

Composites of natural rubber with curaua fibers

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Abstract

This paper analyzed the thermal and mechanical properties of natural rubber/curaua fiber composite with different weight contents (2.5% to 15%). Alkali treatment was used to modify the surface of curaua fiber and improve interfacial bonding. Results showed that alkali treatment did not affect the dispersion pattern of composites. TGA revealed that both fiber content and treatment minimally influenced the thermal stability of composites. However, thermal conductivity showed up to 34,2% reductions compared to the matrix for low fiber contents, with an increased tendency for higher contents. The tensile strength of composites showed improvements up to 161% for 2.5% loads, but there was a decreasing tendency when filler load increased, indicating problems with dispersed phase distribution. Overall, composites made with treated fibers exhibited better mechanical properties than those without treatment, showing better interfacial adhesion, and the 2.5% fiber content presented the best combination of thermal and mechanical properties.

Keywords: *curaua fiber, natural rubber, composite, lignocellulosic.*

Data Availability: Research data is available upon request from the corresponding author.

How to cite: Azevedo, J. D. L., Giacon, V. M., & Bortoleto, J. R. R. (2025). Composites of natural rubber with curaua fibers. *Polímeros: Ciência e Tecnologia*, 35(2), e20250015. <https://doi.org/10.1590/0104-1428.20240111>

1. Introduction

The demand for environmentally friendly and low-cost materials has increased over the past decades. This is partly due to society’s growing awareness of the environmental impacts of human activities, driving the use of materials mostly derived from renewable or natural sources. Brazil has a high potential for contribution in this context, as it is a major producer of lignocellulosic fibers, with various species cultivated within its territory^[1].

Among the many natural fibers, curaua fiber (*Ananas erectifolius*), a bromeliad from the pineapple family (*Ananas comosus*), stands out, being typically found in the Amazon region^[2]. The plant was already known to pre-Columbian peoples, who used the fiber to make ropes and fabrics^[3]. In Brazil, rational planting began in the early 20th century along the Amazon River, mainly in Pará, with the peak of cultivation occurring in the municipality of Santarém (PA)^[4].

Typically, curaua leaves can reach up to 1.50 m in height and 4.00 cm in width and are rigid, upright, and flat^[5]. The plant also produces a fruit similar to the common pineapple but smaller and unsuitable for human consumption^[5]. The fiber chemical composition comprises 73.6% cellulose, 9.9% hemicellulose, 7.5% lignin, 0.9% ash, and 7.9% moisture^[4,6]. Its density ranges from 0.57 to 1.43 g/cm³, and it can withstand tensile stresses from 500 to 1150 MPa, placing it among the strongest lignocellulosic fibers^[7-9]. Combining these factors results in a highly resistant, low-

density, renewable, biodegradable, and low-cost fiber with the potential to replace glass fiber in certain applications^[10,11]. Several researchers have used curaua fiber in polymer matrix composites such as EPS (expanded polystyrene), PP (polypropylene), epoxy, or PLA (polylactic acid)^[11-15] and cementitious matrices^[16,17]. In these composites, the fibrous structure of the vegetal fiber tends to improve mechanical performance^[13] and, in some cases, thermal stability^[12,14].

Another abundant natural material with potential for polymer composite fabrication is natural rubber (NR), a completely natural elastomer produced from latex (a milky and viscous fluid) extracted from the rubber tree (*Hevea brasiliensis*), as well as from other species^[18,19]. NR consists of large hydrocarbon chains with a cis-1,4-polyisoprene structural unit, which, due to its high molecular weight, is elastic, has good impact and abrasion resistance, is resilient, and remains flexible at low temperatures, making it a good alternative for use in composites with natural fibers^[19]. Several researchers evaluated NR composites reinforced with lignocellulosic fibers such as jute, sisal, coconut, and bamboo (among others) and observed improvements in properties such as Young’s modulus and hardness^[19,20]. Moreover, some authors point out that natural latex rubber holds strategic relevance because it cannot be fully replaced by synthetic materials in most applications^[19,21]. Due to the widespread use of NR, new combinations of this elastomer with various fillers are being explored, aiming at producing new composites, particularly in efforts to reduce the use of synthetic rubbers^[18].

