210	3.4	22.6
	200-400	338		67.5	
BBF	50-100	55	210	4.4	19.8
	200-400	338		66.3	
HIPS	300-500	426	350	99.0	0.1
HIPS/10ABF	250-375	340	250	9.6	1.3
	375-500	430	375	86.5	
HIPS/20ABF	250-375	340	250	17.3	4.2
	375-500	430	375	75.1	
HIPS/30ABF	250-375	340	250	21.3	6.5
	375-500	430	375	67.1	
HIPS/10BBF	250-375	336	250	10.1	1.2
	375-500	426	375	86.7	
HIPS/20BBF	250-375	336	250	18.6	2.9
	375-500	432	375	74.3	
HIPS/30BBF	250-375	336	250	23.6	6.9
	375-500	432	375	65.4	

is associated to the decomposition of hemicellulose, pectin and part of lignin [35, 36]. This peak, more evident for UBF, became less intense for ABF and also imperceptible for BBF due to the removal of some amorphous and non-cellulosic constituents [36].

The third and most important stage (300°C until approximately 400°C), with peak at 355°C and 338°C, for untreated and treated (ABF and BBF) fibers respectively, due to the weight loss promoted by cellulose thermal decomposition through reactions of dehydration, depolymerization, and decomposition of glycoside units [35], was shifted to low temperatures for treated fibers due to the more cellulose exposure. The shifting and narrowing of the peaks for treated fibers is also an indicative of a more homogeneous fibers chemical composition. In addition, the initial degradation temperature for ABF and BBF was 10°C higher than UBF, indicating that chemical treatment are able to improve the fibers thermal stability.

Between ABF and BBF no siginificant difference was observed, confirming the little influence of bleaching treatment on the fibers thermal properties regarding alkaline treatment.

HIPS is thermally stable in a temperature ranging between 50°C and 300°C. From this temperature, the thermal decomposition of HIPS started in a single stage until 500°C, as observed in TGA and DTG curves (Figure **1b** and **c**). The same behavior was observed for Agung *et al*. [37] which relates that the weight loss process of pure HIPS presented a transition temperature between 348.88°C and 468.83°C.

With the addition of ABF and BBF, there was a decrease in the material thermal stability, and the degradation process started to occur in two distinct stages. The first one, associated with decomposition of fibers constituents, occurred between 250°C and 375°C for all composites and the second one, between 375°C and 500°C, was associated with the HIPS decomposition.

It is important to highlight that the weight loss in the first stage, as described at Table **1**, was close to the fiber amount percentages added in composites.

Analyzing Table **1**, it was possible to observe that the addition of ABF and BBF in the polymeric matrix promoted an increase of residue amount at the end of the thermal degradation, according to the amount of added fiber. This can be explained because such fibers have constituents that do not suffer thermal decomposition until 550°C due to the presence of aromatic groups, as can be observed in the thermogravimetric curves of bagasse fibers in Figure **1a**.

3.2. Morphology of Fibers in the Composites

From images obtained by optical microscopy analysis, it was possible to qualitatively evaluate fibers distribution in the matrix according to the amount and type of reinforcement.

Figure **2** shows images obtained by the microscopy analysis of each composites surface. Regarding those images, with the increase of fibers volume in the matrix, the presence of defects become more frequent, because many fibers are pulled during polishing, leaving cavities on the material surface.

It was also possible to verify the presence of particles with circular shape and also elementary fibers which are according to Le Moigne *et al*. [32], a single long plant cell. Bundles, which represents more than one elementary fibers together, were also observed. It is important to highlight that although the fibers have been sieved, longer fibers (elementary fibers) with small diameters passed through the sieve in the same way that the smaller and circular particles fibers did.

Le Moigne *et al*. [32] in the study of polypropylene composites reinforced with flax, sisal and wheat straw fibers, also observed this variation in fibers size and morphology.

A quantitative analysis of size and shape particles distribution was performed using the image processing, as described in the methodology section.

From the surface microscopic analysis of the composites reinforced with 10, 20 and 30 wt% of bagasse fibers, it was possible to determine fibers morphology by measuring their circularity. A processing routine in which an elliptic model was applied to determine the longest ellipse axis as the fiber length and the minor ellipse axis as its width, was used.

