

## CHEMICAL MODIFICATION EFFECT ON THE MECHANICAL PROPERTIES OF HIPS/ COCONUT FIBER COMPOSITES

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Lignocellulosic fibers from green coconut fruit were treated with alkaline solution (NaOH 10%*m/v*) and then bleached with sodium chlorite (NaClO<sub>2</sub>) and acetic acid (CH<sub>3</sub>COOH). Alkali-treated and bleached fibers were mixed with high impact polystyrene (HIPS) and placed in an injector chamber in order to obtain specimens for tensile tests. Specimens of HIPS/alkali-treated and bleached coconut fiber composites were tested in tensile mode, and the fracture surfaces of the composites were analyzed by scanning electron microscopy. Untreated, alkali-treated, and bleached coconut fibers were analyzed by scanning electron microscopy and X-ray diffraction. Alkaline treatment was effective for removing the extractives and increasing the roughness of surfaces, while the bleaching treatment intensified the effect of alkaline treatment, while increasing the crystallinity index and surface energy of fibers. Results of tensile tests showed that the addition of 30% alkali-treated and bleached fibers reinforcing the HIPS matrix provided considerable changes in the mechanical properties of composites in comparison with the pure HIPS. On the other hand, chemical treatments were not totally effective for improving the adhesion between the fiber and matrix, as was observed in the analysis of the fracture surfaces of composites materials.

*Keywords:* Polymer-matrix composites; Lignocellulosic fiber; Injection moulding; Mechanical properties

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### INTRODUCTION

New environmental legislation as well as consumer pressure has forced the manufacturing industries to search for new materials that can substitute for conventional non-renewable reinforcing materials, such as carbon or glass fibers. On account of this, in recent years, the use of natural fibers including banana, sisal, hemp and flax, jute, coconut, and oil palm have attracted scientists and technologists for applications in consumer goods, low-cost housing, and other civil structures (Jústiz-Smith et al. 2008; Spinacé et al. 2009; El-Taybe 2009). Natural fibers are very attractive for composite materials because of their advantages compared to synthetic fibers; these include lower levels of skin irritation and respiratory system during handling, reducing tool wear during the processing, good recyclability, abundant supply, low cost, low density, high specific strength to weight ratio, non-toxicity, and biodegradability (Spinacé et al. 2009; Gu 2009; John 2009).

The use of coconut fibers as reinforcement in a polymer matrix is important, because it is an inexpensive material when compared with glass fiber, decreases the

amount of waste accumulated in landfills, and yet improves the mechanical properties of composites materials. Coconut fibers have been studied for reinforcement application in polymers such as polyester (Monteiro et al. 2008), polypropylene (Ishizaki et al. 2006), polyethylene (Mulinari et al. 2009), and biodegradable polymers (Rosa et al. 2009) by changing the mechanical properties of these compounds such as tensile strength and elongation at rupture (Harish et al. 2009).

However, the use of natural fibers in composites materials presents a few drawbacks due to some characteristics such as the formation of fiber aggregates during processing, low resistance to moisture, variability of composition, and poor compatibility with hydrophobic matrix polymers (Bessadok et al. 2009).

On the other hand, various treatments have been used in order to improve compatibility of natural fibers, which are polar and hydrophilic due to the presence of hydroxyl groups, with non-polar and hydrophobic thermoplastic matrix materials. These treatments can be physical or chemical, with the goal of modifying the surfaces of the fibers (Bessadok et al. 2009; Pietak et al. 2007).

Among all chemical treatments applied to natural fibers, the most used is alkaline treatment, also called mercerization. The alkaline treatment promotes the removal of partially amorphous constituents such as hemicellulose, lignin, waxes, and oils soluble in alkaline solution, and therefore reduces the level of fiber aggregation, making a surface rougher (Troedec et al. 2008; Esmeraldo 2006; Gomes et al 2007; Razera 2006). During the alkaline treatment, the OH groups present in the fibers react with sodium hydroxide according to equation (1):



According to Razera (2006) this interaction between fiber and NaOH is an acid-base reaction that should not occur in a quantitative manner due to the weak acid character of the hydroxyl groups.

