Processing Characterization of Sisal/Epoxy Prepregs

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Abstract: Quality control to obtain composite laminates is frequently applied to synthetic fibers/epoxy prepregs. The gel time test, resin, volatiles and fiber content, drape measurement and tack tests together with water absorption capacity are methods currently employed. However, for natural fibers prepregs there is a gap in the literature, which makes their application difficult. Thus this work will investigate sisal fibers, which have low cost, high biodegradability and low specific weight, following the common methods to manufacture composites from natural fibers/epoxy prepregs. First, the prepregs were prepared by hand lay-up, aligning the fibers with epoxy, keeping 15% by weight content of fiber. After the quality control characterization, 3 mm thickness composite was prepared by using a press, and tensile tests and scanning electron microscopy (SEM) were applied. As a result, the resin fraction values and the solid content of the matrix showed little variation between the different samples. The natural fibers prepregs absorbed water quickly in the initial stage until reaching the saturation level. The NaOH-treated sisal/epoxy prepreg had a tension of 71.06 ± 8.28 kPa for the tack test and tensile strength of 69.24 ± 11.69 MPa. Finally, the NaOH-treated sisal 15 wt%/epoxy resulted in composites with a better performance than the neat epoxy resin. There was good adhesion between the fibers and matrix, as confirmed by SEM and mechanical tests.

Keywords: Natural fibers prepregs, sisal, gel time, resin, fibers and volatiles content, tack test, drape measurement.

1. INTRODUCTION

Although in the literature there are several studies on epoxy prepregs, little has been developed regarding natural fibers prepregs, especially with sisal fibers. The quality control of these materials is extremely necessary, as natural fibers have the capacity to replace or complement synthetic fibers to manufacture high-quality composites [1].

In general, the prepreg is an intermediate material ready for molding, although it is formed by a set of reinforcement fibers and thermosetting polymeric matrix, and is partially cross-linked [2]. Due to its low specific weight and high strength and stiffness values, this class of materials can replace some traditional metallic materials. Currently, the most common method of obtaining composites in the industry is through prepregs in the form of a tape or fabric, which is molded and cured without adding new products or resins [3].

The resin, fibers and volatile content, gel time, adhesion and water potential uptake are the main factors that guarantee the quality of a prepreg. Traditional analytical methods such as solvent extraction, weighing and combustion are often used to measure the resin, fiber and volatiles content of the prepreg [4]. When the prepreg has a low amount of volatile substances, its aging is induced. The right quantities of resin and fibers in the prepregs provide the right degree of flexibility and the ideal handling capacity during the layer stacking process in the manufacture of laminated composites [1,5]. Thus, quantifying these values is extremely important in the process of manufacturing prepregs, especially because the fiber and resin content is hard to achieve for natural fibers.

Some other methods are used by the industry to analyze the quality and performance of prepregs before their effective use. Among them, the gel time checks the reactivity of the system from the time between the liquid phase and the first appearance of the solid phase [6]. This allows the ideal curing time and temperature to be evaluated to determine the best conditions to manufacture the prepreg and the composites [7].

Other important factors are the adherence capacity (tack test). This method checks the capacity of the prepreg to adhere to surfaces and to itself during molding. The drapeability capacity test of prepregs is commonly verified with the placement of the material in curved molds [8,9].

Due to the hydrophilic character of the fibers, the water absorption capacity within the prepregs must

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also be verified, since a high aqueous capacity means a low adhesion between the fibers and the resin [10]. Costa *et al*. [11] reported that moisture absorption reduces the mechanical properties of these materials. Especially regarding natural fibers, the process of moisture absorption can occur due to capillary action, involving the pores or channels present in the fibers.

Although the mechanical properties of natural fibers are lower than synthetic ones, their density (~ 1.4 g/cm³) is approximately half that of glass fiber, to which the price would be approximate when compared to carbon fiber, which has a density similar to natural fibers [12]. In this context, composites reinforced with sisal fibers stand out for having high impact resistance, and good tensile and bending properties [13]. Sisal (*Agave sisalana*) is an attractive natural fiber option due to its rapid growth in a wide range of climatic conditions, and sisal lignocellulosic fiber is light, non-toxic and has a high modulus and specific resistance, in addition to causing less damage by abrasion to equipment and molds than inorganic fibers [14].