To obtain these values, it was carried out a scan in the samples in order to acquire about 20 images of four samples entire surface from each tensile specimen. The average values of circularity, length and width are shown in Table **2**.

As the matrix HIPS has micro-particles of polybutadiene, in order not to considered these particles as fibers, the same routine image processing

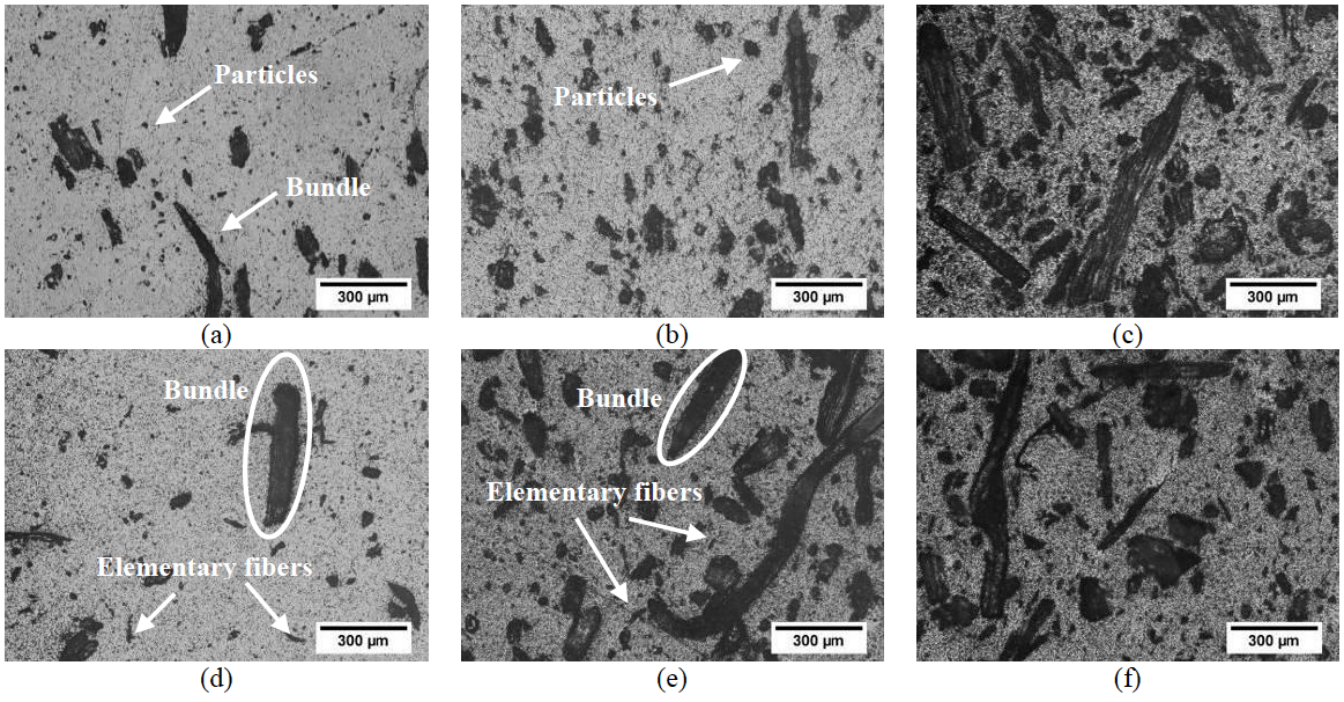

Figure 2: Optical micrograph of composite polished surface: (**a**) HIPS/10ABF, (**b**) HIPS/20ABF, (**c**) HIPS/30ABF, (**d**) HIPS/10BBF, (**e**) HIPS/20BBF e (**f**) HIPS/30BBF.

was performed for pure HIPS samples. Afterwards, it was possible to separate rubber particles and the matrix defect from circular fibers.