The major drawback of polymer composites reinforced with lignocellulosic materials lies in the natural fibers' hydrophilic nature, which hinders adhesion between the surfaces of their components. However, the literature indicates that surface treatments can be applied to vegetal fibers to improve bonding between matrix and reinforcement. In this regard, alkali treatment with sodium hydroxide (NaOH), or mercerization, of natural fibers is widely recognized as an easy, practical, and inexpensive method for treating them, potentially improving the mechanical properties of polymer matrix composites^[3,6,8,11,14,19-23]. It's because alkali treatment removes some of the fiber components such as lignin, hemicellulose, pectin, fats, and surface impurities, exposing cellulose and increasing fiber roughness, which can enhance interfacial adhesion^[6,8,24]. Moreover, removing amorphous components in lignin and hemicellulose also improves vegetal fibers' crystallinity index and stiffness^[25-27]. However, alkali treatment with high NaOH concentrations or excessive exposure time (immersion) can cause severe fiber degradation, deteriorating its physical and mechanical properties^[25,28].

A study on curaua fiber and epoxy resin biocomposite evaluated the effects of reinforcement loading and alkali treatment with different immersion times and NaOH concentrations. The results showed a strong influence on the tensile properties of the composite, where higher concentrations require less immersion time, or *vice versa*, to achieve the ideal roughness capable of promoting strong matrix/reinforcement adhesion^[14].

Although there are currently numerous studies interested in the properties of NR/natural fiber biocomposites and their potential applications^[19,23], to date, no research has been identified addressing the interaction between natural rubber and curaua fiber. Therefore, this paper aims to prepare NR composites filled with curaua fiber to analyze the effects of reinforcement loading and alkali treatment with NaOH on selected physical, thermal, and mechanical properties.

2. Materials and Methods

2.1 Materials

Curaua fiber samples, used as filler, were obtained at Novo Airão city (Amazonas - Brazil), produced by hand in riverside communities, without chemical treatment, and sold in local craft centers as rolls of thread for knitting and ropes.

NR used as a matrix was obtained from pre-vulcanized latex supplied by *Bassan Indústria e Comércio de Látex Ltda*, located in São Paulo (Brazil), which kindly provided the chemical composition shown in Table 1. The product is composed of centrifuged natural latex, additives, and stabilizers. This composition allows the latex compound to vulcanize at room temperature. The pre-vulcanized latex was used without changes in the present study.

2.2 Sample preparation

To improve adhesion at the matrix/fiber interface, alkaline treatment was applied to part of the curaua fibers, using 5% aqueous sodium hydroxide (NaOH) solution to the curaua fibers for 2 hours, producing samples with treated and untreated fibers. The fibers were washed with distilled

Table 1. Chemical formulation of natural rubber, in phr (parts per hundred of rubber).

Component	Concentration
Natural rubber (NR)	100 phr
Sulfur	3,0 phr
ZBEC (Zinc dibenzylthiocarbamate)	1,2 phr
ZnO (zinc oxide)	1,8 phr
Antioxidant (SKF)	1,2 phr
KOH (Potassium hydroxide)	1,5 phr
Potassium laurate (C ₁₂ H ₂₅ KO ₂)	1,0 phr

water to remove impurities from the process until the pH was fully neutralized. The treated fibers were left to rest for 48 hours at room temperature and dried for 24 hours in an air-circulating oven^[29-31].

The production of the composite samples involved manual mixing of pre-vulcanized latex with varying amounts of ground curaua fibers, both treated and untreated, using a knife mill (Marconi, MA048, mesh 32). The biocomposites were prepared with fiber contents by weight, ranging from 2.5% to 15%, with increments of 2.5% (Table 2). An air-circulating oven at 70 °C accelerated the curing process^[29-31].

2.3 Experimental techniques

Bulk density tests of the curaua fiber were performed using a graduated cylinder^[32], where the graduated cylinder's volume variation containing distilled water and fiber was measured. The test used a pycnometer following ASTM D297-21 for natural rubber and its composites.

Functional groups on sample surfaces were analyzed using Fourier Transform Infrared Spectroscopy with Attenuated Total Reflectance (FTIR-ATR) with a Shimadzu Spectrometer, model 'IRAffinity-1S and IV. Curaua fiber and composites underwent microscopic analysis to evaluate the surface morphology and adhesion between the matrix and the fiber. A high-resolution scanning electron microscope (SEM) (Jeol, JSM IT500 HR) was employed. Samples' crystallinity indexes were determined using a Shimadzu X-ray diffractometer (Maxima XRD 7000) with a Cu-K α wavelength (1.541874 Å), a voltage of 40.0 kV, and a current of 30.0 mA. The scan range was from 5° to 100° (2 θ), with a size of 0.02°/step. The empirical Segal method was used for lignocellulosic fibers crystallinity index calculation^[33].

