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Hybrid Composite Based on Natural Rubber Reinforced with Short Fibers of the *Triumfetta cordifolia*/*Saccharum officinarum* L.: Performance Evaluation

Abel Emmanuel Njom[1,2,3], Armel Mewoli[1,4], Marie Josette Ndengue[1,4], Fabien Betene Ebanda[1,4], Augustine Demze Nitidem[1,5], Sandrine Biloa Otiti[1,4], Yannick Devario Youssi Bang[1,4], Legrand Ndoumou Belinga[1,3,4], Pierre Marcel Anicet Noah[1,4], Atangana Ateba[1,4]

[1]Department of Mechanical Engineering, ENSET, University of Douala, Douala, Cameroon
[2]Laboratory of Materials Mechanics, Structures and Integrated Manufacturing, National Advanced School of Engineering, University of Yaoundé 1, Yaoundé, Cameroon
[3]Laboratory of Engineering Civil and Mechanics, National Advanced School of Engineering, University of Yaoundé 1, Yaoundé, Cameroon
[4]Laboratory of Mechanics, University of Douala, Douala, Cameroon
[5]Mechanics and Adapted Materials Laboratory (LAMMA), ENSET, Douala, Cameroon
Email: mewoliarmel@yahoo.fr

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Abstract

This article contributes to the development of the new class of fully biodegradable "green" composites by combining fibers (natural/bio) with biodegradable resin. The vegetable fibers (*Triumfetta cordifolia* and sugarcane bagasse) treated with NaOH and bleached were incorporated into a natural rubber matrix. The influence of the fiber ratio on the physical properties, tensile strength and surface hardness of the hybrid composites was analyzed. The results show that the addition of fibers in the natural rubber matrix increases the water absorption capacity but gradually reduces it with increasing fiber ratio. The hybrid composites of the NRT50-50B proportions show the best tensile strengths at 20 phr and a shore A hardness of 43.7 at 30 phr. The combination of two fibers has improved the physical and mechanical properties of the hybrid composites which can be used in engineering applications.

Keywords

Hybrid Composites, *Triumfetta cordifolia* Fiber, Sugarcane Bagasse Fiber, Natural Rubber Shore A Hardness

1. Introduction

Research efforts are currently underway to develop a new class of fully biodegradable "green" composites by combining (natural/bio) fibers with biodegradable resins [1] [2] [3]. The appeal of green composites including natural rubber and natural fibers is that they are environmentally friendly and biodegradable.

The rubber tree grows in a tropical climate, particularly in Cameroon where many rubber plantations are exploited. This country produced more than 70,000 tons of rubber in 2021, far behind Côte d'Ivoire [4] [5], but the characteristics of this natural rubber (NR) are poorly documented in the literature. NR is one of the main elastomers and is widely used to prepare many rubber compounds [6]. It does not naturally possess the hardness and modulus necessary for commercial acceptability [7].

Green reinforcements, including vegetable fibers, are an alternative to carbon black [8]. Indeed, vegetable fibers offer a low environmental impact, in addition to being renewable, widely available and healthy; they have relatively high specific mechanical properties combined with a low density [9] [10] [11] [12] [13]; they pose technological problems because they present low compatibility with the rubber matrix and are, an incompatibility related to their hydrophilic character. Indeed, the fibers are coated in surfaces of waxy substances (waxes and pectins) of dusts… which can involve a reduction of the tensile strength [14] without however forgetting the presence of the hydroxyl groups responsible for the hemicelluloses. On this last example, the fiber of *Triumfetta cordifolia* contains a very high rate of hemicellulose (30.8%) [9] which requires treatment before use in such a matrix. These problems are mainly minimized by the use of coupling agents to increase the adhesion between the fiber and the rubber matrix or by the application of chemicals to modify the surfaces of the fillers, thus minimizing water absorption and improving the mechanical properties of the composites [15]. For this purpose, alkaline treatment with sodium hydroxide (NaOH) solution of the fibers improves the adhesion characteristics due to the increase in roughness [16] [17]. In addition, the removal of lignin and other waxy surface substances by alkaline solution increases the interfacial adhesion between natural fibers and polymer matrices [17] [18] [19] [20]. Although very rare in the literature, hydrogen peroxide treatment has also attracted the interest of researchers due to the simplicity of its implementation and the improvements observed on the mechanical properties of the tested materials [21]. The investigations of Ramlee *et al.* [21] also corroborate this result by SEM on palm nut and sugarcane bagasse fibers; this treatment presented the appearance of pores and fibrils on the fiber surface, which would demonstrate a thermal efficiency. Thus, the effective surface area of the fiber available for binding with the matrix is increased.

