

The incorporation of untreated and alkali-treated banana fiber in SEBS composites

Leticia Cuebas¹, José Armando Bertolini Neto², Renata Tâmara Pereira de Barros², Alexandre Oka Thomaz Cordeiro², Derval dos Santos Rosa¹ and Cristiane Reis Martins^{2*} 

¹*Centro de Engenharia, Modelagem e Ciências Sociais Aplicadas – CECS, Universidade Federal do ABC – UFABC, Santo André, SP, Brasil*

²*Instituto de Ciências Ambientais, Químicas e Farmacêuticas – ICAQF, Universidade Federal de São Paulo – UNIFESP, Diadema, SP, Brasil*

*cr.martins@unifesp.br

Abstract

In this work, banana fiber was used as reinforcement for the preparation of a thermoplastic elastomer composite (TPE). Few studies are exploring the natural fiber incorporation on TPEs, with no one using banana fiber. The fiber was extracted from banana pseudostem and modified with an alkaline solution. The untreated and treated banana fibers were incorporated in 2%, 5%, and 10% in SEBS. The mixture was performed in a thermokinetic mixer (K-Mixer) and plates prepared by compression molding. The composites were characterized by Scanning Electron Microscopic (SEM), tensile testing, mechanical dynamical analysis (DMA). It can be observed that the samples with untreated fibers presented a higher tensile strength, except for the incorporation of 5% of BF. Young's modulus increase as the fiber's incorporation grows, indicating greater rigidity of the composite. It was found that the chemically treated banana fiber composites, e.g., TPE/TBF5 and TPE/TBF10, shows a 15.4% and 22.2% higher elongation.

Keywords: *banana fiber, composite, SEBS, thermoplastic elastomer, thermokinetic-mixer.*

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1. Introduction

Natural fibers are growing increase importance as reinforcing materials in composites due to some advantages, such as low density, inexpensive, no toxicity, biodegradable in nature, good mechanical properties, and provide a market reputation as an eco-friendly material^[1]. Recent studies using natural fiber as reinforced material in rubbers offer an attractive, easy, and economic friendly approach to originate commercially feasible natural rubber composite^[1-3].

Hashim et al.^[4] investigate the incorporation of mengkuang (*Pandanus tectorius*) leaf fiber in blends of ethylene-vinyl acetate and natural rubber, and ethylene-vinyl acetate and epoxidized natural rubber. The authors showed better filler-matrix interaction between ethylene-vinyl acetate and epoxidized natural rubber with fiber filler causing stronger tensile properties than non-epoxidized rubber blend. Rice straw fiber was incorporated by Paran et al.^[5] as a filler in nitrile butadiene rubber (NBR) and poly (vinyl chloride) (PVC) thermoplastic elastomer also containing an organoclay. Their work shows the higher rice straw concentration leads to an increase in thermal stability and higher thermal decomposition temperature. This improvement is believed that the physical structure of rice straw can act as an obstacle for the volatile products resulted from the thermal decomposition of the polymer. Miedzianowska et al.^[3] evaluated ethylene-octane

copolymer in the presence of several fibers, as wheat, oat, rye, barley, and triticale fiber as well compared the size of particles as a filler. The results demonstrated that the smallest particle size filler was characterized as having the lowest viscosity, and the minimum torque increased with the increasing content of lignocellulosic material in the composite. The addition of the fiber increased the stiffness of the composites, as evidenced by the increase in the maximum torque value in compared to pure ethylene-octene rubber. They appointed the type of fiber, fiber dispersion, porosity, and interfacial strength as the main factors affecting mechanical performance.

Despite the improvements offered by natural fibers, incompatibility between the polymer matrix and fiber surface has been identified as a major difficulty in processing polymer composites containing natural fibers^[6]. Alkaline treatment, or mercerization, is one of the chemical treatments of natural fibers used to modify the network structure, disrupting hydrogen bonds and increasing surface roughness. In this treatment, lignin, hemicellulose, and waxes are removed from the fiber, increasing fiber density. Many studies have explored the alkali treatment on natural fibers and the incorporation in polymer matrices. Yantaboot et al.^[7] reported the improvement of the adhesion of pineapple leaf

fiber into natural rubber. They used in their work untreated fiber and 10% sodium hydroxide (w/v) treated fiber during 30 minutes. The moduli strains of the composites containing treated fiber are higher than those containing untreated fiber. Pineapple leaf fiber treated with 10% NaOH was compared with aramid fiber (Kevlar) in reinforcement of natural rubber^[8]. At 25 °C, as fiber content increases, Kevlar fiber has a slightly greater effect than pineapple leaf. As the temperature increased to 60°C, 2 and 5 phr content, for both fibers, the effect of both fibers remains similar. However, at a fiber content of 10 phr, natural fiber demonstrates a much greater effect that does Kevlar becoming a good and cheap alternative for reinforcement filler.

