

# The Influence of Bleached Jute Fiber Filler on the Properties of Vulcanized Natural Rubber

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There is growing interest in replacing the synthetic reinforcements used for natural rubber (NR) composites with natural fibers from renewable, environmentally sustainable sources. Jute fibers may be suitable reinforcements for NR because they have a high tensile strength, which is comparable to other fillers such as glass fibers. Thus, we studied the incorporation of bleached jute fibers into NR, characterizing the mechanical, dynamic mechanical, and morphological properties of the composites. The addition of fibers to the NR latex was achieved by chemical coagulation, followed by vulcanization in a laboratory two-roll mixing mill. The elastic modulus at 100 and 300% elongation of the NR doubled after the addition of 10 phr of fibers without significant loss in the tensile strength or the elongation at break. The hardness was increased by 47%, and the storage modulus also increased, indicating the excellent interactions between the surfaces of the fibers and the NR matrix.

**Keywords:** latex, natural rubber, jute fibers, composites, mechanical properties

## 1. Introduction

Vulcanized natural rubber has several properties that are superior to those of synthetic rubbers, such as high elasticity and high tensile strength<sup>1,2</sup>. Such properties make it an exceptional material for use in several technological products<sup>2</sup>. However, there has been growing interest in the substitution of the synthetic fillers, which are widely used in industrially manufactured rubber composites, by natural fillers that are extracted from a renewable sources because of the sustainability of natural rubber<sup>3-5</sup>.

Natural fibers may be suitable as reinforcements in the natural rubber matrix if they have a tensile strength comparable to those of synthetic fillers, such as glass and carbon fibers<sup>1,5</sup>. Jute fiber is a commercially available and cheap natural fiber; furthermore, it has a high cellulose content<sup>6,7</sup>. To use these lignocellulosic fibers in polymeric composites, removal of the lignin and other amorphous materials is necessary to allow the separation of the fibrils on their structure<sup>4,7</sup>. The most common procedure to remove these materials is alkali treatment using a high-concentration sodium hydroxide solution<sup>4,5,8</sup>. The alkali-treated fibers can be used as reinforcement for different types of polymer composites, such as polypropylene<sup>9,10</sup>, epoxy resin<sup>8,11,12</sup> and poly (lactic acid)<sup>13,14</sup>. These studies have shown improvement in the mechanical properties of the polymer matrices with a fiber content of less than 20%.

Natural rubber composites containing natural fibers have been prepared using a variety of methods, for example, using a laboratory two-roll mixing mill<sup>15,16</sup> and a Brabender batch mixer<sup>17</sup>, and the properties of these composites have

been analyzed. Pittayavinai *et al.*<sup>15</sup> studied the effect of the manipulation of the crosslinking density of the fiber-containing natural rubbers. For some formulations, there was an increase in the tensile strength compared to natural rubber. Formela *et al.*<sup>17</sup> studied the properties of natural rubber and wheat bran biocomposites. This study reported an increase in the tensile modulus with the addition of fillers, explaining that the fibers can cause strain-induced crystallization in natural rubber. The dispersion of the fibers in the composites, however, remains challenging because they tend to agglomerate<sup>5</sup>. For nanoparticle dispersions in natural rubber composites, an effective method for the dispersion of particles in latex has already been demonstrated<sup>1,18-20</sup>.

The objective of this work was to incorporate and disperse treated jute fibers in latex and to evaluate the mechanical, dynamic mechanical, and morphological properties of the vulcanized composite materials.

## 2. Materials and Methods

### 2.1. Materials

High-ammonia natural rubber latex was purchased from BDF Ltda (SP, Brazil), while the raw jute fiber was supplied by Castanhal (PA, Brazil), and the styrene phenol antioxidant oil was supplied from Proquitec Ltda (SP, Brazil).