Another important treatment used in order to change some chemical and surface characteristics of natural fibers is bleaching, a chemical process used mainly to obtain pulp for paper production, with the objective of increasing the whiteness. The cellulose and the hemicelluloses present in lignocellulosic fibers do not contribute significantly to coloration, due their naturally white color characteristics. On the other hand, some substances as lignin, dirt, and extractives contribute to its darkness and therefore should be removed during bleaching. For lignocellulosic fibers applied as reinforcement in composite materials, this treatment has as a main objective to attack and to remove the residual lignin. It is important to remove lignin because, despite the fact that it increases the stiffness of the fiber, it is inflexible and prevents the reorientation of the fibers required for the proper transfer of load (Santos et al. 2006; Esmeraldo 2006, Saha et al. 2010; Venson 2008). In addition, lignin acts as a cement between fibrils, and when removed, allows an increase in surface area, thereby improving the fiber/matrix adhesion provided by alkaline treatment.

Many research projects have been conducted in order to study the mechanical properties, and in particular the interfacial performance, of composites based on natural fibers. Attention has been focused on the poor bonding between the hydrophilic natural

fibers and the hydrophobic polymer matrices (Li et al. 2008; Corrales et al. 2007; Acha et al. 2007).

Rout et al. (2001) studied the influence of surface modification on coconut fibers through chemical treatment with sodium hydroxide at concentrations from 2 to 10% on polyester/coir composites and found a 26% of increase in tensile strength values for composites reinforced with 2% NaOH-treated fiber, when compared with untreated fibers/polyester composites.

Rosa et al. (2008), in a study of extraction and characterization of cellulose "whiskers" from coconut fiber, observed by scanning electron microscopy technique the start of a microfibrillation process in the case of bleached coconut fibers. This microfibrillation occurred because the residual lignin materials holding the microfibrils together are extracted during the bleaching process.

On account of this, in the present work, surface modification of coconut fibers with alkaline solution and bleaching, and its effect on mechanical properties of HIPS/coconut fibers composites were evaluated.

## **EXPERIMENTAL**

### **Materials**

Green coconut fruit was obtained from a local supplier. Fibers were extracted from exocarp and mesocarp, then dried at 100°C for an hour, and after being ground in a mill, finally sieved to obtain a sample that passed through a 45 mesh (opening 354 µm). High Impact Polystyrene (HIPS 825) obtained from Videolar was used as matrix.

### **Treatment of the Green Coconut Fibers**

#### *Mercerization*

Green coconut fibers (100g) were pre-treated with 1 L alkaline solution containing 10 g sodium hydroxide (10% 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 fibers were washed with distilled water until neutral pH was attained. Then, fibers were dried in an oven at 50°C for 24 hours.

#### *Bleaching*

The alkali-treated fibers (24 g) were bleached with 200 mL solution containing 1 mL acetic acid and 3 g sodium chloride (80%). This solution was stirring for 2 hours at 70°C, followed by filtration under vacuum and washing with distilled water until neutral pH. Finally, the bleached fibers were dried in an oven at 50 °C for 12 hours.

### **X-Ray Diffraction**

The crystallinity of untreated, treated, and bleached green coconut fibers was evaluated by X-ray diffraction. X-ray patterns were obtained with a Shimadzu diffractometer model XRD6000, under the following conditions: CuK $\alpha$  radiation with graphite mono-chromator, 30 kV and 40 mA. Patterns were obtained in 10 to 50° 2 $\theta$  angular interval, with 0.05° step and 1s of counting time.

The crystallinity index (CI) was calculated using equation (2), where  $I_{002}$  is the maximum intensity of the  $I_{002}$  lattice reflection and  $I_{101}$  is the maximum intensity of X-ray scattering broad band, due to 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.

$$CI(\%) = [(I_{002} - I_{101}) / I_{002}] \times 100 \quad (2)$$

### Composites Preparation

The alkali-treated and bleached green coconut fibers were mixed with the polymeric matrix (HIPS) in a thermokinetic mixer model MH-50H, with the speed rate kept at 5250 rpm, in which fibers were responsible for 10 and 30 wt% of the composition. After mixing, composites were dried and ground in a mill model RONE. Then, coconut fibers/HIPS composites were placed in an injector chamber at 200°C and heated at a 2°C/min rate. The melted material was injected in required dimensions, in a pre-warm mold (210°C) in order to obtain tensile specimens.