In parallel with the development of green composites, studies indicate that this availability remains low since flax, hemp and coir fibers represent more than 80% of the world's production of natural fibers excluding cotton [22] [23]. There is a need to diversify the plant resources of reinforcements of rubber composites.

To this end, the orientation of local biomass is an opportunity for African countries in the equatorial region to develop new fibers such as *Cola lepidota* (CL) bark [10] [11], fibers from *Rhectophyllum camerunense* (RC) [24] and *Neuropeltis Acuminatas* (NA) [25] and *Triumfetta cordifolia* (TC) bast fiber [3]-[9] *Triumfetta cordifolia* bast fibers traditionally used to produce ropes are widely available and sugarcane (*Saccharum officinarum* L.) bagasse fibers mainly used as fuel (in distilleries or sugar mills) [26] are available in Cameroonian biomass and the latter could be very interesting as reinforcement of the natural rubber matrix as they present a potential for many technical applications [3]-[27].

However, the use of natural rubber (NR) as a green matrix can be considered disadvantageous because the mechanical properties of the final composites at very high strains remain rather poor [28] [29]. This last point can be overcome by using a hybrid reinforcement. With such an appropriate material design, one type of fiber can compensate for aspects of performance that are lacking in the other [18]. Hybridizing these two different fibers (TC, Bagasse) would be a more effective way to improve the mechanical properties of natural fibers [22]-[30]. Therefore, these modern materials require a hybrid combination that cannot be satisfied by monolithic materials, especially for the development of automotive materials (Nair & Joseph, 2014). These materials have many advantages over conventional materials. Most of the hybrid materials consist of lightweight components with significantly better mechanical properties as noted in the literature [18] [28] [29] [30] [31].

Although many studies have been conducted on fiber-reinforced polymer systems, as mentioned above, the mechanical properties of lignocellulosic fiber-reinforced natural rubber hybrid composites have received little attention.

The main objective of this work is to investigate the effect of hybridization of plant fibers (*Triumfetta cordifolia*/Bagasse of sugarcane) on the physical and mechanical properties of the developed natural rubber matrix composites. Thus, the bulk density and water absorption rate were evaluated. Tensile tests were performed according to EN-ISO 37:1998 and finally shore hardness (A) tests were conducted.

2. Materials and Methods

2.1. Raw Materials

Two types of plant fibers are used in this work (**Figure 1** and **Table 1**): *Triumfetta cordifolia* (TC) bast fibers harvested in Mbankomo-Cameroon and extracted by water retting in accordance with the method used in the literature [9] and sugarcane bagasse (BS) fibers harvested in Kyossi-Cameroon. These fibers were reduced to particles and sieved (0.1 to 3 mm). Raw rubber latex was collected from Hevecam rubber plantations in the coastal region of Cameroon. Vulcanizing materials such as zinc oxide, sulfur, stearic acid and acetic acid were supplied by PYCNOLAB (Douala, Cameroon). Sodium hydroxide (NaOH) granules used for alkali treatment were supplied by the same facility.

Table 1. Physical and mechanical properties of plant fibers used [3].

Fibers	Characteristics of the vegetable fibers							
	Length (cm)	Density (g/cm^3)	Tensile stress (Mpa)	Modulus of elasticity (Gpa)	A (%)	Cellulose (%)	Hemicellulose (%)	Lignin (%)
TC	0.1 - 1.3	1.47	526 ± 12	12.4 ± 6.9	2.25	44.4	30.8	18.9
BS	-	1.23	220	26.8	1.0	36.9	22.67	23

Figure 1. Some images of the fiber extraction process of the study. (a)—TC plant, (b)—TC bark, (c)—Extracted TC fibers, (d)—Sugar cane stalk, (e)—Bagasse, (f)—Extracted BS fibers.