Therefore, sisal/epoxy prepregs are of considerable commercial interest. According to Webo *et al*. [15], the automotive industry is already considering the use of natural fibers, which would help not only in providing data for the industrial sector but also in the discovery of new materials that can be used in the future, since they are abundantly available. Besides that, the crop culture can promote opportunities to improve the living standards of people in a region [16].

Several studies have reported the use of synthetic fibers as a reinforcement for matrix polymers in particular prepregs; however, few have discussed the potential use of sisal fiber as a reinforcement of prepregs and how the quality of the processing of these materials interferes with the potential use by industries in general. Thus, this work aims to gain a better understanding of the sisal/epoxy prepreg behavior regarding the resin, fiber and volatile content, the adhering properties before curing such as tack and drapeability capacity, and the potential water uptake in order to optimize the processing to obtain natural fibers composites with considerable mechanical performance.

2. MATERIALS AND METHODS

2.1. Materials

Sisal fibers were supplied by (SisalSul Comércio e Indústria Ltda), from Sisaleira region Bahia, Brazil. Before treatment, the sisal fibers were cut to 25 cm length. Regarding the matrix, the AR 260 epoxy resin and AH 260 hardener were supplied by E-composites, both from DML Chemicals in Brazil.

2.2. Sisal Fibers Treatment

The fibers were mercerized with a sodium hydroxide (97% pure; Greentec) solution at a concentration of 5% (w/v). For that, they were immersed in a NaOH solution with a ratio of 10:1 (solution:fiber) in a water bath at 80 °C for 2 h under constant stirring. After the treatment, the fibers were washed with distilled water until neutral pH. Finally, the filtered fibers were placed on a polypropylene tray at room temperature (~ 25 °C) for 96 h [17] until completely dry.

2.3. Prepregs Fabrication

The epoxy prepregs were obtained by the hand lay-up method (Figure **1**), containing 15 wt% of fibers. First, the fibers were dried in an air-circulating oven (F2-DM, Fornitec, Brazil). Then, they were placed unidirectionally in a mold of 200 mm length (Figure **1A**). The polymeric material was prepared by mixing the epoxy resin with a hardener at a ratio of 100:21 according to the manufacturer's instructions. Next, the resin mixture was applied to the fibers (Figure **1B**).

Figure 1: (**A**) The fiber is placed into the mold; (**B**) the fiber is wetted with epoxy resin and (**C**) the resin is spread over the fibers.

Further, the resin was homogeneously spread with a metallic roller (Figure **1C**). The prepreg was kept at room temperature until it reached stage B, followed by refrigeration (-18 ºC).

2.4. Prepregs Characterization

2.4.1. Gel Time, Differential Scanning Calorimetry (DSC) and Water Percentage Uptake

The gel time was based on the American Society for Testing and Materials Standard (ASTM), ASTM D3532. The temperatures used to observe the gel time of the resin were determined from the DSC curve data, where three points of interest were chosen. Samples of the prepregs were placed on a glass slide previously heated for 20 s on a heating plate (752A, Fisaton, Brazil) and probed until the resin did not show any flow. The time between the point at which the temperature was applied and the gel stage was recorded as the gel time. The experiments were performed in triplicate for each temperature.

For the DSC measurement, prepreg samples weighing 10 ± 0.5 mg were deposited on an alumina pan and covered with an alumina lid. The samples were analyzed in a simultaneous (TGA-DSC) thermal analyzer (Q600 SDT, TA Instruments, USA) under a nitrogen atmosphere with a gas flow of 50 mL.mm^{-1} at a heating rate of 2.5 ºC.min^{-1}, from 25 to 300 ºC.