### 2.2. Treatment of the jute fibers

The jute fibers were milled using a mechanical milling machine with 0.5 mm sieve opening. The fibers (30 g) were dispersed in deionized water at 70 °C and stirred for 3 h to remove the soluble extractives. The fiber bleaching procedure

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was based on the TAPPI T19m-54 standard method, as described by W. G. Trindade *et al.*<sup>21</sup> After washing and filtering, the fibers were dispersed in deionized water at 70 °C, and sodium chlorite (25 g) and glacial acetic acid (10 mL) were added to the aqueous suspension. The system was vigorously stirred for 1 h. This procedure was repeated twice, that is, sodium chlorite and glacial acetic acid were added again after a 1-h interval. After the final addition of the reagents, the system was stirred for 3 h. The total reaction time was 5 h. After washing and filtering, the fibers were subjected to an alkaline treatment to remove the acid residues<sup>10</sup>. The fibers were then immersed in a 2% NaOH (w/w) solution, stirred at 80 °C for 2 h, filtered until a pH of 7 was obtained, and, finally, dried in a vacuum oven at 60 °C for at least 24 h.

### 2.3. Preparation of the NR/bleached jute fibers composites

The NR/bleached jute fibers (NR-BJF) composites were obtained by mixing an aqueous suspension of treated fibers (2 wt.%) with NR latex (60 wt.%) so that the fiber concentrations reached 2.5, 5, and 10 phr. The mixtures were dispersed using an Ultra Turrax dispersor (IKA) at 10,000 rpm for 5 min. Coagulation was then performed by adding sufficient acetic acid (3 mol/L) to small latex portions until the solidification of the rubber was complete. Next, the rubbers were immersed in deionized water for five days. The rubber mixtures were prepared in a laboratory two-roll mixing mill. First, the NR was masticated for 5 min, and, then, the components shown in Table 1 were added in the order presented, with mixing times of 5 min for each material. The samples were then cured at 160 °C in an electrically heated hydraulic press at 20 MPa for their respective vulcanization times ( $t_{90}$ ). These values were determined using an oscillating disc rheometer ODR 2000 (TEAM, Brazil) based on ASTM D-2084-11.

### 2.4. Methods

The X-ray diffraction (XRD) analysis of the raw jute fibers and the treated fibers were performed in an XPERT-MPD diffractometer, with Cu- $K_{\alpha}$  radiation ( $\lambda = 1.544 \text{ \AA}$ ). The scattered radiation was detected in a  $2\theta$  range of 5 to 40° at a scanning rate of 0.02°/min. The crystallinity index (CI) was estimated using Equation 1<sup>22,23</sup>, where CI expresses the relative degree of crystallinity,  $I_{200}$  the intensity of the peak corresponding to the plane (200), and  $I_{am}$  is the intensity of the diffracted peak at  $2\theta = 18^\circ$ .

$$CI(\%) = \frac{I_{200} - I_{am}}{I_{200}} \times 100 \quad (1)$$

To observe the surface morphology of the raw jute fibers, the treated fibers, the natural rubber, and the fracture surface of the composites, the samples were coated with a thin layer of conductive material (gold) and observed using a field-emission scanning electron microscope (FEI, INSPECT F50 SEM-FEG) at an applied accelerating voltage of 15 kV.

**Table 1.** Basic formulation of natural rubber compounds.

Material	Parts
Natural rubber	100
Bleached jute fibers	0-10
Antioxidant oil <sup>a</sup>	1.5
Zinc oxide	4
Stearic acid	1
M.B.T.S. <sup>b</sup>	1
T.M.T.D. <sup>c</sup>	0.5
Sulfur	1.5

<sup>a</sup> Styrene Phenol

<sup>b</sup> 2,2' Dibenzothiazyl Disulfide

<sup>c</sup> Tetramethylthiuram Disulfide

The NR composite samples were cryogenically fractured. The sample hardness was determined using a Shore A-type durometer. Compound samples (6-mm thick) were used to carry out measurements. Tensile tests were performed based on the ASTM D412-15a standard on a universal testing machine (Alpha Technologies, T2000) fitted with a contact-style extensometer. A crosshead speed of 500 mm/min and 1 kN load cell were used.