2.2. Fiber Treatment

The TC and BS fibers were ground into small sizes and then sieved to have lengths of approximately 0.1 to 3 mm. These fibers were treated to improve their adhesion with the matrix [18]. These plant fibers were soaked for 24 h in the 5% sodium hydroxide (NaOH) solution for alkaline treatment [32] [33]. After that, the treated TC and BS fibers were washed to pH7. Then, the fibers were bleached with peroxide (H_2O_2) for 2 hours. Finally, after drying at 70˚C for 24 h, the fibers are ready to be characterized for application in composites.

2.3. Characterization of Materials

2.3.1. FTIR-ATR Spectrometry

FTIR analysis is performed using a Shimadzu IRAffinity-1 CE instrument equipped with a diamond crystal operating in ATR mode. The ground fiber sample was placed on the crystal and then pressed with a flat-tip piston (150 N). The IR spectra were analyzed in transmission mode and recorded in a wave range of 400 to 4000 cm^{-1}, by an accumulation of 24 scans and with a resolution of 4 cm^{-1} in a room with a temperature of 23˚C relative humidity 65% RH.

2.3.2. Determination of the DRC

The DRC was performed according to the two standards, ISO 124 for total solids

content (TMST) and ISO 126 for dry rubber content (DRC) of the latex concentrate respectively. The determination of dry rubber content (DRC) is calculated using Equation (1). A test sample of latex concentrate is diluted to 20% (by mass) of total dry extract and acidified with acetic acid. The coagulated rubber is then formed into a sheet and dried at 70˚C ± 5˚C.

$$\mathrm{DRC} = \frac{m_1}{m_0} \times 100 \tag{1}$$

where m_0 = mass in grams of the test sample; m_1 = mass in grams of the dried leaf.

2.3.3. Examination of the Microstructure by Scanning Electron Microscopy (SEM)

Scanning electron microscope observations were performed on the longitudinal surfaces of treated and untreated TC and BS fibers. These samples were coated with a thin layer of gold by sputtering with an Edwards metalizer before being analyzed with a HITACHI TM 3000 Scanning Electron Microscope (SEM) with an accelerating voltage of 15 kV.

2.4. Manufacturing of Biocomposites

All materials were weighed and mixed according to the ratios shown in **Table 2**. Mixing of the composite components was performed using a twin-screw mixer rotating in opposite directions. The rotor speed and roll spacing were set at 45 rpm and approximately 3 mm, respectively. The initial mixing temperature was maintained at about 70˚C. The mixed TC/BC/NR plant fiber composites were pressed using a 40-ton hydraulic press with platens heated to 150˚C and shaped into composite sheets (**Figure 2(a)**) with dimensions of 150 mm × 200 mm and the thickness of 3 mm was fixed by the use of the metal wedges after dehumidification for 2 hours at 65˚C. Once the sample sheet was ready, it was cut according to the requirements of the test sample (**Figure 2(b)** and **Figure 2(c)**).

2.5. Water Absorption Test

Ten 20 mm × 20 mm × 3 mm specimens for different composite formulations were prepared according to ASTM D570. The biocomposite specimens were previously dried in an oven at a temperature of 60˚C for 24 h. They were immersed in distilled water for eight days and weighed every 24 h using a balance with an accuracy of 10^{-3}. They were immersed in distilled water for eight days and weighed every 24 hours using a balance with a precision of 10^{-3}. The water absorption rate was calculated using Equation (2).

$$Abs = \frac{W(t)_f - W(t)_i}{W(t)_i} \times 100 \tag{2}$$

where $W(t)_f$ is the mass of the sample at the time *t*, $W(t)_i$ initial mass of a test specimen.

Table 2. Composition of Natural rubber mixtures.