The water percentage uptake was based on the method of Bhardwaj et al. [18], where six samples with dimensions of 100 x 100 mm were weighed and their values were recorded as G1. Then, the samples were immersed in water for 5 min at 25 ºC. After this time, the excess water was removed with absorbent paper and the weights were recorded as G2. The average of the weights for the six samples was used to obtain the percentage of water absorption with the following Equation (1), where the WPU is the water percentage uptake:

$$WPU(\%) = \left(\frac{G2 - G1}{G1} \right) \times 100 \qquad (1)$$

2.4.2. Resin, Fiber and Volatile Content

The resin content (R%), fiber content (FC%) and volatile content (V%) were measured according to the ASTM D3529 and ASTM D3530 standards. Two samples (50 x 50 mm) denoted as A and B were weighed separately, and their values were recorded as GA and GB. Then, the sample A was dissolved in 100 mL of acetone for 3 min under constant stirring and the

excess material was filtered in a Buchner funnel previously weighed (GA1). After the filtration, the funnel was placed in an oven with air circulation at 160 ºC for 15 min to dry the sample completely. After the drying, its weight was recorded as GA2. The previously weighed sample B was also placed in the oven with air circulation at 160 ºC for 10 min and immediately afterward in a desiccator. After cooling, the sample weight was registered as GB1. The required data was calculated using the following Equations (2-4):

$$R(\%) = \left(\frac{GA - (G2 - G1)}{GA} \right) \times 100 \qquad (2)$$

$$V(\%) = \left(\frac{GB - GB1}{GB} \right) \times 100 \qquad (3)$$

$$FC(\%) = \left(\frac{GA2 - GA1}{GA} \right) \times 100 \qquad (4)$$

2.4.3. Drapeability Capacity

The method developed by Shin et al. [19] was used for prepregs samples. Three sisal/ epoxy prepregs layers were placed at a 75° angle, two with the partially polymerized resin/ hardener set (D-1 and D-2) and one with the fully polymerized set (F-1). D-1 was placed on the mold shortly after being manufactured, while D-2 after an interval of 1 h after five days of refrigeration. In addition, five layers of the material (D-2) were superimposed and placed in a mold, in order to see if the 'book effect' described by Lengsfiel et al. [20] occurred, where it is successful if no voids form inside the laminates. The objective was to compare the drapeability behavior at room temperature of the partially cured and fully cured samples and the ability to form molds with overlapping layers. The material was observed under a magnifying glass.

2.4.4. Tack Test

The tack test was carried out with the intention of investigating the tension necessary for the pre-impregnated layers to separate. The test was performed based on the literature, as there are no standards that regulate this procedure. The test specimen was prepared with three prepregs layers of 100 × 50 × 1 mm (outer layers) and 50 x 50 x 1 mm (center layer). One side of the specimen was pressed with a load of 500 N for 30 s in a hydraulic press, forming a sandwich, while the larger sides were not pressed. The part not pressed was the region that would be trapped in the claws of the testing equipment. The experiments were performed in triplicate on a

universal testing machine (Instron 8801, Instron, USA) equipped with a 100 kN load cell at a speed of 2.5 mm.min^{-1}.

2.5. Composite Characterization

2.5.1. Composite Manufacturing by Using the Prepregs

The composites plates with dimensions of 220 × 200 × 3 mm were prepared using 3 layers of prepregs. The not fully cured prepregs were molded using the dry lamination technique, which consists of stacking the pre-impregnated layers in a metallic mold. The mold/prepreg set was inserted in a hydraulic press (P15T, Ribeiro, Brazil) under a load of 4.9 kN at room temperature (25 $^{\circ}$C) for 1 h. The total cure followed at room temperature for 24 h. Finally, post-curing was carried out in an oven at 60 °C for 12 h, as indicated by the manufacturer.

2.5.1. Tensile Mechanical Test

The tensile test was performed by ASTM D3039-14. The test specimen size was 200 × 25 × 2.5 mm. The test was performed on a universal testing machine (Instron 8801, Instron, USA) equipped with a 100 kN load cell at a speed of 1 mm.min^{-1}. Five (n = 5) specimens of each composite were characterized for each type.

2.5.2. Scanning Electron Microscopy (SEM)

The surface morphology fractured surface of the composites was investigated by SEM (TM-4000Plus, Hitachi, Japan). There was no covering of the samples. The images were then captured using a voltage of 15 kV and 40 × and 150 × magnification.