In the literature, the concentration of alkali agents (NaOH, KOH, etc.) and the treatment time varies in a wide range from 0.03 wt% to 40 wt% and from a few minutes to 48 h^[9]. The adjust of these parameters is essential to remove lignin, hemicellulose, and waxes partially, and if the treatment parameters are not optimized, the mercerization can cause fiber defibrillation and pore formation^[10]. In Cai et al. work^[11], abaca fibers was treated with 5, 10, and 15% NaOH solution and its effects on the interfacial adhesion. The results showed that mild treatment such as 5 wt % NaOH for 2 hours improves the fiber-matrix interfacial strength which ensures the good mechanical properties of developed composites. Oushabi et al.^[12] investigated the effect of mild alkali treatment on palm fibers surface reinforced polyurethane composites. The treatment was carried out with NaOH, with low to high concentrations; 0 wt%, 2 wt%, 5 wt% and 10 wt% for 1 hour. The experimental results showed the optimal alkali concentration was reported at 5 wt% in terms of reduction of the amorphous parts of the fibers.

Thermoplastic elastomer (TPE) composites have attracted much attention because of their advantages over conventional elastomeric materials^[4]. This type of material can be processed and recycled and replacing vulcanized rubber and PVC. They can also be processed using conventional thermoplastic processing equipment^[13]. The copolymer styrene-(ethylene butylene)-styrene (SEBS) represents an important type of TPE. However, the systematic study based on the hybridized reinforcement of SEBS composites with natural fiber is very rare. Few studies have been done about the reinforcement of SEBS with natural fibers such as oil palm fiber^[14], hemp fiber^[15] and mango wood fiber^[16], pineapple leaf fiber^[17,18], curaua fiber^[19], and wood fiber^[20-22].

Banana harvest generates residue from the leaves, pseudostem, and roots. After the banana is harvested, the pseudostem is discarded, this residue is fibrous in nature and enriched in strands with cellulose content^[23]. Balaji et al.^[24] prepared hybrid polymer composites of epoxy reinforced with sisal, banana, coir, and a mix of the three fibers. The authors observed that the tensile strength of the pure epoxy composite is much lower than all composites. The composite with banana fiber presented higher impact strength, 1.31 kJ.m⁻² because the fibers have introduced some brittleness as an increase in hardness, which led to a decrease in impact strength. They found that the tensile strength of the banana fiber is 68 MPa and 1.0 to 3.5% of elongation. Zaki et al.^[19] found 20.18 MPa for tensile strength in epoxy resin with 10% of banana fiber treated with 6 wt% NaOH. Polyester-banana

fiber composite prepared Kumari et al.^[25] showed a tensile strength of 20.1 for untreated fiber and 26.0 for treated fiber.

Given the possible damage to the fiber caused by high concentrations of alkaline agents and the increasing interest in natural fibers as reinforcing fillers in rubbers, the main goal of this present study was to develop a natural fiber-based SEBS composite using banana fiber (BF), untreated and mild alkaline treated, as filler. The influence of the BF content in thermoplastic elastomer (TPE) composites and their chemical treatment was examined in terms of morphological, mechanical, and dynamic mechanical properties.

2. Materials and Methods

2.1 Materials

Banana pseudostem of *Musa sapientum* was obtained from Agência Paulista de Tecnologia do Agronegócio (APTA, Registro, São Paulo, Brazil) and from Cooperativa de Artesãos de Miracatu (BANARTE, Vale do Ribeira, São Paulo, Brazil). The matrix was a thermoplastic elastomer (SEBS Fortiprene TPE-7140) donated by FCC[®]. NaOH (purity ≥99%) was supplied by Vetec[®].

2.2 Methods

2.2.1 Fibers extraction from banana pseudostem

Pseudostem was cut, and layers manually defibrillated. Layers were dried at 55°C in a hot air circulating oven. Fibers extraction from pseudostem was performed by the following method. The layers were immersed in distilled water for two days, were shredded with a wire brush, washed with water, and dried in an air oven until the complete drying, obtaining banana fiber (BF). Fibers were ground in a knife mill and sieved to obtain particles of about 87 µm in size.