Ingredient	Composition (phr)		
	NR40T-60B	NR50T-50B	NR60T-40B
NR	100	100	100
Stearic acid	2	2	2
Zinc oxide	2	2	2
Acetic acid	5	5	5
Short vegetable fibers (0.1 - 3 mm)	0, 10, 20, 30	0, 10, 20, 30	0, 10, 20, 30
Sulfur	5	5	5
Soya oil	2	2	2

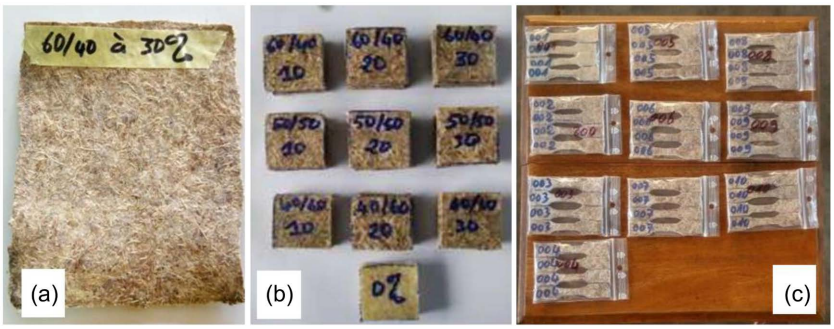

Figure 2. Some images of the fabricated hybrid composites. (a)—sample sheet, (b & c)—sample for testing.

2.6. Determination of Density

The density of the developed composites was determined in accordance with the NF P 94-054 standard. The composites were cut to dimensions of 30 mm × 30 mm × 3 mm. For this test five samples were prepared. The density was calculated using Equation (3):

$$\rho_c = \frac{m_c}{L \times l \times e} \tag{3}$$

where m_c is the mass of the composite in (kg), L the length in (m), l the width in (m), e the thickness in (m) and ρ_c the apparent density in (kg/m^3).

2.7. Mechanical Properties

2.7.1. Mechanical Properties in Traction

The tensile elastic properties (tensile strength and elongation at break) of the composite sheets were characterized according to EN-ISO 37:1998 using a universal tensile testing machine (LDW-1, China) in the shape of a dumbbell. The tensile speed was 500 mm/min. Four different dumbbell-shaped specimens (**Figure 2(c)**) with an average thickness of 3 mm over the measurement length were tested, and the average value for each formulation was reported.

2.7.2. Hardness Test

The hardness of the samples was determined using a Shore A hardness tester with a Leeb hardness tester model TH110g equipment used by Tamwo *et al.* [34]. Composite samples (50 mm × 50 mm × 3 mm) were used to perform the measurements.

3. Results and Discussion

3.1. Examination of the Morphology of Plant Fibers

Figure 3(a) and Figure 3(b) show the longitudinal surface of the plant fibers in the study before and after alkaline treatment. It is possible to see in Figure 3(a) and Figure 3(b) the presence of impurities and small rough areas on the raw fiber surface of TC and BS. These can lead to a decrease in the tensile strength [14]. With chemical treatment, the surfaces that were covered with impurities become clean. This surface cleaning has also been observed in the literature [17] [18] [19] [20]. However, the treated TC fiber does not show a tilt angle unlike BS fibers which show micro-fibrillar angles. These phenomena observed on the surfaces of treated fibers (Figure 1(c) and Figure 1(d)) would contribute to improving the adhesion characteristics due to the increase in roughness which is rather beneficial for adhesion.

3.2. Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectra were used to highlight the effect of chemical treatment on TC and BS plant fibers presented in Figure 4. In this study, an emphasis is placed on the functional groups of lignin, hemicellulose, cellulose which have more influence on the mechanical properties.