Thermal analyses were performed using an SDT Q600 analyzer (TA Instruments) with samples of approximately 10 mg. The heating rate was 10 °C/min up to a final temperature of 800 °C, with a 30 mL/min nitrogen gas flow.

The samples' thermal conductivities were determined using the Modified Transient Plane Source (MTPS) method and the TCi sensor from C-Therm, following ASTM D7984-21.

Shore A hardness, suitable for soft materials such as rubbers and foams, was measured using a digital durometer (Kaptron, FE-0063). The tests were conducted according to ASTM D2240-05.

Tensile strength tests were conducted according to ISO 37. A universal testing machine (INSTRON 5984) with integrated BLUEHILL 3 software was used. It was equipped with a 15.0 kN load cell at an ambient temperature of 24 °C and a 200 mm/min testing speed.

Table 2. Natural rubber composite formulations with varying curaua fiber content. Sample code designates the fiber load (%) by weight and whether it is treated (T) or untreated (UT).

Curaua Fiber	Fiber content	Sample Code
Untreated (UT)	2.5 to 15%w	C2.5-UT, C5-UT, C7.5-UT, C10-UT, C12.5-UT, C15-UT
Treated (T)		C2.5-T, C5-T, C7.5-T, C10-T, C12.5-T, C15-T

3. Results and Discussions

3.1 FTIR-ATR

The spectra of treated and untreated fibers were similar to those observed in the literature, with notable cellulose, hemicellulose, and lignin bands, as seen in Figure 1a^[4,6,8,12]. The decrease in the intensity of the 3341 cm⁻¹ band after alkaline treatment suggests the removal of hydroxyl groups because NaOH trends to break hydrogen bonds of OH groups present on the fiber surface, forming fiber-O-Na groups and softening the initial hydrophilic characteristic^[12]. The reduction of the 2905 cm⁻¹, 1732 cm⁻¹, and 1040 cm⁻¹ bands indicates a decrease in cellulose and hemicellulose. The absence of the 1514 cm⁻¹ and 1240 cm⁻¹ bands in the treated spectrum suggests the dissolution of lignin. The 1370 cm⁻¹ band increase indicates that the cellulose structure is more exposed^[4,18]. The changes in the fiber spectrum shown in Figure 1a demonstrate that alkalization effectively modifies the fiber surface.

The spectra of composites with different fiber loads (2.5%, 10%, and 15%) were compared with those of NR (Figure 1b). The results show characteristic peaks of poly(cis-1,4-isoprene) and bands for sulfonates (SO₂), indicating vulcanization. Increases in the bands at 3341 cm⁻¹, 1040 cm⁻¹, and 670 cm⁻¹ are associated with hydroxyl groups and components from the curaua fiber^[34]. The spectra of the composites indicated the predominance of NR, with no significant shifts in peaks, suggesting the absence of chemical reactions between the matrix and the dispersed phase, indicating only physical interaction^[35].

3.2 XRD

Figure 2 presents the X-ray diffraction (XRD) patterns of curaua fibers, NR and composites with 2.5%, 10%, and 15% w/w fiber loadings. Untreated fibers exhibited a crystallinity index of 59.9%, while those treated with NaOH increased to 71.3%, indicating a 19.2% increase due to the removal of amorphous phases (lignin, hemicellulose, and semi-crystalline cellulose). This increase in the crystalline phase corroborates the hypothesis that alkali treatment may have altered the surface of the treated fibers, in agreement with the FTIR.. The NR XRD pattern shows a scattering around 2θ=18°, typical of amorphous materials. All composite samples maintained the same amorphous pattern as NR, indicating that the introduction of curaua fibers did not significantly change the matrix structure, independently of the fiber concentration or alkali treatment.

3.3 TGA/DTG

Figures 3a and 3b show the TGA and DTG curves for NR and composite samples. These curves allow one to analyze the effects of fiber loading and alkaline treatment on the thermal stability of composites relative to the matrix.

Three events can be noted. The first is a subtle mass loss from 60 °C to 150 °C due to volatiles on the material's surface and moisture in the natural fiber loads, which are naturally hydrophilic^[30]. The second one occurs between 300 °C and 450 °C, where a pronounced mass loss is observed, likely due to the degradation of holocellulose (hemicellulose and cellulose) and hydrocarbons. The third one is a more gradual mass loss observed between 480 °C and 550 °C, resulting from the matrix's lignin decomposition and extractives^[31]. Overall, the thermogravimetric behavior observed in the TG and DTG curves was similar between the composites and NR. Nevertheless, previous research has indicated slight reductions in the thermal stability of NR composites filled with natural fibers, mainly due to the early degradation of the dispersed phase in these materials^[36,37].