Table 2: Dimensional Characteristic of HIPS and its Composites Reinforced with Sugarcane Bagasse Fibers (ABF and BBF)

Samples	Length (μm)	Width (μm)	Circularity
HIPS	2.00	1.04	0.73
HIPS/ABF	17.78	10.01	0.81
HIPS/BBF	19.69	10.48	0.78

From data analyses the data in Table **2**, it was not observed a definite relationship between dimensions and shape of fibers with a chemical treatment. Generally, fibers added to composites presented dimensions between 10 and 19 μm with circularity close to 0.8.

Through comparison between these values with the images in Figure **2**, it was possible to observe fibers with length up to 100 μm. However, the amount of these particles present in the composite was very small regarding the amount of fibers with smaller dimensions. A representative example of the size fibers distribution is presented in the histogram of Figure **3**.

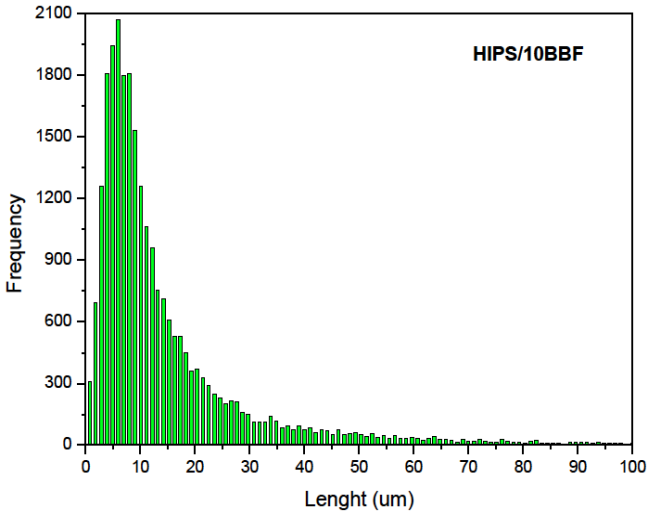

Figure 3: Histogram distribution values of the length fibers in the composites HIPS/ 10BBF.

With the histogram analysis, it was possible to verify that particles amount which present length shorter than 2 μm, size of polybutadiene particles, it was relatively small compared to particles amount with length between 5 and 20 μm. The same behavior was noted in the histograms with width values.

3.3. Composites Mechanical Properties

The effect of addition fibers into the polymeric matrix can be seen through the mechanical properties of HIPS and its composites presented in Table **3**.

Analyzing the composite with ABF and BBF, it was observed that addition of fibers did not cause significant changes in the mechanical properties of the composite until the maximum tensile strength, but contributed to an expressive reduction in the elongation at break.

With the increase in fibers volume, higher values of tensile modulus and tensile strength with a significant reduction in the elongation at break were observed.

This phenomenon was associated with the fact that further reinforcement promoted a reduction in the material ductility. Thus, the composite brittle nature was higher with fiber volume increasing in accordance with results reported in literature [7].

No significant variations were observed in properties obtained from tensile tests from composites reinforced with ABF from those reinforced with BBF.

HIPS composites reinforced with 40 wt% of sugar palm fibers treated with alkaline solution (concentration at 4% w/v) presented increases of about 10% and 7% in tensile strength and tensile modulus, respectively, compared to pure HIPS [38].

In the present study, addition of 30 wt% of alkali treated bagasse fibers (alkali concentration at 10% w/v) resulted in tensile strength and tensile modulus increase in about 20% and 97%, respectively.

To better understand results obtained in the tensile tests, fractured samples were investigated with a scanning electron microscopy (SEM). Fractured surfaces micrographs of composites are presented in Figure **4**.

From micrographs of composites with ABF, it can be seen more evidence of some defects such as pull-out, voids and lack of adhesion between fiber/matrix. On the other hand, a slight improvement in adhesion was observed for composites with BBF.