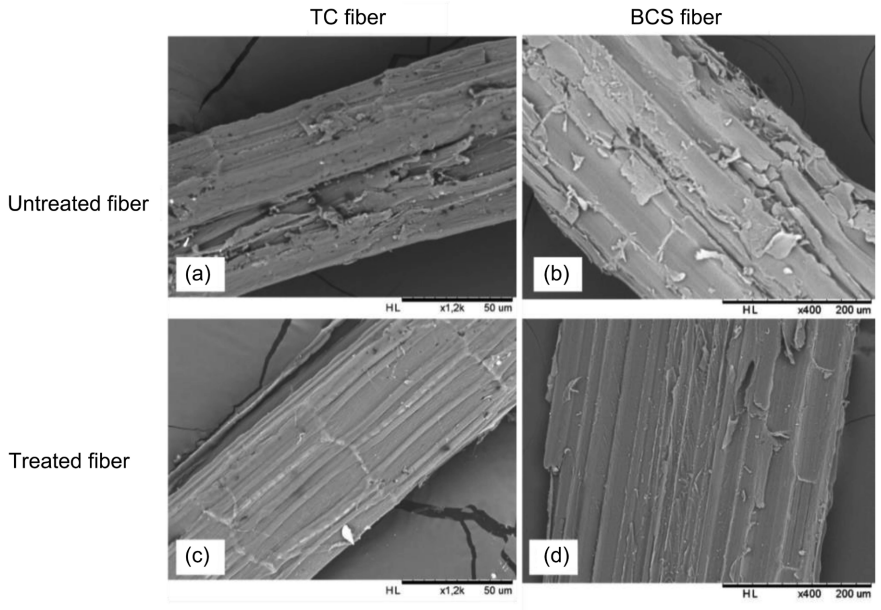

Figure 3. Plant fibers studied: (a) raw TC fiber, (b) raw BS fiber, (c) treated TC fiber, (d) treated BS fiber.

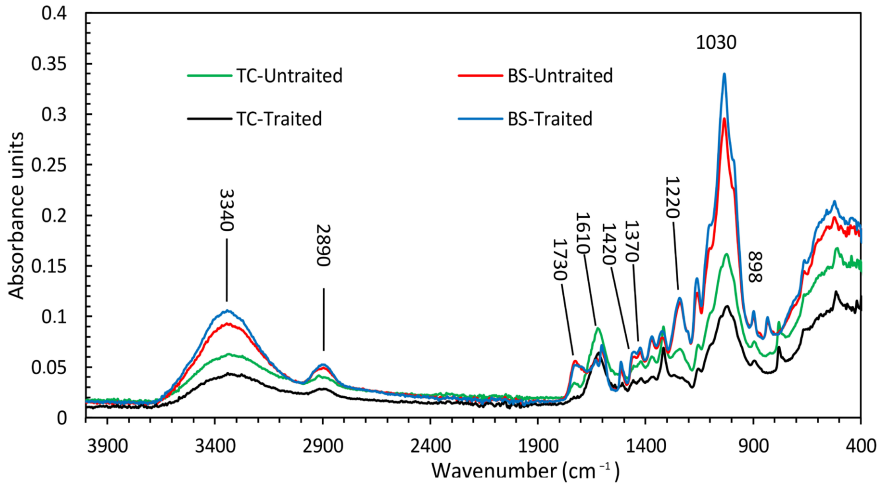

Figure 4. FTIR spectra of TC and sugarcane bagasse fibers before and after chemical treatments.

The FTIR spectra are presented in this figure, and show particularly seven characteristic peaks; the first with a length of about 3345.33 cm^{-1} represents the hydrogen bonds (O-H) of cellulose and the hydroxyl groups of hemicellulose [10]. An absorption band characteristic of the presence of liquid water is more or less bound to the polymeric network constituting natural fibers. The value 1726.23 cm^{-1} represents that characteristic of the bond of the stretching of the carbonyl C=O of the carboxylic acid. It could also possibly be the ester representing the non-cellulosic group internal to pectins, waxes and acetyl groups present in hemicelluloses. The latter is attenuated on the TC fiber by the chemical treatment. Peaks 1604.08 cm^{-1} and 1514.10 cm^{-1} represent lignin, characteristic of C=C stretches of the aromatic backbone of lignin. The absorption band at 1424.86 cm^{-1} is associated with the CH_2 symmetric bending present in cellulose [10]-[24]. The two peaks presented at 1370.80 cm^{-1} and 1318 cm^{-1} respectively to the bending vibration of (C-H) and (C-O) groups of the aromatic ring in polysaccharides. A reduction of this peak is observed after treatment on the TC fiber. The absorption band 1239 cm^{-1} and 1159.68 cm^{-1} corresponds to the (-COO) group of the hemicellulose. Finally, the peak at 897.49 cm^{-1} can be attributed to the β-1,4 glycosidic bonding of the (C-O) groups of cellulose [10]. This analysis demonstrates not only that sugarcane bagasse and *Triumfetta cordifolia* that the different treatments influence the structure of TC and BCS fibers as observed in the literature [21]. This confirms the increase in the percentage of cellulose in the fibers, especially after hydrogen peroxide bleaching.