### Tensile Tests

Five specimens of composites and pure HIPS were analyzed in a Shimatzu testing machine (model AG-X 50 kN). Tests were carried out according to ASTM standards D638 with 5 mm/min crosshead speed.

Tensile strength, maximum strength and tensile modulus values were automatically calculated by the software “Trapezium X”. This software was provided by Shimatzu. The values of elongation at break were obtained using equation 3:

$$\text{Elongation at break}(\%) = \frac{\text{Tensile Strength}}{\text{Tensile Modulus}} \times 100 \quad (3)$$

### Scanning Electron Microscopy

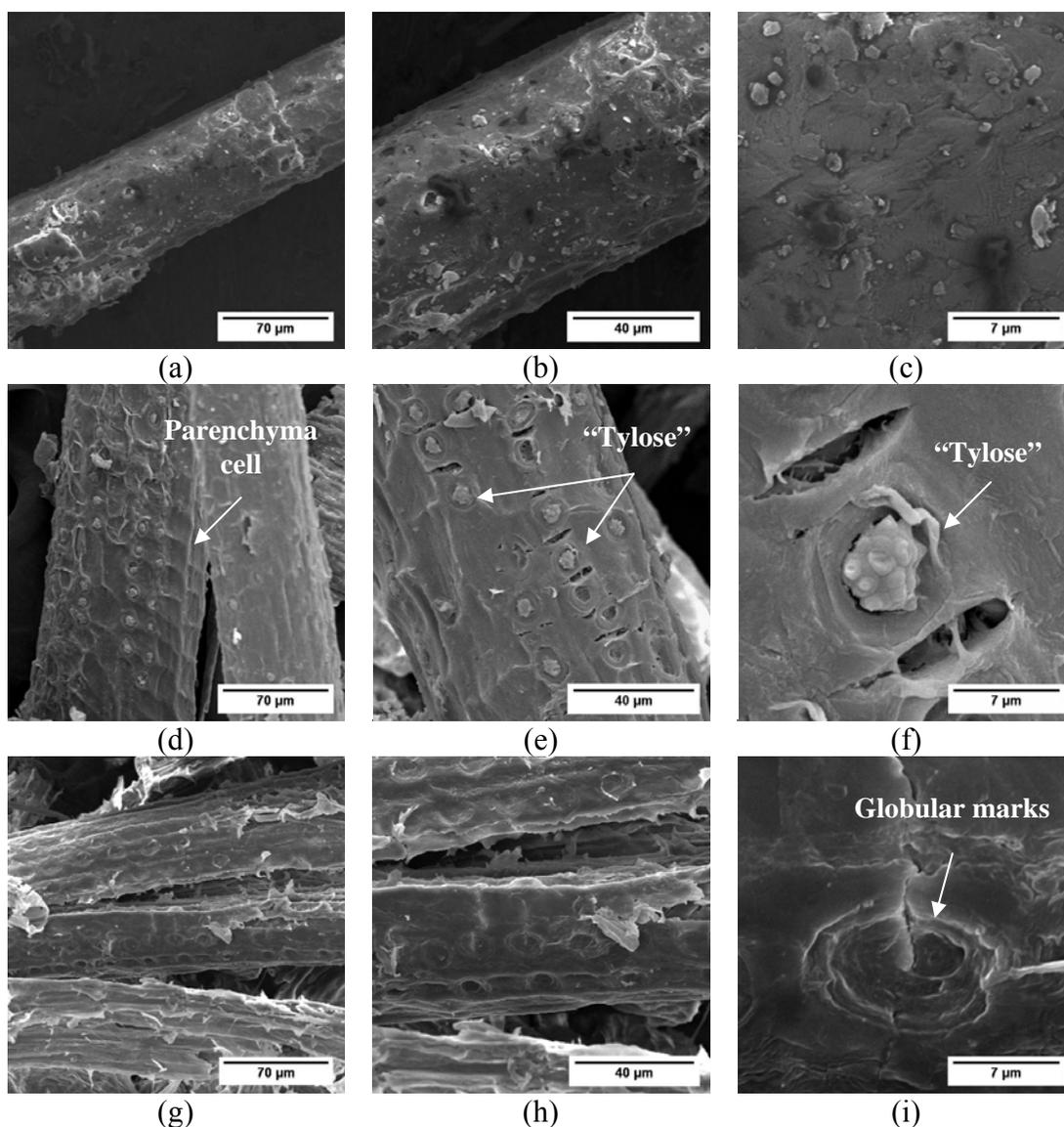
Untreated, alkali-treated, and bleached green coconut fibers, as well as the intact fracture surface of the composites were analyzed with a LEO 1450V scanning electron microscope with a tungsten filament operating at 20 kV, utilizing a low vacuum technique and a working distance of 12 mm.