However, small increases in the onset degradation temperature were observed in samples C2.5-UT and C2.5-T, reaching 354 °C and 347 °C, respectively, less than 2% higher than NR. The other composite samples (5% to 15%w) showed a moderate reduction in thermal stability, with sample C5-UT being the smallest, with a Tonset equal to 326 °C (<6.3%). Considering the initial degradation temperature, it can be inferred that the composites are thermally stable up to 300 °C, similar to what was found by Masłowski et al.^[18] for NR composites filled with nettle fiber (*Urtica dioica* L.). Karim et al.^[38] also reported a thermal stability reduction in NR eco-composites, where kenaf fiber (*Hibiscus cannabinus* L.) was reinforced. According to the authors, the decrease in thermal stability is due to the low degradation temperature of lignocellulosic materials, which typically begin to degrade from 200 °C. On the other hand, the final degradation temperature (endset) showed more significant variations compared to NR, with an increase from 566 °C to 646 °C (14.3%) for the C7.5-UT sample. The minor variation in the thermogravimetric test parameters indicates that the increase in curaua fiber loading has few influence on the thermal behavior of the matrix. No significant discrepancies were observed between the treated (T) and untreated (UT) fibers samples. Moonart and Urata^[39] also reported no significant difference in thermal stability regarding the chemical treatment of hemp fiber as reinforcement in NR.

3.4 Bulk density

The apparent density found for curaua fiber without alkaline treatment was 1.250 ± 0.001 g/cm³, close to values reported in previous studies^[4,7,32]. After alkaline treatment, the density increased to 1.43 ± 0.04 g/cm³, representing a 14.4% increase. This change can be attributed to removing amorphous components such as hemicellulose and lignin, resulting in a more efficient packing of the fibers^[28,40]. Tukey's test confirmed the significance of this difference (P ≤ 5%), as listed in Table 3, where means with different letters are statistically distinct. This indicates that this

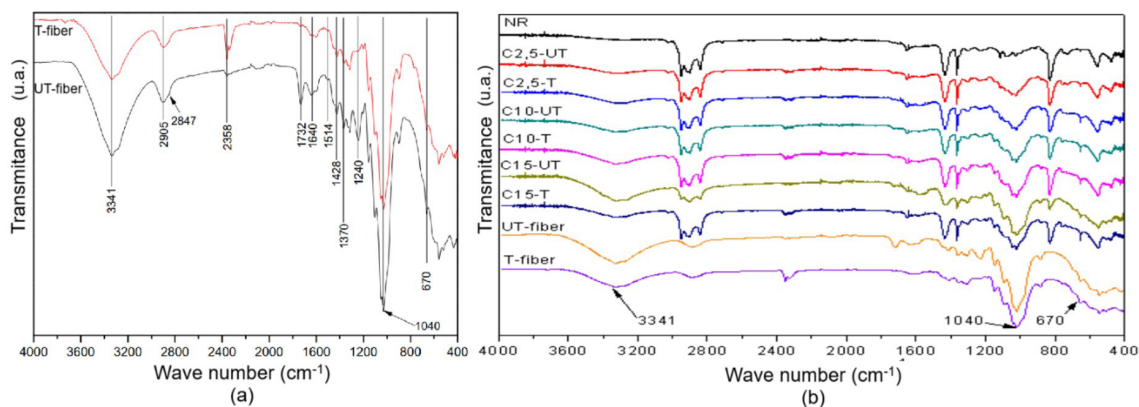


Figure 1. FTIR for untreated (UT) and treated (T) curaua fiber (a) and for NR and its composites (b).