3.3. DRC Content

The results of the DRC of the NR are made in duplicate and agree to within 1% (in mass) of the average value according to the standard. The different DRC obtained show the following values 35.981% and 36.923%, with an average value of 36.452% DRC for all experiments.

3.4. Characterization of Biocomposites

3.4.1. Apparent Density

The bulk density of the composites containing 10, 20, 30 phr of treated TC and BS fibers was analyzed. The TC and BS fibers taken in the mass ratio of 40: 60, 50:50 and 60:40 respectively are presented in **Figure 5**. It is observed that the addition of the plant fibers in the virgin NR matrix lightens the composite materials [36]. This could be beneficial for automotive carpets in particular and the lightening of parts used in automotive interiors in general. It is also observed that there is a decrease in density as the volume fraction of fibers increases in the NR matrix. Hybrid composites with an equal fiber ratio (NR50T-B50) offer a good mass gain compared to the others. This can be justified by the density of the TC fiber reinforcement (1.47 g/cm^3) which is slightly close to that of the BS fibers (1.23 g/cm^3). At the same time, this increased lightness in the composites is also favored by the chemical treatment performed. Indeed, after such treatment, the decrease in the diameter and weight of the fibers is proven due to the removal of lignin, impurities and non-lignocellulosic compounds that the fibers contain on the surface as indicated in the literature [18] [20] [35] [36].

3.4.2. Water Absorption

The trend in the graph (**Figure 6**) indicates that as the fiber volume fraction increases, the value of the water absorption coefficient also increases.

At the same time, the polymer composites with a higher amount of TC fibers (NR60T-40B) showed a lower water absorption value. This suggests that this is the optimal ratio. This, therefore, indicates that the chemical treatment performed on the raw TC fibers decreased the high presence of hydroxyl groups that are responsible for the excessive water absorption and moisture as shown in the FTIR spectra (**Figure 4**) and reported in the literature [9], thus enhancing the good adhesion between the fiber and the NR matrix of our composites. Similar work has shown that by treating the fiber, the hydrophilicity was reduced which improves the interfacial bonding between the fiber and the matrix [14]-[36].

3.4.3. Tensile Behavior

In the present study, the behavior of composites containing 10, 20, 30 phr of

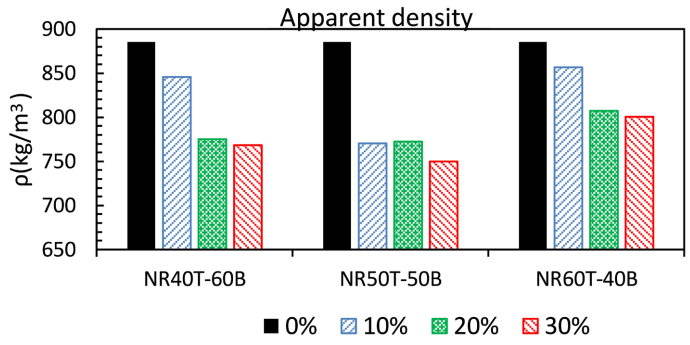

Figure 5. Hardness comparison of NR hybrid composites (TC/BS).