3. RESULTS AND DISCUSSION

3.1. Gel Time, Water Absorption and Volatiles Content of the Prepregs

The gel time is the stage during the thermoset curing reaction, which can be classified as the transition from a liquid to a solid-state. The gel point occurs when the polymer begins to exhibit pseudoelastic properties. During the matrix processing, gelling should be avoided before the final shape is molded, since the material cannot be re-molded [21,22].

According to the DSC curve (Figure **2A**), three temperatures of interest were selected in the material's gelation region: 50, 80 and 100 °C. The average values

for each temperature are plotted in Figure **2B**, where it is possible to observe that the higher the temperature, the faster the gelation process takes place. The gradual increase in the viscosity that was observed at the beginning of the DSC curve could be due to a slow restructuring of the material system [23]. However, simply observing the evolution of the viscosity, it is not possible to determine the gel time accurately. Previous studies state that the heating rate of 2.5 °C.min^{-1} is the most suitable for curing prepregs as this helps to control the curing of the system, and at temperatures below 100 ºC. Curing in an autoclave is unfeasible [9,23], so the temperature of 100 °C is the most suitable for the consolidation process of the composites using the current resin (AR 260), since this temperature allows more controlled curing and prevents the formation of voids.
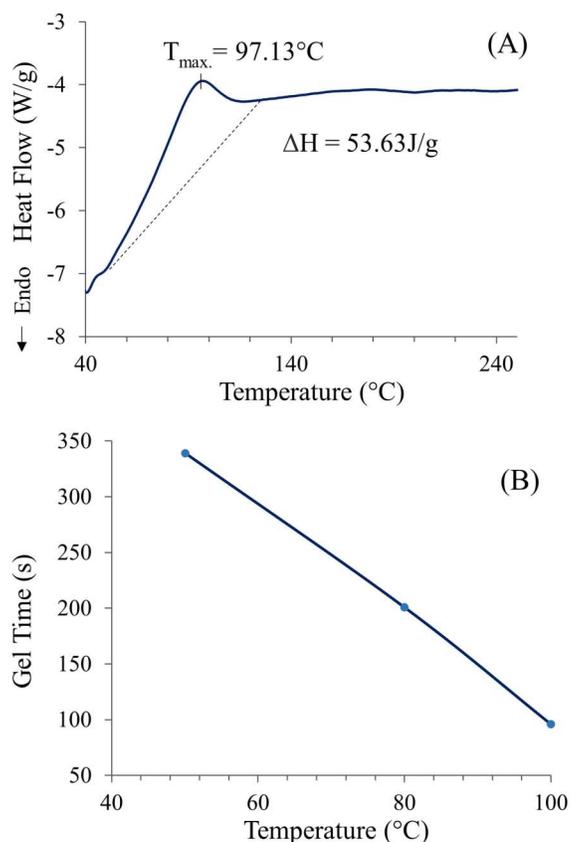
Figure 2: DSC curve (**A**) and gel time *versus* temperature (**B**).

According to Oh *et al*. [24] reported in their study regarding gel time for epoxy composites, the temperature of 80 ºC was the longest, which contributes to a more efficient curing process.

The water percentage uptake is the ratio between the weight of the water absorbed by a material and the

weight of its respective dry material. This test can only be performed during the prepreg manufacturing process, before freezing, because the storage and handling affect the measurement results [21,25].

Table **1** shows the average water percentage uptake for the resin and prepregs after immersion in water at 2 h and 24 h. According to Sreekumar *et al*. [10], the fibers absorb water quickly in the initial stage until the saturation level is reached, after which no further increase is obtained. This can be confirmed by the behavior of the sample, since, when the absorption reaches equilibrium, the weight gain for the untreated sisal/epoxy prepreg was 15.37% compared to the starting weight. The partially cured resin absorbed hardly any water, which can be explained by the hydrophobic character of the material.

As shown in Table **1**, for the NaOH-treated sisal/epoxy, the WPU value was approximately 10.78% at 24 h and increased over time, to around 14.31% compared to the weight of the sample after 2 hours immersed in water. According to the study by Abdelmola *et al*. [26], the saturation increases as the void content increases, so when the void volume is filled, the water absorption by the composite is equalized.