## RESULTS AND DISCUSSION

### Fibers Characterization

The effect of chemical treatment on fiber surface morphology was analyzed by scanning electron microscopy (SEM). Figure 1 compares SEM micrographs of untreated, alkali-treated, and bleached green coconut fibers. The SEM micrographs of untreated fiber in Fig. 1 (a-c) indicates that green coconut fiber surface is covered with a layer of substances such as oils, waxes, and extractives, part of the natural constitution of lignocellulosic fibers. This layer was also observed by Vilay et al. (2008) in the analysis

of untreated sugarcane bagasse micrographs and by Huang Gu (2009) in the analysis of the tensile behavior of brown coir fiber.



**Fig. 1.** SEM micrographs of fiber surface of: (a), (b), and (c) untreated fiber; (d), (e), and (f) alkali-treated fiber; and (g), (h), and (i) bleached fiber

Micrographs in frames (d) through (f) of the figure show a rough surface, which is a consequence of the alkaline solution treatment effect. According to Troedec et al. (2008), this chemical treatment removes extractives, waxes, and oil from fiber surfaces and thus increases the overall roughness of surface. With the removal of these substances, it was possible to verify the presence of parenchyma cells that are the natural constituents of lignocellulosic fibers, as well as the presence of globular protusions, which are fatty deposits called “tyloses” (Rout et al. 2001). These globular protusions, shown in detail in frame (f) of Fig. 1, are arranged on the fiber surface at regular intervals. Their presence

on the surface of coconut fibers was also observed by Bigda et al. (2010), Bismarck et al. (2001), and Calado et al. (2000).

Finally, after bleaching treatment, as can be seen in Fig. 1 (h-i), most of these fatty deposits were removed, which revealed, on the fiber surface, empty cavities, bringing out a rough surface with globular marks, with consequently higher surface energy.

From the micrograph of bleached fiber in frame (g) of Fig. 1 it is possible to observe that the fibers had been partially disintegrated, which occurs due to the extraction of residual lignin materials that holds the fibrils together.

Figure 2 shows the presence of "pits" longitudinally arranged along the entire cell wall, inside and outside the parenchyma cells of alkali-treated and bleached fibers. These "pits" are circular holes about 1  $\mu\text{m}$  of diameter, and according to Luz et al. (2008), are responsible for transporting water and nutrients throughout various cells to the roots and leaves (Martin et al. 2009).

In general, pits are hidden on the surface of untreated fibers due to the superficial layer of wax and extractives; however with layer removal by alkaline treatment the pits are revealed.

The presence of pits and globular marks after chemical treatment are important for an increase in the effective surface area and a higher increase of the roughness, with a consequently improving mechanical bonding with the polymeric matrix (Pietak et al. 2007; Vilay et al. 2008).