Dynamic mechanical thermal analysis (DMTA) was performed on a DMTA instrument (DMA 800, PerkinElmer) using rectangular samples with dimensions of 12 × 8.5 × 2.1 mm. The experiment was carried out at a frequency of 1 Hz from -100 to -12 °C at a heating rate of 2°C/min and an oscillation amplitude of 50  $\mu$ m.

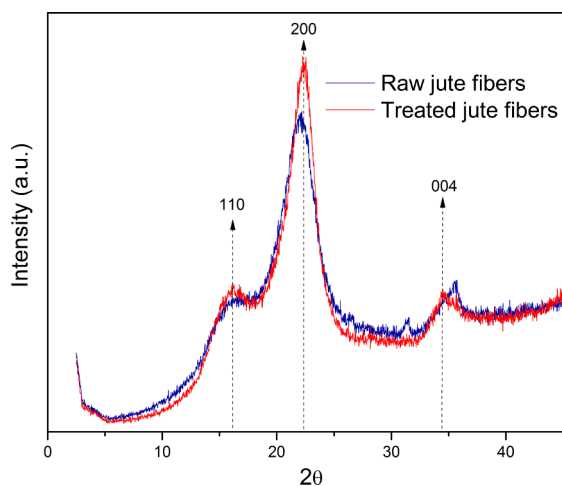
## 3. Results and Discussion

### 3.1. X-ray diffraction of fibers

The X-ray diffraction curves of raw and treated jute fibers are shown in Figure 1. Peaks at approximately  $2\theta = 16, 22, \text{ and } 34.5^\circ$  arise from the cellulose (110), (200), and (004) planes, respectively. This indicates the presence of type I cellulose in the fibers, both before and after bleaching treatment<sup>23-25</sup>. The increase in the peak intensity of the (200) reflections indicates that the crystallinity has increased. Furthermore, the small change in  $2\theta$  from 22.1 to 22.4° indicates the transformation of cellulose  $I_{\beta}$  into cellulose  $I_{\alpha}$ , promoted by the alkali treatment; this occurs even at low concentrations of NaOH, such as that used in this work. Such transformations have also been reported in the literature<sup>25,26</sup>. The crystallinity indices for the raw and treated fibers were 58 and 66%, respectively. The results confirm the increase in the crystallinity of the jute fibers after the bleaching process, which may contribute to its applications in polymer composites<sup>8,27,28</sup>.

### 3.2. Surface morphology of the fibers

Figure 2 shows SEM micrographs obtained for the fibers, both raw and after the chemical treatment. The SEM



**Figure 1.** X-ray diffraction curves of raw and treated jute fibers.

micrographs show the differences in the fiber surfaces by comparing images taken before and after the bleaching process. The surfaces of the treated fibers are smoother and contain fewer solid aggregates when compared to the surface of the raw fibers. Furthermore, the treated fibers show less agglomeration, which may indicate that the bleaching treatment was able to remove a significant amount of the amorphous mass from the fiber surfaces, such as lignin, impurities, and low molar mass polysaccharides, as has also been previously reported<sup>23,29,30</sup>.

### 3.3. Vulcanizing behavior

The vulcanizing behavior of the investigated compounds is presented in Table 2. As expected, the addition of fibers to NR increases the minimum ( $M_L$ ) and maximum torque ( $M_H$ ) values of the composites. The vulcanization times obtained for all compositions were similar. As reported<sup>15,17,31</sup>, the torque values of natural rubber increased with the fiber addition,

**Table 2.** Vulcanizing behavior of the compounds at 160°C.

Sample	$M_L$ (dNm)	$M_H$ (dNm)	$t_{90}$ (min)
NR	2.4	18.6	6.22
NR + 2.5 BJF	3.6	23.7	6.67
NR + 5 BJF	3.9	24.6	6.93
NR + 10 BJF	4.2	24.8	6.52