The WPU values showed that the NaOH-treated sisal/epoxy prepregs had positive results when compared to untreated fibers/epoxy prepregs. In addition to obtaining a more homogeneous material, the few voids and the better wettability make it difficult for moisture to enter due to the good adhesion between the reinforcement and the matrix. Sreekumar *et al*. [10] also reported that mercerized sisal fiber reinforcing materials had a lower water absorption rate.

The initial weight content for the resin adopted for the production of prepregs was 85 wt%. However, during the processing, some experimental issues such as weight losses, volatiles and the natural fibers humidity content can change the resin and fibers content. The measurement of the fiber/resin fraction was determined from the size of the mold and the density of the resin as indicated by the manufacturer (1.15 g.cm^{-3}), as we described in the method part of this paper. As shown in Table **1**, the real resin content in the prepregs was 82.78 wt%, and the fiber content was 17.22 wt%. Considering the resin content and the solid content of the matrix, they presented little variation between the different samples. It is possible to infer that the existing difference was influenced by the volatiles content present in the resin or voids contained in the material.

The volatile content obtained from the aforementioned standard showed an average of 1.64 wt%, which follows the acceptable values [5]. Moreover, Xia *et al*. [27] observed that the lower the volatile content, the higher the rate of use of the system, since the number of voids contained within the material is reduced, which causes an increase in properties in composites. The prepreg in question had five days of freezer cooling, and although this study does not consider the storage time, it is possible that some of the volatile loss occurred during this time, since the storage time influences the volatile content in the material [28].

3.2. Drapeability and Tack Test for the Prepregs

Figure **3** shows the drapeability capacity with a 75º angle to simulate the shape of a corner mold. The partially cured samples proved to be flexible and easily cut, whereas the cured sample had reduced flexibility and increased rigidity, as can be seen in Figure **3A**, thus losing the ability to adhere to other layers, making it impossible to mold under these conditions. Shin *et al*. [19] reported a similar behavior in their work with thermoplastic resin, differentiated only due to the possibility of heating, which restored the material's

Table 1:   Water Percentage Uptake (WPU) and Content Data for the Resin and Prepregs

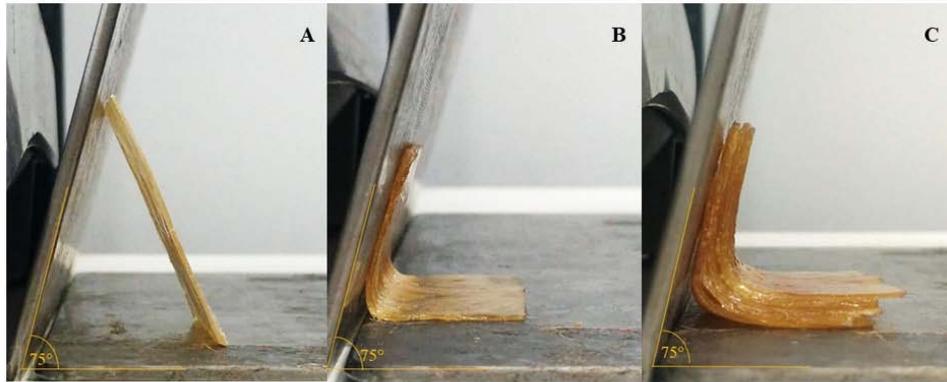| Specimen/ Properties	Partially cured resin	Untreated sisal 15 wt%/ epoxy	NaOH-treated sisal 15 wt%/ epoxy
WPU after 2 h (%)	0.68 ± 0.17	14.31 ± 2.10	9.43 ± 3.37
WPU after 24 h (%)	0.78 ± 0.27	16.51 ± 3.73	10.78 ± 3.64
Resin Content (wt%)	-	-	82.78 ± 2.18
Volatile Content (wt%)	-	-	1.64 ± 0.43
Solid Matrix Content (wt%)	-	-	82.48 ± 2.72
Fiber Content (wt%)	-	-	17.22 ± 2.61

Figure 3: NaOH-treated sisal/epoxy fully cured prepreg (**A**), one layer of partially cured prepreg (**B**) and five layers of partially cured prepregs (**C**).

ability to adhere and mold, which is not possible with thermoset resins.