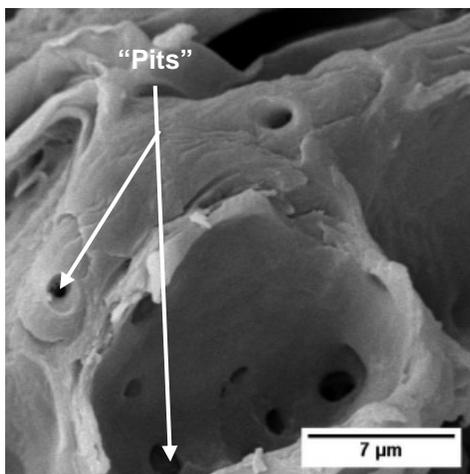
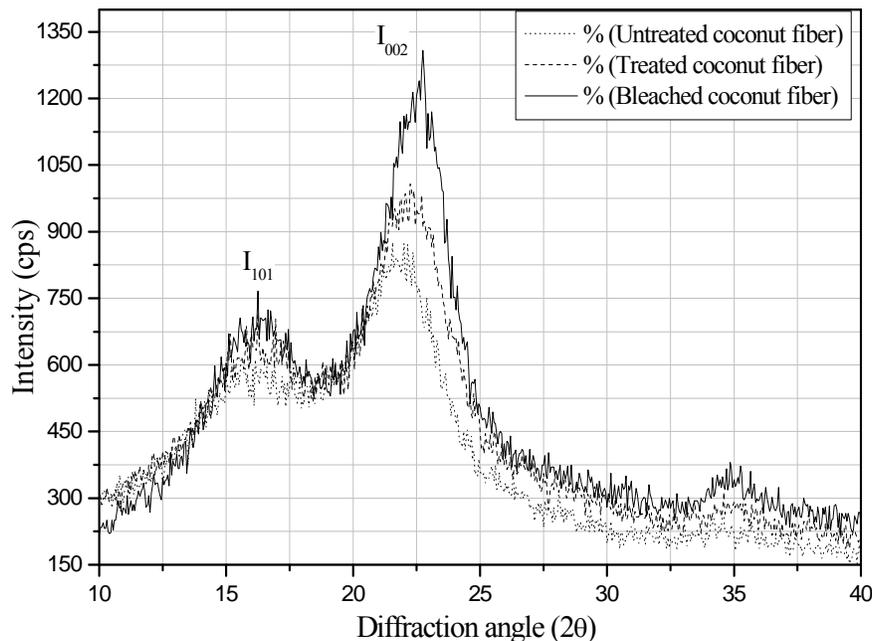


Fig. 2. SEM of alkali-treated coconut fibers.

Figure 3 presents the X-ray diffractogram for untreated, alkali-treated, and bleached green coconut fibers. For fibers untreated and with different treatments the occurrence of two intense peaks was observed, close to values of  $2\theta = 16^\circ$  and  $2\theta = 22^\circ$ , representing the cellulose crystallographic planes  $I_{101}$  and  $I_{002}$ , respectively. The X-ray diffraction peaks observed can be attributed to crystalline scattering and the diffuse background associated with disordered regions.



**Fig. 3.** X-Ray diffractograms of cellulose

The spectrum corresponding to untreated fibers shows diffraction peaks at the  $2\theta$  angles of  $16.15^\circ$  and  $22.01^\circ$ . The same peaks for alkali-treated fibers were observed at  $16.25^\circ$  and  $22.23^\circ$  and for bleached fibers at  $16.25^\circ$  and  $22.77^\circ$ . The superposition of the X-ray diagrams shows that the signal characteristics of fibers with different treatments were almost similar. However, alkali-treated and bleached fiber peaks were more intense than untreated fibers' peaks, which means that both treatments were able to remove part of the amorphous material covering the fiber, thus exposing the cellulose.

The crystallinity index ( $CI$ ), calculated according to equation (1), can be observed in Table 1. The treated fiber showed a decrease of 3% in the crystallinity index; however, the bleached fiber exhibited about 40% higher crystallinity index than untreated fiber, associated with the treatment effect on the fiber chemical composition, showing the complementarity of the two surface treatments.

**Table 1.** Crystallinity Index of Coconut Fibers

Material	$I_{(002)}$	$I_{(101)}$	$I_c$ (%)
Untreated fiber	873.42	611.98	29.93
Alkali-treated fiber	1008.76	715.94	29.02
Bleached fiber	1309.57	760.85	41.90

The reduction of 3% in the crystallinity index for alkali-treated fiber, although the diffraction peak showed an increase of intensity, is associated to the fact that alkaline treatment was not totally effective in removing a sufficient amount of lignin in order to expose the cellulose and consequently increase the crystallinity. With the bleaching, the

removal of residual lignin increased the exposure of the cellulose, resulting in the higher crystallinity index.