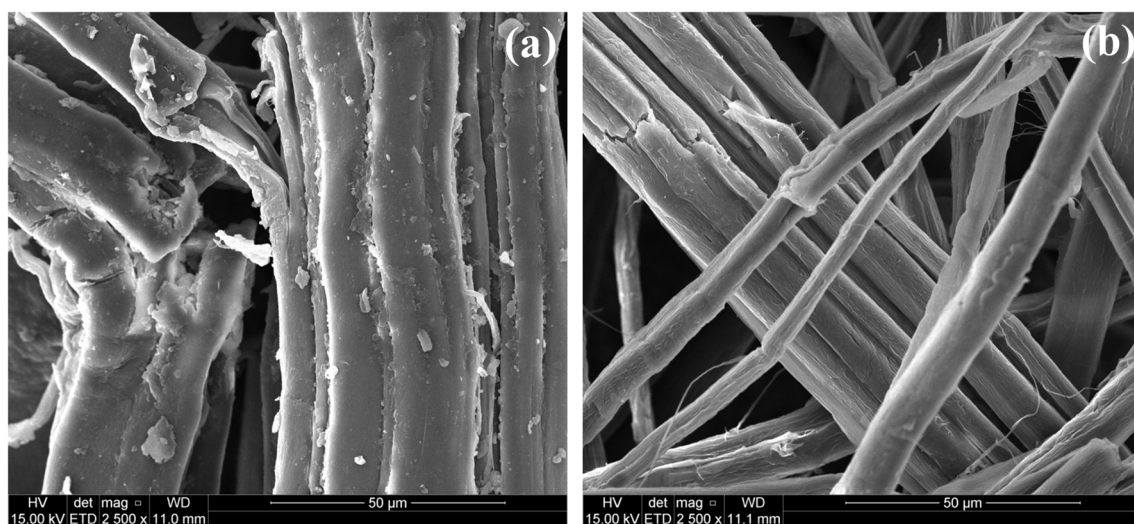
indicating an increase in the stiffness of the vulcanized compounds. This is due to the restriction in the mobility of the rubber molecules caused by the fibers<sup>31</sup>. However, studies have shown<sup>17,31</sup> that the insertion of fibers generally decreases the vulcanization times of the rubber composites. That is because, when fibers are added during processing, the mixing time must be increased to obtain adequate homogeneity. Thus, there is an increase in temperature that favors the prevulcanization reactions on the mills<sup>17,31</sup>. In this work, because the fibers were added during the coagulation step, a decrease in  $t_{90}$  was not observed. The  $t_{90}$  values obtained in this test were used for the vulcanization of the composites produced in this work.

### 3.4. Hardness

Table 3 lists the hardness values of the samples. As expected, the hardness of the composites increased with increasing NR fiber content. Studies<sup>15,32</sup> have reported that the addition of fibers causes a significant increase in the hardness of natural rubber. This is typical of traditional composites, confirming the increase in the stiffness of the compounds.<sup>17</sup>

### 3.5. Mechanical properties

The mechanical properties of bleached jute fibers and NR-based composites are presented in Table 3. A slight decrease in the tensile strength, from 17 to 15 MPa, was observed on the addition of 10 phr of fibers to the natural rubber. Other



**Figure 2.** SEM micrograph (x2,500) of raw (a) and bleached (b) jute fibers.

**Table 3.** Mechanical properties of the samples.

Sample	Tensile strength (MPa)	Elongation at break (%)	$M_{100}$ (MPa)	$M_{300}$ (MPa)	Hardness (shore-A)
NR	17 ± 2	692 ± 33	0.78 ± 0.07	1.68 ± 0.11	37.3 ± 0.3
NR + 2.5 BJF	17 ± 1	715 ± 23	1.13 ± 0.06	1.90 ± 0.05	43.2 ± 0.6
NR + 5 BJF	18 ± 2	692 ± 15	1.47 ± 0.05	2.19 ± 0.05	51.3 ± 0.3
NR + 10 BJF	15 ± 1	691 ± 15	2.10 ± 0.1	2.60 ± 0.10	± 0.5

studies<sup>31-33</sup> have also shown a reduction in both the tensile strength and elongation at break when raw and treated natural fibers were added to natural rubber. However, in this work, the values of elongation at break were not affected by the increasing quantity of fibers.