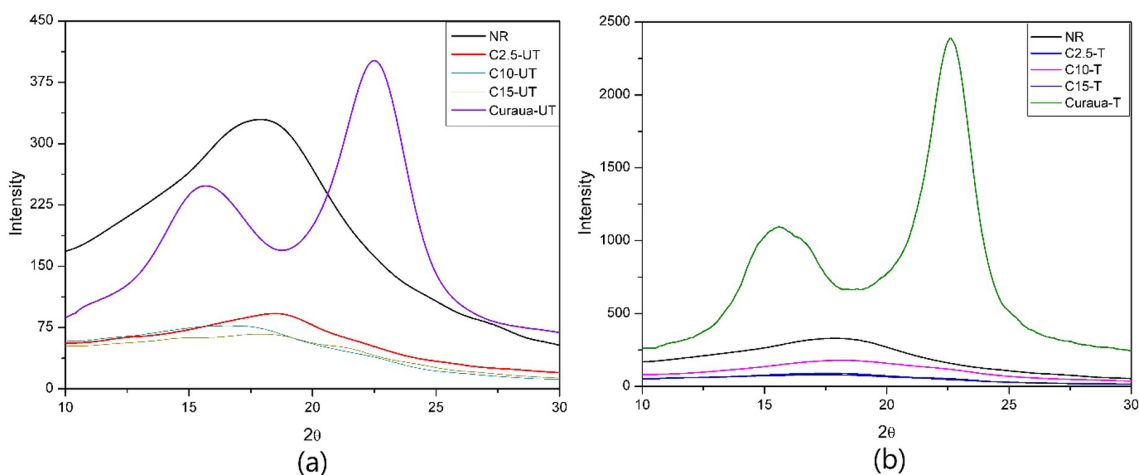


Figure 2. XDR of NR, composites, untreated fiber (a), and treated fibers (b).

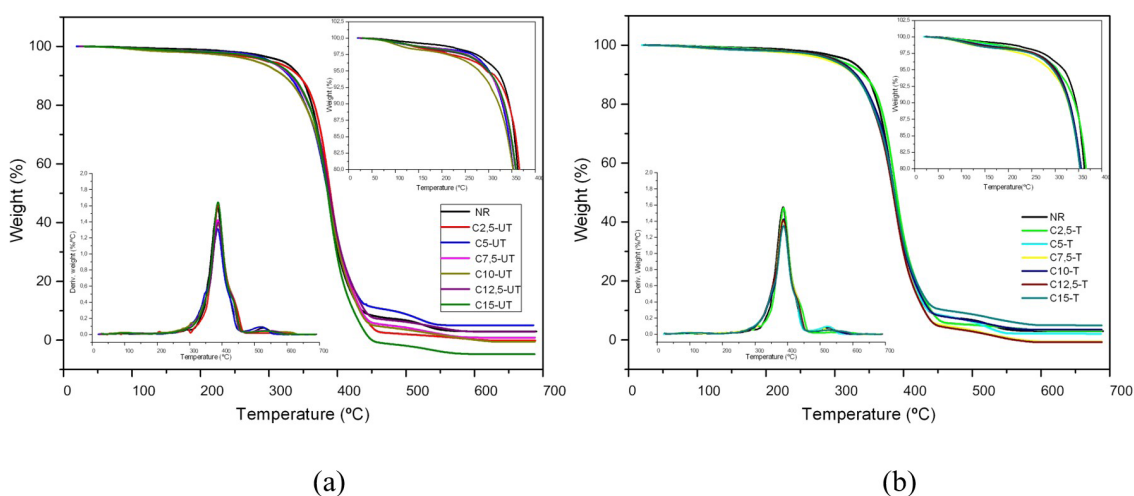


Figure 3. TGA/DTG curves of composites with (a) untreated and (b) treated fibers.

property was influenced by mercerization. Curaua fiber is still lighter than other synthetic fibers, such as E-glass (2.5 g/cm^3)^[22]. The composites showed average densities

lower than NR (1.03 g/cm^3), and the C5-T sample is the lowest one (0.78 g/cm^3), representing a 24.3% reduction compared to the matrix. Tukey's test revealed that only the

Table 3. Properties (mean \pm standard deviation) of the samples produced.