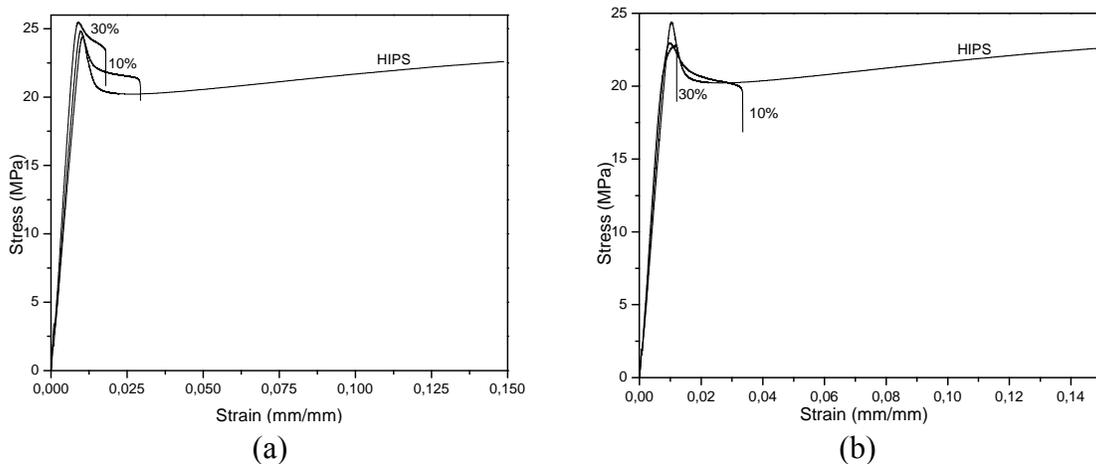
### Characterization of Composites

Mechanical properties such as elongation at break, tensile strength, and tensile modulus for pure HIPS and for composite materials containing different green coconut fibers treatment are shown in Table 2. The volume fractions of alkali-treated and bleached green coconut fibers inserted into the polymeric matrix were 10 and 30 wt%.

**Table 2.** Mechanical Properties of the Composite Materials

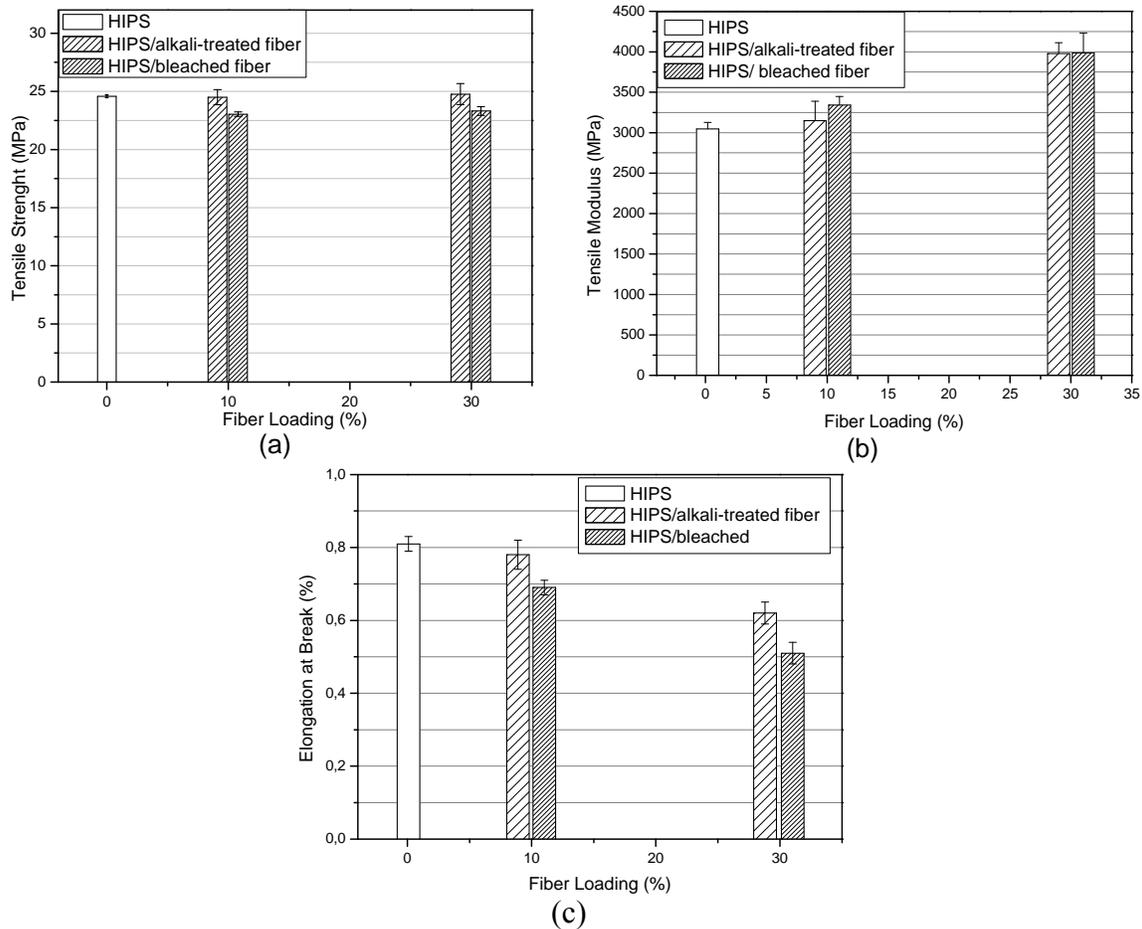
Samples (Reinforcement in wt%)	Properties		
	Tensile strength (MPa)	Tensile modulus (MPa)	Elongation at break (%)
HIPS	24.58 ±0.12	3045.68 ±81.42	0.81 ±0.02
Alkali Treated coconut fibers (10%)/HIPS composites	24.51 ±0.65	3146.90 ±242.01	0.78 ±0.04
Alkali Treated coconut fibers (30%)/HIPS composites	24.77 ±0.90	3977.34 ±133.80	0.62 ±0.03
Bleached coconut fibers (10%)/HIPS composites	23.04 ±0.20	3340.49 ±106.75	0.69 ±0.02
Bleached coconut fibers (30%)/HIPS composites	23.32 ±0.37	3986.31 ±244.96	0.51 ±0.03