2.2.2 Alkali treatment

Figure 1 shows the banana fibers after extraction (untreated and treatment) used for the preparation of TPE-based composites. Untreated banana fiber (UTBF) was subject to alkali treatment by soaking in 5% sodium hydroxide solution (w/v) for 1 hour and washed with water up to neutral pH. Treated fiber (TBF) was dried in an oven at 55 °C and sieved (Ø 85µm).

2.2.3 Preparation of thermoplastic elastomer/banana fiber composite

Figure 2 describes the experimental aspects used for processing the composites. TPE/BF composites were made from untreated banana fiber (UTBF) and treated banana fibers (TBF). Composites were processed in a K-Mixer (MH 50H; MH Equip. Ltda., Unifesp Diadema, São Paulo, Brazil) and plates prepared by compression molding.

Appropriate quantities of thermoplastic elastomer (TPE) polymer and banana fiber were pre-weighed to the desired loading, combined, and added into the K-Mixer. The contents of banana fiber in the TPE/UTBF and TPE/TBF composites used were 2, 5, and 10 wt%, giving the samples names TPE/UTBF2, TPE/UTBF5 and TPE/UTBF10 for untreated fibers, and TPE/TBF2, TPE/TBF5 and TPE/TBF10 for treated fibers, respectively. In the process, the increase of amperage

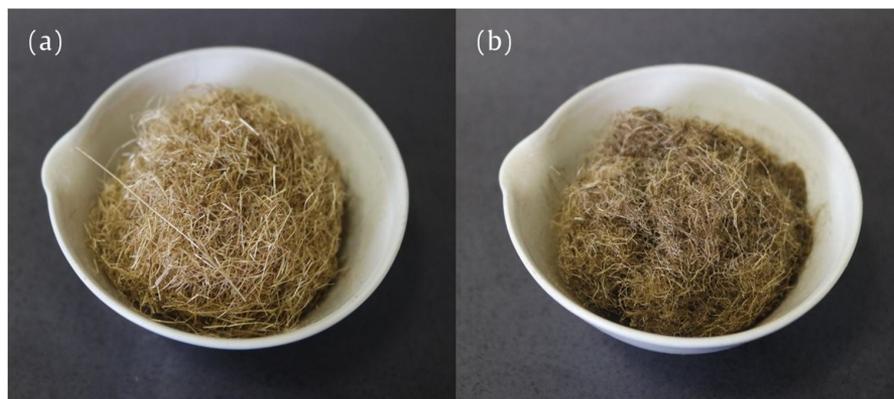


Figure 1. Banana fibers after extraction: (a) untreated and (b) after alkali treatment.

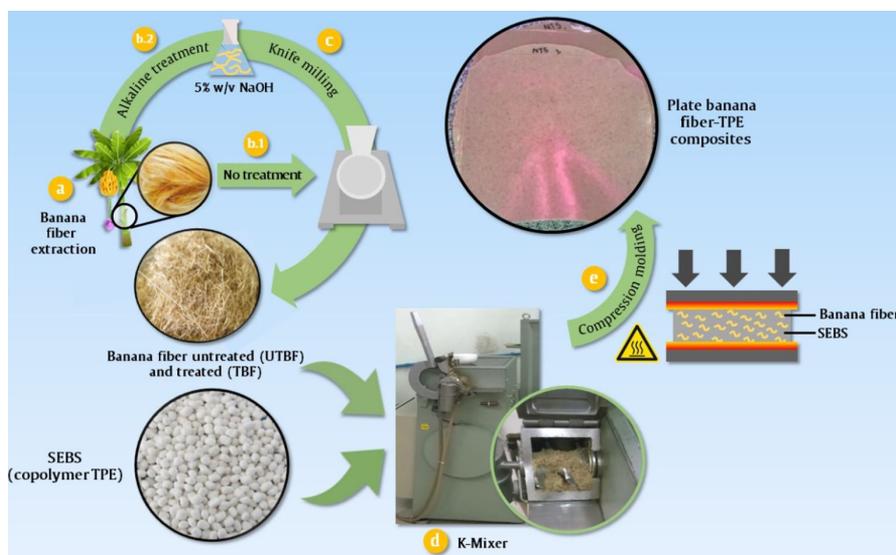


Figure 2. Composites preparation: (a) Banana fiber extraction, (b.1) Banana fiber untreated and (b.2) alkali-treated, (c) Knife milling of fibers, (d) K-Mixer processing, (e) Production of plates banana fiber-TPE composites in the compression molding.