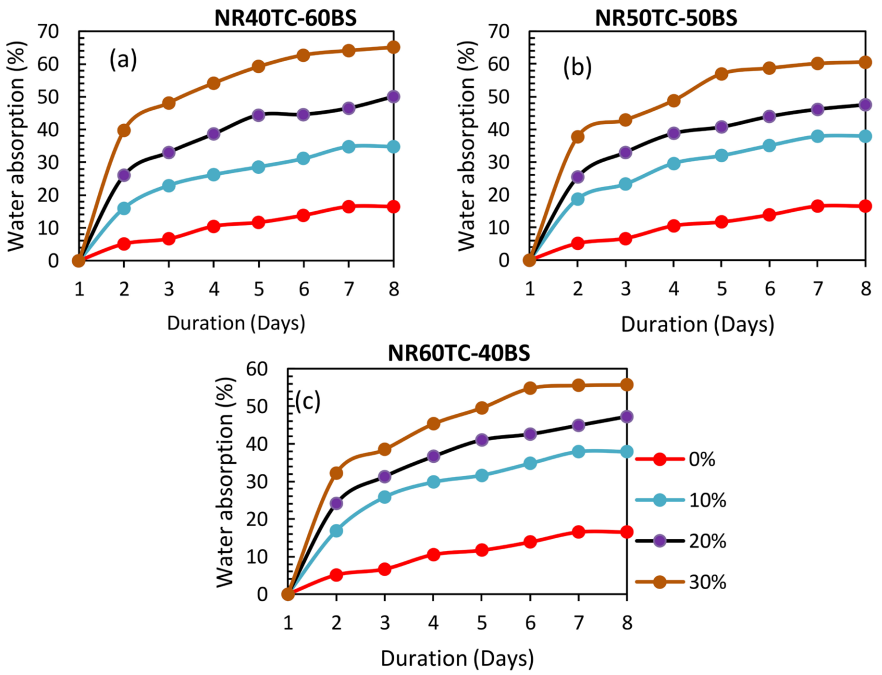

Figure 6. Water absorption behavior of TC/BS hybrid composite as a function of immersion time.

treated TC and BS fibers was analyzed. The TC and BS fibers were taken in the ratio of 40:60, 50:50 and 60:40 respectively. It can be seen that the tensile strength of the virgin NR matrix increased with the addition of the plant fibers. This may be an indication of the excellent interactions between the fiber surface and the natural rubber matrix.

These results corroborate those reported in the literature [37] [38] [41]. From Figure 7, it is clear that the tensile strength increases up to 20 phr of fibers in natural rubber for composites containing an equal fiber mass ratio (TC/BS) (NR50T-50B20). This family of composites has maximum tensile strength (3.74 MPa) and minimum ductility. This suggests that this is the optimal ratio. However, it is also observed that composites containing a dominant TC fiber mass fraction (NR60T-40B) also offer more interesting mechanical properties in tension than those with more sugarcane bagasse fibers in the constitution (NR40T-60B). Indeed, when the concentration of TC fibers increases, the tensile strength also increases. This suggests that the tensile strength of the composite depends more on the weight of the TC fibers, cellulose (44.4%) rather than that of the sugarcane bagasse fibers (22.7% cellulose), which could be due to the high strength of the TC fiber without forgetting their flexibility and their ability to plug the pores well in the composite. In addition, this improvement in properties also depends on the treatments performed on the fibers. Indeed, it has been reported in the literature [18] [28] [37] [39] that the alkaline treatment increases the roughness of the fiber surface and consequently increases the surface available for contact with the matrix.

As for the elongation of hybrid composites, it is found that when the volume

fraction of the fibers also increases, the elongation at break decreases. This reduction indicates that the ductility decreases when TC and bagasse fibers are added to the natural rubber matrix. Similar observations were observed in the work of Moonart & Utara [39]. The literature Manaila *et al.* [40] also reports that this behavior is due to the restriction of molecular chain movement by the fiber.

3.4.4. Hardness

The hardness of composites containing 10, 20, 30 phr of treated TC and BS fibers was analyzed. The TC and BS fibers were taken in the same proportions as before and are presented in **Figure 8**. The maximum hardness is obtained with the addition of the fibers in the NR matrix. This is due to the reduction of elasticity generated by the reinforcement of the molecules [28].