Figure 4 shows the stress-strain curves obtained for the pure HIPS and composites with different green coconut fibers treated. The curves for the pure HIPS showed a ductile character material with extensive plastic deformation. With the addition of alkali-treated fibers (Fig. 4 (a)) and bleached fibers (Fig. 4(b)) in the polymeric matrix, the curves show that the composites failed after a maximum point, with a small amount of plastic deformation.



**Fig. 4.** Stress-strain curves for the HIPS matrix and the composites with different (a) alkali-treated fiber contents, and (b) bleached fiber contents.

By analyzing the data in Table 2 and the graphics in the Fig. 5, it is possible to observe that values of tensile strength increased for composites reinforced with alkali-treated fiber, while they decreased for composites with bleached fiber. Values of tensile modulus increased with the addition of alkali-treated fiber and bleached fibers. Composites with 30% of fibers (alkali-treated and bleached) resulted in an increase of approximately 31% in the tensile modulus values, when compared with pure HIPS. The increase of tensile modulus according to the increase of fiber volume for both chemical treatments can be better viewed in Fig. 5 (b).



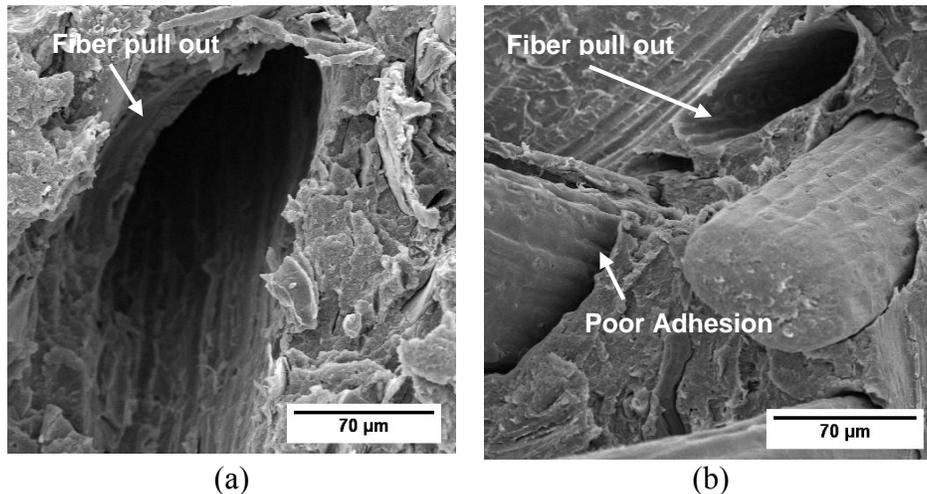
**Fig. 5.** Mechanical properties of pure HIPS and composites with different fiber loadings: (a) tensile strength; (b) tensile modulus; and (c) elongation at break.