is indicative of the polymer melt and ending of processing. Then, each process occurred by 65 s at 1750 rpm when it is possible to hear when fluxing of the materials occurred followed at 3550 rpm for complete mixing with BF. Further, plates composites with dimensions of 120 x 80 x 2mm were prepared in compression molding equipment (Hidro MH-P8-MT; MH Equip. Ltda., Unifesp Diadema, São Paulo, Brazil) using molds at 160 °C for 3 minutes and 8 bar pressure, producing 2.0 mm thick plates. After the plates were prepared, the specimens were stamped using a manual press (Tecnal Equipamentos Científicos Ltda., São Paulo, Brazil) and specimens were obtained for application of mechanical tests.

2.3 Characterization

2.3.1 Chemical composition analysis

Compositional of fiber contents (extractives, cellulose, and lignin) was carried out with some modifications according to TAPPI's protocols described in^[26-28]. Holocellulose was determined according to the method described by Wise et al.^[29]. The hemicellulose fraction was calculated as the difference

between the holocellulose and α -cellulose content. The lignin fraction was calculated as the sum of non-soluble and soluble lignin. The results presented are the mean and standard deviations of three replicate determinations of each sample.

2.3.2 Fourier transform infrared spectroscopy (FT-IR)

FTIR is performed to verify chemical compositional change after chemical treatment. Five percent of both untreated and treated banana fibers were carried out dispersing this powdered BF on KBr pellets and using a Shimadzu spectrophotometer (Model Prestige 21-IR, Unifesp, Diadema, São Paulo, Brazil) with a resolution of 4 cm^{-1} , accumulation of 32 scans and in the range of 4000-500 cm^{-1} . Triplicate data were collected for each sample.

2.3.3 Scanning electron microscopy

Scanning electron microscope (SEM, JEOL JSM, Model 6610 L20, Unifesp, Diadema, São Paulo, Brazil) with an acceleration voltage of 10 kV was used for the study of the morphological behavior of the untreated and chemically modified fiber surface, and the analysis of the composite.

Fracture surface of composites was prepared by placing samples in liquid nitrogen and breaking it. These surfaces were coated with gold coating.

2.3.4 Tensile Testing

Tensile test measurement was carried out according to ASTM Standard D412^[30], and dumbbell-shaped samples (Die D) were cut from the molded sheets presented in the method. Samples were submitted in the universal testing machine (EMIC DL-10000, FATEC, São Paulo, Brazil). A load cell of 5kN was used, and the cross-head speed was maintained at 10 mm/s. Six replicates were made for each sample.

2.3.5 Dynamical mechanical properties

For the determination of dynamic parameters of composites under cyclic external forces, dynamic mechanical analysis (DMA) was done. The dynamic parameters such as storage modulus (E'), loss modulus (E''), and damping factor ($\tan \delta$) are temperature dependent and provide information about interfacial bonding between the reinforced fiber and polymer matrix of composite material. Specimens with a nominal size of $30 \times 5 \times 1$ mm were cut from the compression molded plates and measures were carried out on a TA Instruments DMA (model Q800, UFABC, Santo André, São Paulo, Brazil) with a heating rate of 3 °C/min, in strain mode. Duplicate samples were scanned over a temperature range of -100 to 120 °C at a constant frequency of 1 Hz. Loss modulus and loss tangent ($\tan \delta$) were measured and analyzed as a function of temperature.

3. Results and Discussions

3.1 Chemical composition

Chemical characterizations of the raw and treated banana fibers were done to determine the cellulose, hemicelluloses, lignin, and ash content. Results are presented in Table 1.