The values of the elastic modulus at 100 and 300% elongation ( $M_{100}$  and  $M_{300}$ ) underwent a slight increase on the addition of fibers, indicating that the fibers increased the stiffness of the natural rubber<sup>32</sup>. These results corroborate those reported in the literature<sup>32,34</sup>, which indicate that the use of natural fibers as a reinforcement in natural rubber matrices increases the tensile modulus; this may be indicative of the excellent interactions between the fiber surface and the natural rubber matrix.<sup>32,33</sup>

### 3.6. Dynamic mechanical thermal analysis (DMTA)

The storage moduli for natural rubber and the composites are shown in Figure 3. An increase in modulus was observed with increasing fiber content. Similar studies<sup>9,35,36</sup> have reported that an increase in fiber content increases the storage modulus of the polymer matrix. This effect was more pronounced for the addition of 5 phr jute fibers, and the addition of 2.5 phr practically did not increase the storage modulus. The DMTA results corroborate those obtained for the tensile modulus and, again, indicate that the addition of fibers contributed to the increasing stiffness of the NR.

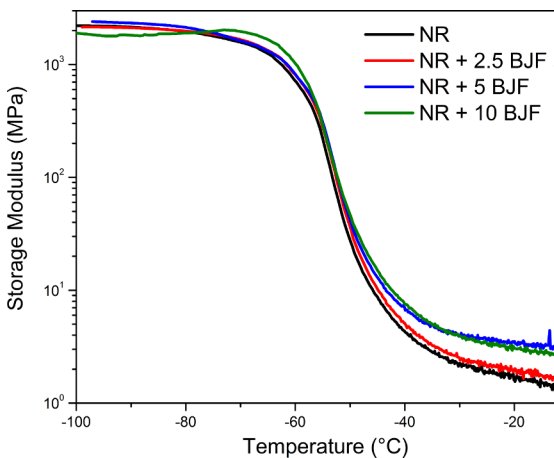
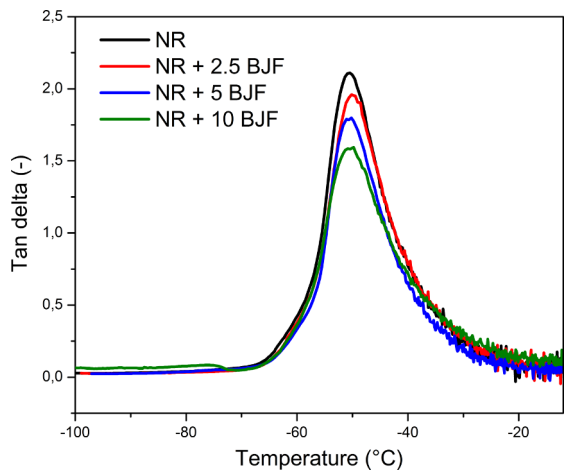
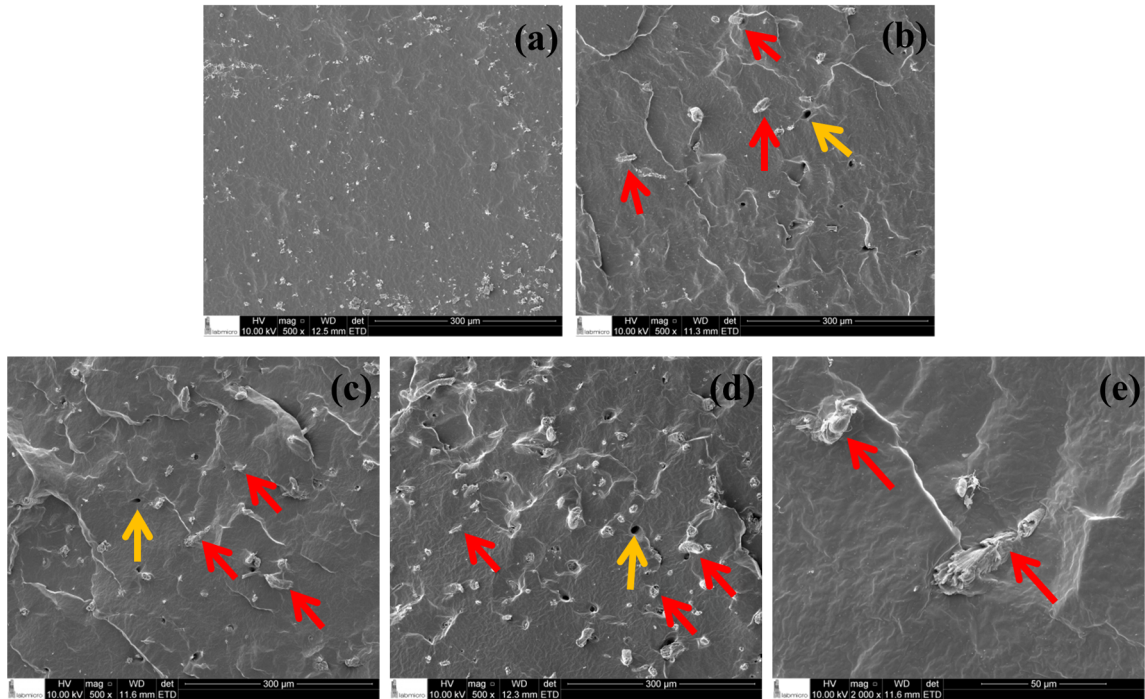
**Figure 3.** Storage Modulus versus temperature for NR composites.