Sh	Bulk density (g/cm ³)		Thermal conductivity (W/mK)		Hardness (Shore A)	
	Untreated	Treated	Untreated	Treated	Untreated	Treated
NR	1.03 \pm 0.09 ^a	-	0.380 \pm 0.004 ^f	-	26.7 \pm 1.6 ^f	-
C2.5	0.95 \pm 0.04 ^{a,b}	0.92 \pm 0.02 ^{a,c}	0.250 \pm 0.002 ^g	0.295 \pm 0.037 ^g	43.7 \pm 0.5 ^d	25.4 \pm 1.9 ^f
C5	0.79 \pm 0.02 ^{d,c}	0.78 \pm 0.03 ^c	0.305 \pm 0.002 ^g	0.290 \pm 0.046 ^g	55.4 \pm 0.6 ^{b,c}	34.2 \pm 1.2 ^c
C7.5	0.85 \pm 0.01 ^{b,c}	0.83 \pm 0.03 ^{c,c}	0.473 \pm 0.026 ^c	1.032 \pm 0.079 ^c	58.8 \pm 0.8 ^{a,c}	45.3 \pm 2.4 ^d
C10	0.85 \pm 0.02 ^{b,c}	0.88 \pm 0.01 ^{b,c}	0.767 \pm 0.002 ^d	1.253 \pm 0.002 ^a	55.9 \pm 3.9 ^{b,c}	56.2 \pm 3.4 ^{b,c}
C12.5	0.89 \pm 0.03 ^{b,d}	0.91 \pm 0.01 ^{b,c}	1.049 \pm 0.007 ^c	1.049 \pm 0.083 ^c	54.8 \pm 3.8 ^c	56.1 \pm 1.1 ^{b,c}
C15	0.93 \pm 0.03 ^{a,c}	0.93 \pm 0.05 ^{a,c}	1.025 \pm 0.008 ^c	1.124 \pm 0.048 ^b	62.5 \pm 0.7 ^a	60.1 \pm 2.6 ^{a,b}
Fiber	1.250 \pm 10 ^{-3f}	1.43 \pm 0.04 ^g	0.0559 \pm 10 ^{-4h}	0.0596 \pm 2x10 ⁻⁴ⁱ	-	-

Sh	Tensile strength (MPa)		Young's modulus at 100% (MPa)		Elongation at break (%)	
	Untreated	Treated	Untreated	Treated	Untreated	Treated
NR	2.83 \pm 0.53 ^e	-	0.42 \pm 0.06 ^f	-	672 \pm 96 ^{b,c}	-
C2.5	5.32 \pm 0.59 ^{b,c}	7.39 \pm 1.23 ^a	0.99 \pm 0.43 ^{c,f}	1.36 \pm 0.04 ^e	857 \pm 188 ^{a,b}	997 \pm 124 ^a
C5	2.85 \pm 0.37 ^e	4.13 \pm 0.05 ^{c,c}	1.41 \pm 0.19 ^c	1.58 \pm 0.08 ^{d,e}	327 \pm 17 ^{d,e}	544 \pm 23 ^{c,d}
C7.5	3.94 \pm 0.96 ^{c,c}	4.07 \pm 0.12 ^{c,c}	2.50 \pm 0.24 ^c	2.41 \pm 0.15 ^{c,d}	241 \pm 81 ^e	340 \pm 22 ^{d,e}
C10	3.36 \pm 0.59 ^{d,e}	5.56 \pm 0.4 ^{b,c}	2.48 \pm 0.12 ^c	3.72 \pm 0.23 ^b	176 \pm 49 ^e	312 \pm 21 ^e
C12.5	4.63 \pm 0.13 ^{b,d}	5.96 \pm 0.26 ^{a,b}	2.32 \pm 0.27 ^{c,d}	4.95 \pm 0.31 ^a	359 \pm 54 ^{d,e}	188 \pm 57 ^e
C15	3.3 \pm 0.2 ^{d,e}	5.51 \pm 0.79 ^{b,c}	1.67 \pm 0.03 ^{c,c}	4.97 \pm 0.79 ^a	311 \pm 33 ^c	156 \pm 49 ^c

Means for the same property with different letters differ significantly ($P \leq 0.05$) by Tukey's test.

composites C2.5-UT/T, C15-UT/T, and C2.5-T did not differ significantly from the density of NR, indicating a relevant influence of fiber loading on the density of the composites. Additionally, no statistical difference was noted between the densities of composite pairs produced with treated and untreated fibers, showing that alkaline treatment did not affect this composite property.

3.5 Thermal conductivity

The average thermal conductivity determined for curaua fiber, without alkaline treatment, was 0.0559 \pm 0.0001 W/m-K, consistent with the typical value of 0.055 W/m-K found for lignocellulosic fibers^[41]. According to Asdrubali et al.^[42], curaua fiber would be classified among materials with intermediate insulating performance (0.05 $\leq k \leq$ 0.08 W/m-K). However, this value is close to those observed in commercial insulating materials such as glass and rock wool (0.04 and 0.045 W/m-K, respectively)^[42,43]. Influenced by alkaline treatment, the thermal conductivity increased to 0.0596 \pm 0.0002 W/m-K (6.6%), as shown in Table 3.