The treatment of natural fibers by sodium hydroxide (NaOH) is widely used to change the cellulosic molecular structure. This provides more access to penetrate chemicals, and hydrophilic hydroxyl groups are reduced and increase the fibers moisture resistance property. It also removes a certain portion of hemicelluloses, lignin, pectin, and waxes. As can be seen in Table 1, the alkaline treatment carried out on the BF resulted in a decrease in the amount of lignin and hemicellulose present in about 66.7 and 35.5%, respectively, showing partial removal of these components. These results showed similarities with chemical compositions previously reported for banana fibers, as reported by Gonçalves et al.^[31] that found 9.0% and 3.0% of lignin for untreated and treated banana fibers, as well as 25.1% and 16.1% of hemicellulose for untreated and treated fiber, respectively. Nery and José's work^[32] was also in accordance with the results. These authors reported 5.2% and 3.8% of lignin for untreated and treated banana fiber and 20.9% and 14.1% of hemicellulose.

3.2 Fourier transform infrared spectroscopy

Fourier transform infrared spectroscopy (FTIR) was used to analyze the functional groups present in the untreated and treated fibers. FTIR spectra of the untreated and NaOH treated

Table 1. Lignocellulosic content of untreated and treated banana fibers.

Banana Fiber (BF)	Lignin (%)	Hemicellulose (%)
Untreated (UTBF)	6.3	18.0
Treated (TBF)	2.1	11.6

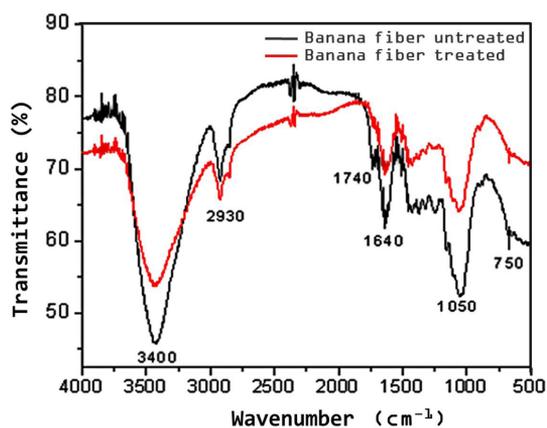


Figure 3. FTIR spectra of banana fiber before (black line) and after alkali treatment (red line).

BF are presented in Figure 3, which seems much similar to banana fiber characterization reported earlier^[33]. From left to right, a band in the region of 3400 cm^{-1} can be observed, indicating the presence of free hydroxyl groups (OH) present in the cellulosic structures in both fibers^[34]. These hydroxyls refer to amorphous cellulose, hemicellulose, and lignin. The decreased presence of this group in the treated fiber shows that after mercerization, the sample became more hydrophobic and abler to interact with the matrix. The peak at 2930 cm^{-1} has been assigned to the C-H stretching vibration from the $-\text{CH}_2$ group of cellulose and hemicellulose^[35]. The peak at 1740 cm^{-1} in both samples corresponds to the carbonyl group ($\text{C}=\text{O}$) of hemicellulose or the ester bond of the carboxylic group of ferulic acid and p-coumeric acid of lignin and hemicellulose^[34]. This peak has a lower intensity for the treated fiber due to the removal of lignin and hemicellulose from banana fibers by alkaline treatment. The band at 1640 cm^{-1} in both samples indicated the presence of $\text{C}=\text{O}$ stretch of the acetyl group of hemicellulose^[36]. The 1050 cm^{-1} stretch present in the spectrum shows the absorbance of the O-C-O stretch present in the cellulose and hemicellulose, and C-O/C-C stretching vibration^[36].

3.4 Scanning electron microscopy

Figure 4 presents the SEM micrographs of the untreated and treated banana fibers. For both treatments, at 2% loading, the appearance of the composite appears homogeneous (Figure 4a and 4b). In untreated banana fibers shown in Figure 4b, c cellulosic structures can be seen outside the matrix suggesting poor interfacial adhesion, which for samples with treated fibers (Figure 4d-f) does not occur.

3.5 Mechanical test

Mechanical test was applied for the untreated and treated banana fibers composites. Figure 5a shows, comparatively, the tensile strength of all compositions. With increasing