The SEM analysis shows that the alkaline treatment removed part of fibers amorphous material and, therefore, caused an increase in the surface roughness. The bleaching treatment promoted a partial

Table 3: Mechanical Properties of HIPS and its Composites with ABF and BBF

Samples	Tensile Properties				Flexural Properties	
	Elongation ($\sigma_{ultimate}$) (%)	Tensile Strength (MPa)	Elongation at break (%)	Tensile Modulus (GPa)	Flexural Strength (MPa)	Flexural Modulus (GPa)
HIPS	3.1 ±0.1	24.6 ±0.1	26.2 ±8.1	3.0 ±0.1	42.0 ±0.1	1.74 ±0.05
HIPS/10ABF	2.9 ±0.1	25.1 ±0.2	7.7 ±0.8	3.5 ±0.1	43.3 ±0.8	2.60 ±0.20
HIPS/20ABF	3.0 ±0.2	27.7 ±0.4	4.1 ±0.4	4.8 ±0.2	55.7 ±0.8	4.22 ±0.06
HIPS/30ABF	3.0 ±0.2	29.6 ±0.7	3.2 ±0.2	5.9 ±0.5	49.9 ±0.5	3.80 ±0.05
HIPS/10BBF	2.8 ±0.1	24.1 ±0.1	8.4 ±0.5	3.4 ±0.1	51.9 ±1.0	4.16 ±0.02
HIPS/20BBF	2.8 ±0.3	26.3 ±0.3	4.9 ±0.5	4.6 ±0.1	56.5 ±0.7	4.24 ±0.05
HIPS/30BBF	3.4 ±0.4	27.4 ±1.1	4.0 ±0.8	5.3 ±0.3	57.8 ±1.3	5.06 ±0.03