The thermal conductivity of composites with untreated fibers ranged from 0.250 \pm 0.002 W/m-K to 1.049 \pm 0.007 W/m-K, while for composites with treated fibers, it ranged from 0.290 \pm 0.046 W/m-K to 1.253 \pm 0.002 W/m-K (Table 3). The C2.5-UT composite showed 0.25 W/mK of thermal conductivity, the lowest among the samples. This result is 24% lower than that obtained for the NR/PALF (pineapple leaf fiber) composite, which was considered a promising thermal insulator^[44].

From 5% fiber content, thermal conductivity tends to increase, reaching maximum values in composites C12.5-UT and C10-T, respectively, 1.049 W/m-K and 1.253 W/m-K. The crystalline phase of curaua fiber may play a significant role in the observed thermal conductivity behavior, as such structures can facilitate thermal conduction and diffusion in the composites^[45]. This is evidenced by the fact that most

composites with treated curaua fiber exhibit higher thermal conductivity than those with untreated fiber. As predicted in the literature, a consequence of alkali treatment is the improvement in fiber-matrix adhesion, which implies an increased contact area, which in turn can maximize thermal conductivity^[46]. Tukey's tests indicate that thermal conductivity is strongly influenced by fiber content. However, between pairs of composites with treated and untreated fibers, it's noted that mercerization is more effective in samples with high fiber content (10-15%).

3.6 Mechanical properties

The hardness tests on composites with untreated fibers showed values ranging from 43.7 ShA to 62.5 ShA, while those with treated fibers varied from 25.4 ShA to 60.1 ShA, presenting an increasing trend depending on the filling load. The highest hardness values were observed in samples C15-UT and C15-T, respectively, 62.5 ShA and 60.1 ShA, which is an interesting characteristic for the footwear industry, such as the NR/Sugarcane bagasse fiber composite, proposed by Paiva et al.^[47], (>55 ShA). The data in Table 3 reveal that hardness of untreated fiber composites significantly increases starting at 2.5% fiber content but stabilizes from 5% onwards. In contrast, for composites with treated fibers, hardness is notably affected from 5%, with stabilization occurring from 10% fiber content.

A tensile strength test was conducted on the composites, varying the content of treated and untreated fibers to evaluate their maximum stresses and strains. NR exhibited an average tensile strength of 2.83 MPa. Composites with untreated fibers ranged from 2.85 MPa to 5.32 MPa, while those with treated fibers ranged from 4.07 MPa to 7.39 MPa. The highest values were observed in composites with 2.5% fiber (UT/T), increasing to 161% compared to NR. Beyond 5% fiber loading, there was a decrease in tensile strength followed by an increasing trend up to 12.5% fiber content; at this point, another reduction was observed. This

behavior indicates a fiber loading limit for positive effects on tensile strength, similar to that observed by Ubi et al.^[48]. The alkaline treatment of curaua fibers improved the mechanical properties of composites, increasing tensile strength and the elastic modulus due to better adhesion between the matrix and mercerized fibers^[47]. Despite this, the tensile strength results for NR/Curaua fiber composites were superior to those obtained for NR/Luffa fiber (*Luffa cylindrica*) composites (0.315 to 0.788 MPa)^[49], where the authors indicated them for energy-absorption applications. The modulus of elasticity at 100% elongation showed maximum values of 2.5 MPa (C7.5-UT) and 4.95 MPa (C12.5-T), with additional increases leading to a deterioration in stiffness. Elongation at break decreased with increasing fiber content as the filler reduced the deformable phase of the rubber, stiffening the

composites^[50]. However, composites with 12.5% and 15% untreated fiber showed greater elongations, in contrast to the trend observed in treated fibers. The Tukey test groupings (Table 3) indicate that mechanical properties are significantly affected by fiber content and alkaline treatment, especially in composites with high fiber content (10 to 15%), where sodium hydroxide treatment had a greater influence.

3.7 SEM

Scanning electron microscopy (SEM) was performed on the composites C2.5-UT/T, C10-UT/T, and C15-UT/T to assess the interaction between matrix and fiber load, as shown in tensile fracture images from Figure 4a to 4f, respectively. The images in Figures 4a and 4b show randomly oriented