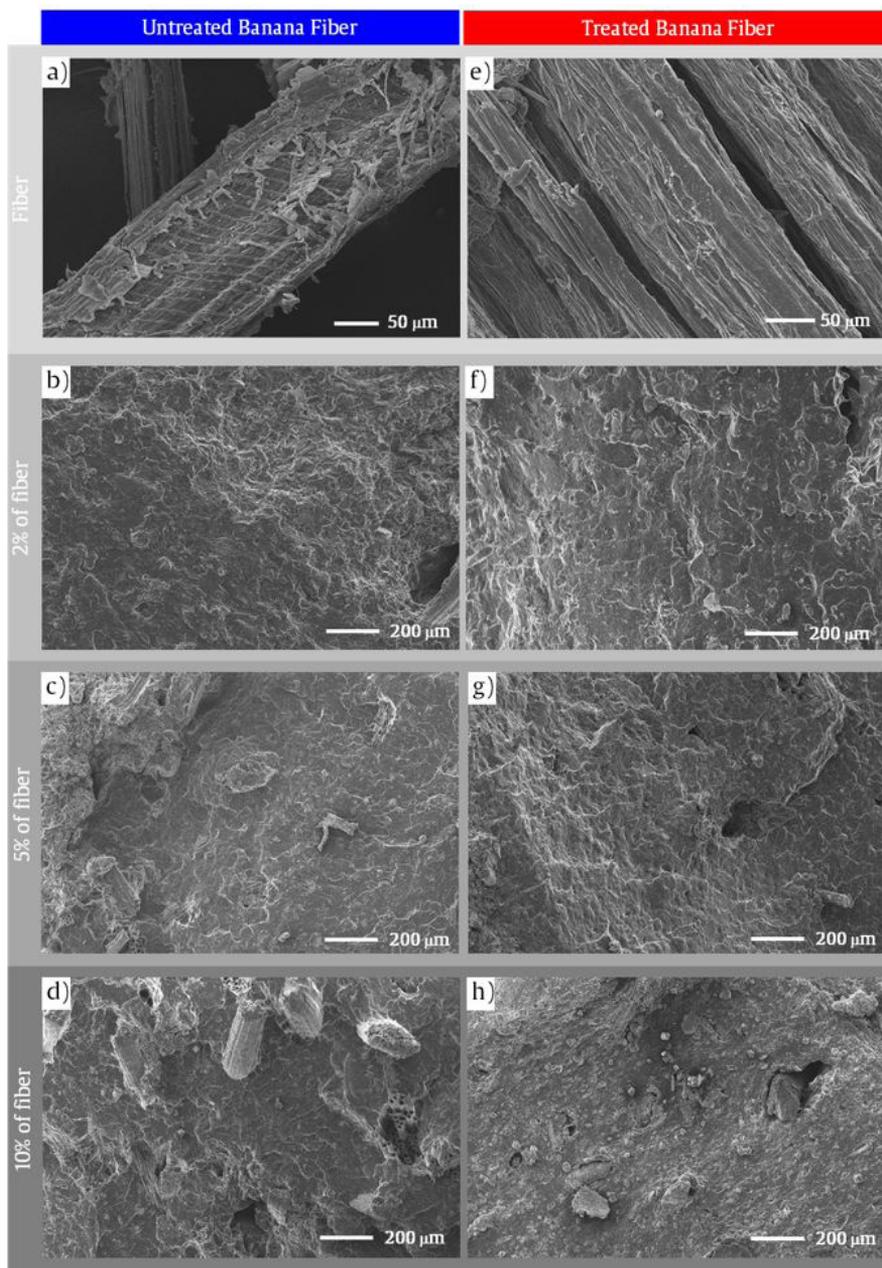


Figure 4. SEM micrographs of (a) untreated banana fiber, (e) alkaline treated banana fiber and, cross-section of SEBS composites with different concentrations of untreated and treated banana fibers: (b) TPE/UTBF2, (c) TPE/UTBF5, (d) TPE/UTBF10, (f) TPE/TBF2, (g) TPE/TBF5 and (h) TPE/TBF10.

incorporation of banana fibers, the tensile strength of TPE/BF composites decreases, indicating that the addition of BF to SEBS decreases the deformation capacity of the material. Comparing the mechanical performance with the fiber treatment, it can be observed that the samples with untreated fibers presented a higher tensile strength, except for the incorporation of 5% of BF, where there was an inversion. This can also be seen in Figure 5b, where Young's modulus increases as the fiber's incorporation grows, indicating greater rigidity of the composite. It is also possible to observe that the non-treatment of the fibers becomes the material slightly more rigid, 8 to 10%, in all compositions. The

greater rigidity of the composite at higher concentrations can be explained by the difficulty in movement imposed by the presence of fibers. Few studies were found about the incorporation of natural fibers in SEBS blends, many of them with polyethylene and no one only with SEBS. Panaitescu et al.^[37] have shown the reinforcement of hemp fibers on polypropylene/SEBS composite. In their work, hemp fibers were treated with a mild condition, 1% NaOH solution during 30 minutes and 1 hour. The soft treatment was chosen because the complete removal of lignin can suppress the action of π electron interactions between SEBS containing an aromatic portion and lignin, which may lead