NaOH-treated sisal/epoxy prepregs were placed at the corner, one layer shown in Figure **3B** and five layers in Figure **3C**. We can see in both cases that there is adhesion on the plate, most effectively for one layer. In the case of the three-layer sample, they did not adhere effectively to the corner of the mold. However, the prepregs layers completely adhered to each other without leaving voids (Figure **3C**). In composites, the stiffness and strength are determined by the reinforcing fibers, so it is essential to maintain the initial and precise positioning of the fibers throughout the manufacturing process according to Wang *et al*. [29], as inefficient handling can cause the spreading/distortion of the fibers and the reduction of volume in certain places since even the partially polymerized the matrix tends to flow.

Figure **4** shows a typical tack test for the prepregs pulled through the two claws of the tensile clamps to simulate the separation of the surface from the laminated layers. During the dismemberment, the fibers move from the resin, while fibrillation begins. Although the adhesion of the prepregs cannot be quantified exactly, it was possible to observe the average values of maximum stress during the separation of the laminates. The NaOH-treated sisal 15 wt%/epoxy prepreg presented a tension of 71.06 ± 8.28 kPa. No maximum stress values were found in the literature, only the separation energy for glass/epoxy and glass/phenolic prepregs [9,30].

It was observed that the NaOH-treated sisal/epoxy prepreg showed a high adherence capacity, since the material is capable of adhering to itself and to surfaces. According to Silva *et al*. [28], this material is qualified as Level V (high adhesion, glue on gloves, adheres to

itself and other surface). As it is a complex phenomenon, it is not possible to decompose the adhesion properties into a single value. Therefore, a whole set of tests must be taken into consideration. Recently, two test methods are being developed by the ASTM Committee: ASTM WK67852 and WK70428 [31]. Thus, this study aims to only inform of the behavior of prepregs at the time when two previously pressed layers separate.

Figure 4: Separation of the laminated layers during tack test, NaOH-treated sisal epoxy prepreg.

3.3. Tensile Behavior and Morphology of Fracture of the Composites

Figure **5** shows the stress–strain curves for the NaOH-treated sisal/epoxy composites built with five

layers of pressed prepregs, compared to the neat matrix. It is clear that the elongation at break for the samples is low, due to the characteristic fragile behavior in thermoset matrices.
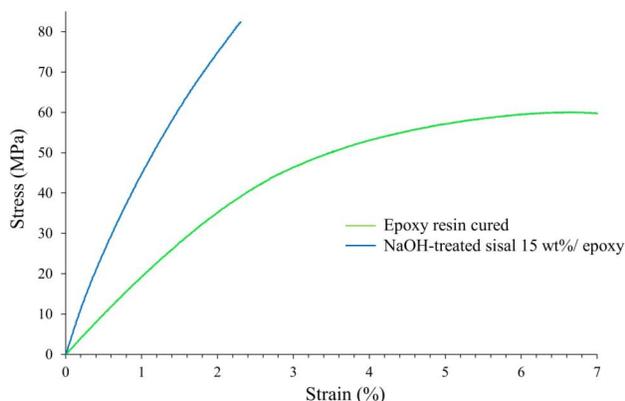

Figure 5: Stress–strain curves for epoxy resin and the sisal/epoxy composites.

According to the data presented in Table **2**, there was an increase in the mean values of tensile strength for NaOH-treated sisal composites to higher than the neat epoxy resin. Thus, there was a 28% increase in the tensile strength compared to the neat matrix. The good tensile properties for the sisal fiber composites can be explained by the aligning of the fiber within the composite. Vimalanathan *et al*. [32] concluded that fiber orientation and adhesion are directly linked to the tensile properties of a composite. In addition, the fibers must comply with the length of the load direction so that the tensile strength of the composites can be applied effectively.

According to Table **2**, the sisal/epoxy composites showed higher Young's modulus values (3.33 ± 0.26 GPa) than the epoxy resin. A study using polyester composites and sisal fibers treated with NaOH also showed higher Young's modulus values (3.02 ± 0.05 GPa) when compared to other treatments [10]. However, the addition of the fibers reduced the elongation capacity of the material [33].