The results also show that NR40T-60B composites have a low hardness and it remains below 40 whatever the fiber content. On the other hand, the incorporation of soft TC fibers at 50% and 60% mass levels in the hybridization with BS fibers in the NR matrix (NR50T-50B and NR60T-40BS) show a gradual increase in hardness as the fiber content increases. The fiber-rubber interphase is strong and better in the composites as well as the dense arrangement in the composite

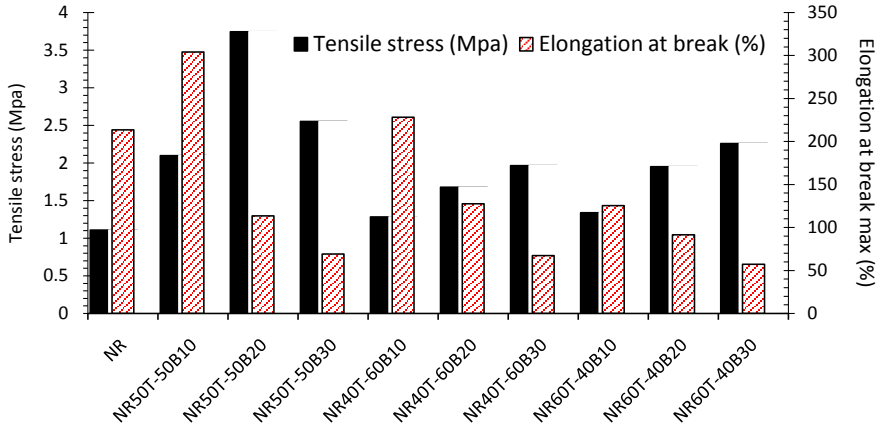

Figure 7. Variation of tensile mechanical properties of hybrid composites (TC/BS): tensile strength and elongation at break.

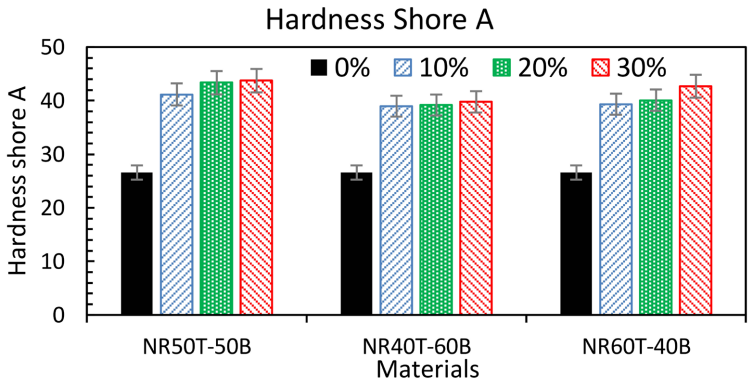

Figure 8. Variation in the Hardness of hybrid composites (TC/BS).

as corroborated by the literature [41]. These composites with strong bonds are harder because they prevent the movement of the matrix in the direction of stress. Thus, the hardness of the developed materials is also improved compared to pure latex.

4. Conclusion

At the end of this study, hybrid biofibers (*Triumfetta cordifolia* and sugarcane bagasse) were incorporated into a natural rubber matrix. The results obtained during the experimental campaign showed that the chemical treatment carried out improved the quality of the fiber-matrix adhesion. The addition of fibers in the developed hybrid composites increases the water absorption capacity but gradually lightens them with increasing fiber shares. Moreover, in terms of mechanical properties, the addition of plant fibers in the virgin matrix of NR improves the tensile strength and hardness but decreases its ductility. The hybrid composites with equal proportion of plant fibers presented better characteristics with optimal ratio of NRT50-50B. In view of the results obtained, the addition of vegetable fibers in the NR matrix is an alternative to carbon black and these materials can find many technical, industrial and commercial applications such as car mats, shock absorbers for doors in the building industry.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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