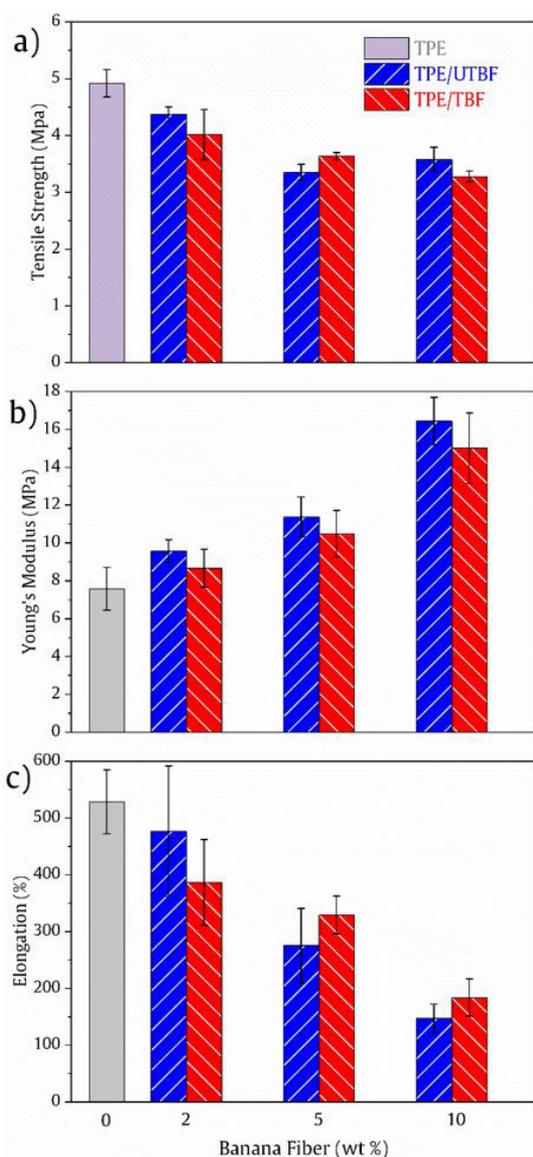


Figure 5. Mechanical behavior of banana fiber-TPE composites. (a) Tensile strength, (b) Young's modulus and (c) elongation at break of TPE/BF composites.

to better interfacial interaction, as shown in Szabó et al. work^[38]. However, in Panaitescu et al. work, composite was formed majority by polypropylene (PP) mixed with 5% of a coupling agent (maleic anhydride grafted polypropylene), 15% of SEBS, and 30% of fiber. Although the increasing of mechanical properties caused by alkaline treatment of fibers was observed in many works with different natural fibers and polymer matrices^[39-41], similar results were found^[42,43]. Besides the alternative explanation of SEBS aromatic groups and the presence of lignin in UTBF, according Adeniyi et al., the fact may be attributed by adhesive effect of matrix bound favorably with the untreated plantain fiber.

Consequently, to Young's modulus, the elongation (Figure 5c) decreases with the increase in the fiber loading. This behavior suggests poor bonding of banana fiber with

the polymer. Except for the 2% sample, the BF treatment showed a longer elongation than the untreated fibers, this may be due to the amount of treated fiber added to the composite that caused a greater specific interaction between the banana fiber and the polymer. It was found that the chemically treated banana fiber composites, i.e., TPE/TBF5 and TPE/TBF10 show a 15.4% and 22.2% higher elongation, respectively, than the corresponding untreated samples. This might be attributed to better fiber-matrix interaction adhesion.

3.6 Dynamic mechanic test of TPE/BF composites

The effect of BF on the viscoelastic behavior of SEBS was investigated by DMA. The loss modulus and tan delta resulting from the dynamic frequency scans of SEBS composites are shown in Figure 6a-d as a function of temperature. Loss modulus shows the maximum value, which corresponds to the viscoelastic behavior of the elastomers when the frequency of the material's internal movements is comparable to the frequency at which the measurement is made. It is seen from Figure 6a-c that the incorporation of untreated BF in SEBS increased the loss modulus of composites between -60°C to -40°C. At a fiber loading of 10% (TPE/UTBF10), the most pronounced effect of the filler was found, 927 MPa of loss modulus, a value 56.5% higher than TPE/UTBF2 and 45.3% greater than TPE/UTBF5, reflecting the increased concentration of fiber with greater capacity to convert mechanical energy into thermal. All treated samples had a loss modulus lower than pure TPE, 278 and 222 MPa, respectively, TPE/TBF2 and TPE/TBF5, TPE/TBF10 obtained a loss modulus similar to TPE, 328 MPa.