Figure 4 shows the mechanical loss factor (tan delta) curves for the NR composites. A decrease in the tan delta peak was noted as the fiber amount increased. This occurs because of the decrease in the mobility of the polymer chains on the addition of the fibers. This trend suggests that fibers and the natural rubber matrix have a strong interfacial adhesion<sup>36,37</sup>. On the other hand, based on the tan delta curves, it was also possible to determine the glass transition temperature ( $T_g$ ) of the compositions, which were -50.5, -49.8, -50.6, and -50.4 °C, for natural rubber and the composites with 2.5, 5, and 10 phr fibers content, respectively. The presence of fibers did not significantly alter the  $T_g$  of the natural rubber.

**Figure 4.** Tan delta versus temperature for NR composites.

### 3.7. Morphology of the NR composites

Figure 5 shows the SEM images for NR and for composites with 5 and 10 phr of fibers. As the fiber content increased, the number of particles dispersed in the matrix increased. The pure NR sample contains particles related to the vulcanization additives, which are smaller in size than the fibers indicated by red arrows in Figures 5b, 5c and 5d. Cavities (yellow arrows), which are probably related to the pullout of fibers during the cryogenic rupture of the sample, are also visible. Compared to the particulate dispersions observed in similar studies<sup>12,32</sup>, those of the composites presented here are finer, indicating that the method of fiber incorporation into NR was efficient.



**Figure 5.** SEM images of: (a) NR, (b) NR with 2.5 phr, (c and e) NR with 5 phr and (d) 10 phr of fibers.

## 4. Conclusions

The morphological analysis of the jute fibers indicated that the bleaching process was satisfactory because the solids were removed from the surface. Using XRD analysis, we found that the treated fibers showed higher crystallinity than the raw jute fibers, confirming that the treatment removed some of the amorphous cellulosic fibers. The composites were obtained by incorporating the bleached fibers into the latex, followed by processing in a two-roll mixing mill. Using tensile and DMTA analysis, we found an increase in the storage modulus and in the elastic modulus at 100 and 300% elongation on the addition of fibers to the NR, indicating the increased stiffness of the natural rubber. Morphological analysis of the composites showed that there was a fine dispersion of the fillers, which may indicate strong interactions between the fiber surfaces and the natural rubber matrix. This study showed that the method of incorporating fibers into latex is efficient, resulting in natural rubber composites with good properties.

## 5. Acknowledgements

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