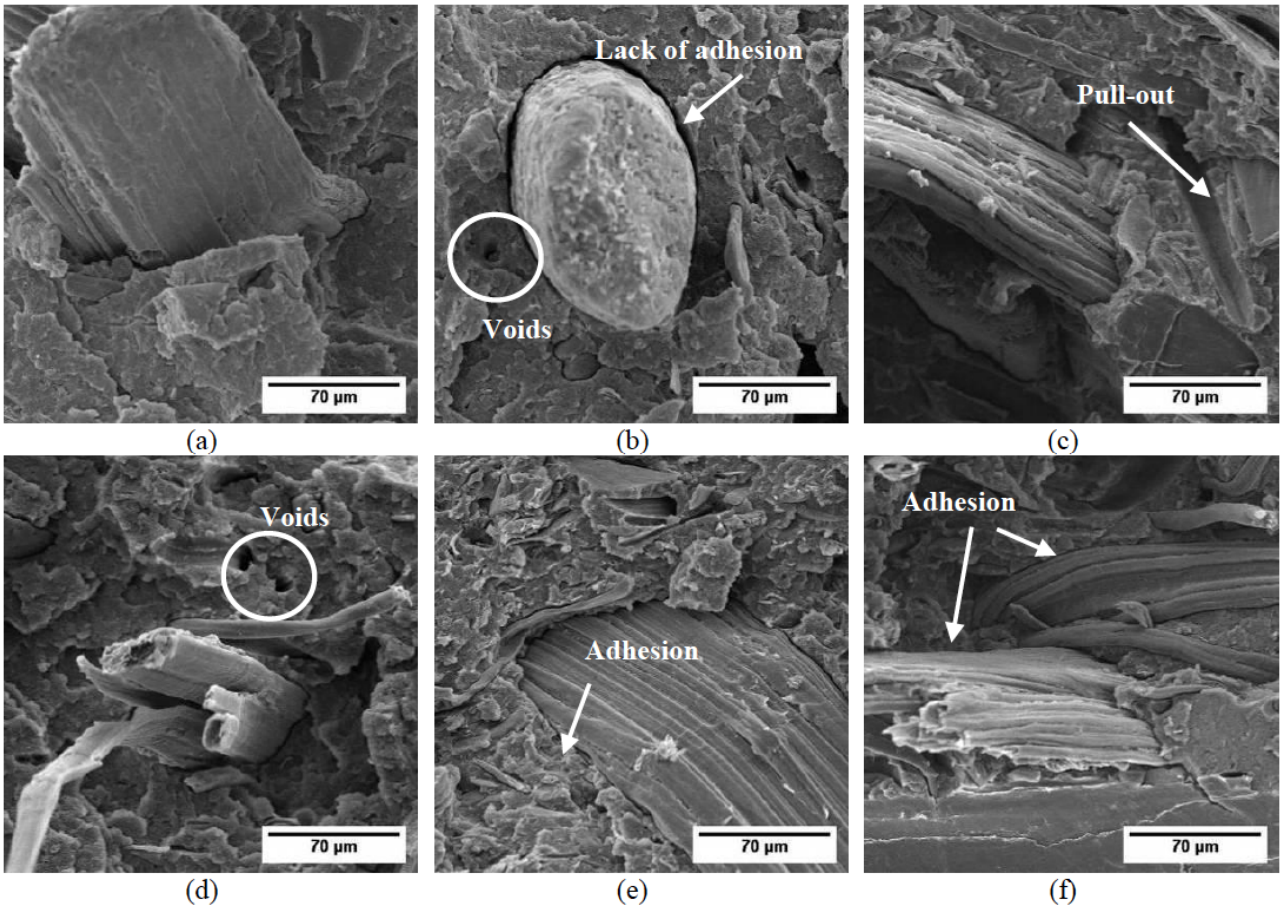

Figure 4: SEM of (**a**) HIPS/10ABF, (**b**) HIPS/20ABF, (**c**) HIPS/30ABF, (**d**) HIPS/10BBF, (**e**) HIPS/20BBF, (**f**) HIPS/30BBF.

disintegration of bagasse fibers, which increased fibers roughness and then improved fiber/matrix adhesion. Similar results were observed by Brígida *et al*. [16] for green coconut fibers treated with NaOCl/NaOH.

Composites manufactured in the present work were also evaluated for flexural tests, and results obtained are shown in Table **3**.

Analyzing flexural tests dates, it was noted an increase in the values of flexural strength and mainly in the flexural modulus of all composites regarding the pure HIPS, according to fibers amount and type added to the matrix and, different from it was observed for tensile tests, the most effective reinforcement was the BBF.

With the addition of 30 wt% of ABF, it was possible to achieve an increase of 118% in values of flexural modulus in relation to the pure HIPS; on the other hand, for composites with 30 wt% of BBF, due to their higher percentual amount of cellulose and the fiber morphology, the increase was about 191%.

Flexural modulus quantifies the material stiffness; hence in applications where the rigidity is more important than ductility, addition of fibers into the HIPS is extremely feasible.

Fibers amount and type added to the matrix also influence the composite flexural behavior. Flexural strength and modulus values increase for higher volume of fibers, except for those reinforced with 30 wt% of ABF, which is also in agreement with the analysis by optical microscopy, where it can be seen that the ABF presented dimensions (length and width) smaller than the BBF.

This reduction can be explained based on the fact that the bleaching treatment caused a reduction in the fiber length, as observed by the surface composite optical microscopy. As the volume of fibers in the composite increased, they began to act as stress concentrators instead of as a reinforcing agent. According to Sawpan *et al*. [13], for composites with short fiber the ends of fibers can act as points of stress concentration, thus acting as regions of cracks initiation.

According to Aziz and Ansell [39] composites flexural modulus depend on the type of chemical bond between the fiber and the matrix. Bleaching of bagasse fibers improves the chemical interaction between matrix and fibers, therefore being responsible for an increase in flexural modulus values, about 139% and 191% for composites with 10 wt% and 30 wt% of fibers, respectively.

4. CONCLUSIONS

With the addition of natural fiber into a HIPS, it was possible to obtain a material with 30 wt% less polymer and with thermal and mechanical properties feasible for certain applications of HIPS, which tensile modulus is more important that ductility, for example.

With respect to the volume of added fibers it was observed that the higher the reinforcement percentage (ABF and BBF) the better the mechanical properties of the composites. However did not change their thermal stability, since the composite reinforced with 30 wt% of fibers had the same initial temperature degradation regarding the other composites.

Analyzing the effect of chemical treatments, it was concluded that the bleaching treatment, although it has not provided an improvement in the tensile and thermal properties of the composites, provided a considerably increased in the values of flexural strength and flexural modulus. In this way it is possible to conclude that the bleaching treatment it is not necessary for tensile applications; however, became very important for materials that will be required mechanically in flexion.

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