Block copolymers such as SEBS have two glass transition temperatures, one for the styrene block and one for the ethylene-butylene block. In Figure 6d the damping properties are expressed by the plot of $\tan \delta$ as a function of temperature. SEBS fiber composites showed two glass transition (T_g), at -50°C referred of ethylene-butylene, and a shoulder peak at 120 °C, probably referred to styrene block. Tjong et al.^[44] found a peak related to ethylene-butylene of SEBS at -35 °C and Hashemi^[45] reported 3 relaxation peaks at -105 °C, -36 °C and 109 °C. According to Tjong et al. the transitions are due to the relaxations of the hard polystyrene domains and soft ethylene-butylene block, respectively, whereas the third transition is caused by the crank-shaft mechanism involving the $-(CH_2)-$ units present in the elastomeric poly(ethylene-butylene) segments^[44]. Compatibility of the fibers with the matrix can be assessed by observing the behavior of the composite over the temperature range. Improvement in interfacial bonding in composites occurs as observed by the lowering in tan delta values between 20 and 60 °C. The higher the damping at the interfaces, the poorer the interface adhesion. In this work, the peak of -105 °C was not found as shown by Kumari^[25], however, the analysis shown in Figure 6 was performed between -100 and 120 °C.

The maximum observed in the loss module in the glass transition region pointed in tan delta curves is due to the high conversion of mechanical energy into heat through the micro-Brownian movements of the main chain segments. Maximum peak corresponds to the maximum dissipation of mechanical energy situation curve.

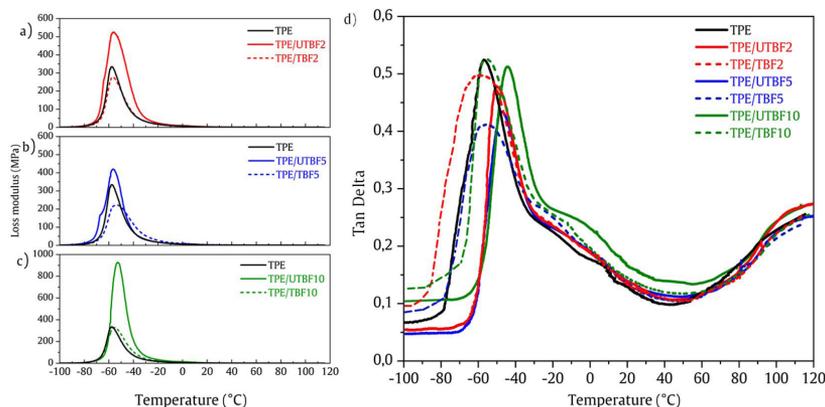


Figure 6. Loss modulus (a-c) and tan delta curves (d) of TPE/BF composites with untreated and treated banana fibers.

4. Conclusions

It was reported that alkali treatment promotes the removal of lignin and hemicellulose from the fiber. The breakage of hydrogen bonds creates many active hydroxyl groups that increase the fiber's hydrophilicity, improving the compatibility of banana fiber with the matrix. It can be observed that the samples with untreated fibers presented a higher tensile strength, except for the incorporation of 5% of BF, where there was observed a reversal pattern. Moreover, the tensile strength of all compositions decreases with the incorporation of fiber, indicating that the addition of BF to SEBS decreases the rigidity of the composite. Hence, we can understand the reason for the increase of Young's modulus of alkali-treated fiber composites for all compositions. It is also possible to observe that the non-treatment of the fibers becomes the material slightly more rigid, 8 to 10%, in all compositions. Finally, based on DMA results of SEBS composites, it is seen the incorporation of untreated BF in SEBS increased the loss modulus of composites between -60°C to -40°C and at a fiber loading of 10% it was obtained the most pronounced effect, 927 MPa of loss modulus, a value 56.5% higher than the 2% and 5% samples, what reflects the increased concentration of fiber with higher capacity to convert mechanical energy into thermal.

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