Due to more brittle character of reinforced composites, a decrease in the values of elongation at break was observed, especially for those samples reinforced with bleached fiber. The decrease in elongation at break according to the amount of fiber and chemical treatment can also be seen in Fig. 5 (c).

In this study, the reinforcement was more effective in increasing tensile modulus values of composites reinforced with 30% of alkali-treated and bleached fibers. On the other hand, the addition of fibers did not contribute to a significant increase in the tensile

strength values, due to lack of adhesion between fiber matrix, as observed by scanning electron microscopy of composites fracture surfaces.

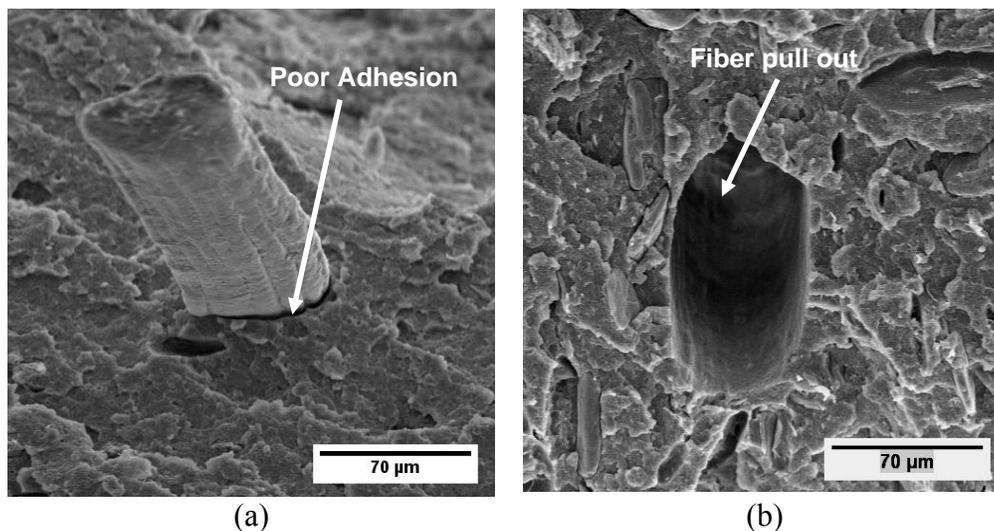
In Fig. 7(a) and (b), it is possible to observe evidence of poor interaction between alkali-treated fibers and the matrix, with the presence of fiber pull-out, confirming that the chemical treatment was not totally effective in improving the adhesion between the fiber and matrix.



**Fig. 7.** SEM of alkali-treated coconut fibers /HIPS composites (500x): (a) 10 wt% and (b) 30 wt%

From fracture surface micrograph in the Fig. 8 it was possible to note also a lack of adhesion between bleached fiber and the matrix, with the presence of voids due to the fiber/matrix pull-out.

In the analyses of composites fracture surface with alkali-treated and bleached fibers it was not possible to note significant differences between the different chemical treatments performed.



**Fig. 8.** SEM of bleached coconut fibers /HIPS composites: (a) 10 wt% and (b) 30 wt%.

## CONCLUSIONS

1. Alkaline treatment of green coconut fibers surface was effective for removing the extractives and increasing the roughness of the surfaces;
2. Bleaching treatment intensified the effect of alkaline treatment, increasing the crystallinity index and surface fiber energy with the removal of residual lignin;
3. The addition of 30% of fibers in the matrix provided considerable changes in the tensile modulus values of composites compared with the pure HIPS;
4. On the other hand, chemical treatments were not totally effective for improving the adhesion between the fiber and matrix. The lack of adhesion could be observed in the results of SEM and was confirmed with the results of tensile tests, which showed small variation in the average values of tensile strength;
5. In the analysis of the fracture surface of composites it was not possible to identify significant differences resulting from the different chemical treatments performed.

## ACKNOWLEDGMENTS

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