Surface images of the NaOH-treated sisal 15 wt%/epoxy composites are shown in Figure **6**. Figure **6A** shows a number of fibers pulled from the matrix, a failure mechanism known as pull-out, which is commonly found in composites reinforced with unidirectional fibers [33]. An increase in the image size (Figure **6B**) shows that there was no fiber defibrillation during extraction, a behavior similar to that reported by Ma *et al*. [34].

In addition, it is observed (Figure **6A**) that there was a good adhesion between fiber/ matrix, which contributed to greater tensile strength. The same behavior was reported by Teixeira *et al*. [33] in polyester resin composites reinforced with curaua fibers. Moreover, according to Sankar *et al*. [35] this better fiber/ matrix interface contributes to a more

Table 2: Tensile Properties for the NaOH-Treated Sisal/ Epoxy Composites

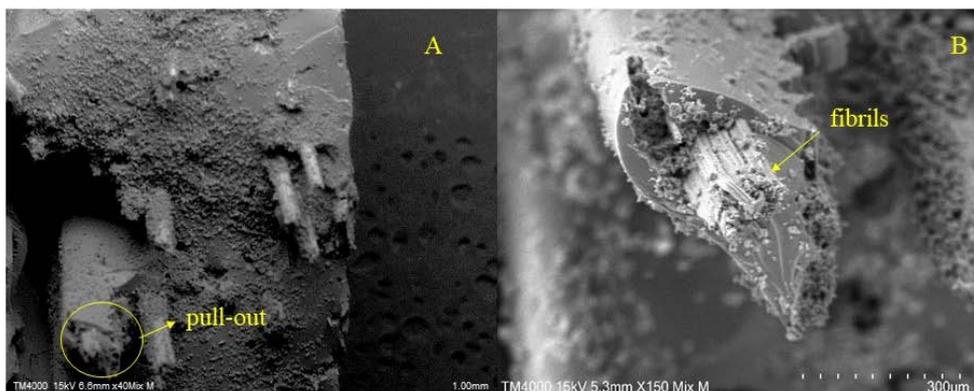| Properties/ materials	Epoxy resin fully cured	NaOH-treated sisal 15 wt%/ epoxy
Tensile strength (MPa)	53.7 ± 7.25	69.24 ± 11.69
Elongation at break (%)	5.32 ± 1.82	2.07 ± 0.23
Young's modulus (GPa)	1.09 ± 0.30	3.33 ± 0.26

Figure 6: SEM images (40x and 150x magnification) of NaOH-treated sisal/epoxy composites.

efficient load transfer. Thus, the fiber strongly bonded to the matrix imparts a high strength and stiffness to the composite.

The composites showed visible resin residues on the fibers. According to Yan *et al*. [36], this behavior is expected due to the previous treatment carried out on the fibers, which removes the layer of lignin and waxes and allows a better interfacial compatibility between the fiber and the matrix. These observations corroborate the results of the mechanical tests, since the perception of the fiber direction within the composite in the fracture region influences the material properties.

4. CONCLUSIONS

The main findings regarding the quality control for sisal fibers/ epoxy prepregs to obtain composite laminates were:

1. The gel time showed that as the temperature increases, the gel time decreases, due to a faster formation of the cross-link process;

2. There was a low proportion of volatile content for the prepregs developed in this study, and the resin and fibers content were as proposed;

3. The drapeability capacity showed that the adhesion on the mold is most effective for one prepreg layer, and the adhesion between the layers seems effective;

4. The tack test obtained mean values of maximum stresses around 71.06 ± 8.28 kPa;

5. The NaOH-treated sisal resulted in prepregs with lower water absorption than the untreated fibers; and

6. The tensile test showed that NaOH-treated sisal 15 wt%/epoxy showed better tensile strength and Young´s modulus results compared to the neat matrix.

To conclude, the commonly used methods to characterize the prepregs can be successfully applied to natural fibers prepregs, resulting in composites with superior tensile properties when compared to neat matrix.

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