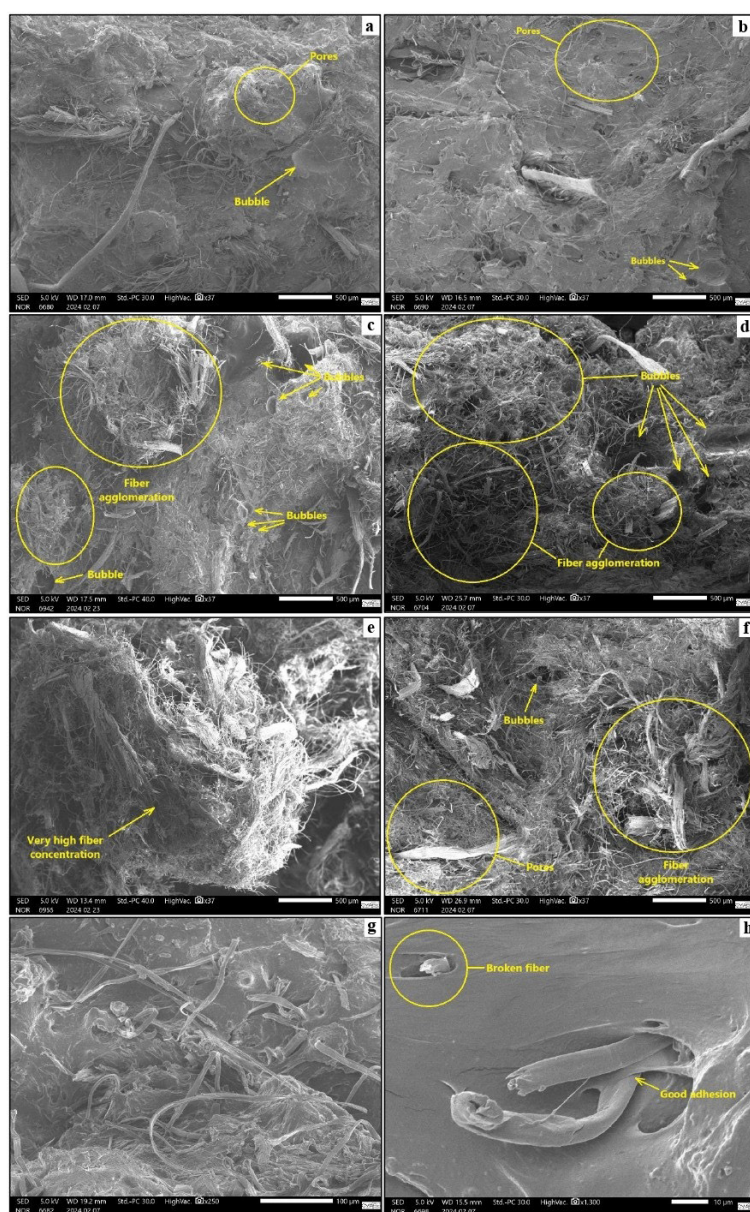


Figure 4. SEM of the composites (a) C2.5-UT (b) C2.5-T (c) C10-UT (d) C10-T (e) C15-UT and (f) C15-T, magnified 37x, (g) C2.5-UT, magnified 250x, and (h) C2.5-T magnified 1300x.

fibers within the elastomeric matrix and pores caused by bubbles, indicating failures due to manual material processing without compaction or pressurization. These bubbles and fiber agglomerations in the matrix, observed in Figures 4c to 4f, act as stress concentrators, compromising the mechanical properties of the composites.

Samples with 2.5% fiber loads exhibited better dispersion and less agglomerations than those with 10% and 15% fiber, which explains their better performance in the tensile test (Table 1). The C2.5-UT sample showed a rough surface and fractured layers (Figure 4a), indicating weak adhesion between the matrix and fiber. In contrast, the C2.5-T sample showed good matrix-fiber bonding and broken fibers (Figure 4h), evidencing strong adhesion and justifying its greater tensile strength.

There was few evidence of fiber pull-out in the samples with 2.5% filler content. The C2.5-UT composite presented a rough surface and fractured layers (Figure 4g), indicating weak adhesion between the matrix and fiber load^[37]. On the other hand, Figure 4h shows that the C2.5-T sample appears to have good adhesion at the matrix/filler interface (yellow arrow), presenting broken fibers (yellow circle) and evidencing strong adhesion^[39]. Such considerations may justify the high tensile strength in composites with 2.5% treated fiber load compared to untreated ones.

The fiber dispersion and matrix/fiber adhesion observed in SEM can also significantly affect thermal conductivity. Composites with poorly distributed fibers and agglomerations, as seen in samples with higher fiber content (10% and 15%w), show increased thermal conductivity since fiber agglomerations may be acting as a preferential path for thermal conduction, which is even more evident in samples with treated fibers^[44]. On the other hand, samples with 2.5%w fiber presented low thermal conductivities, probably due to the good fiber dispersion and pores, which can hinder heat transmission in the material, making them more suitable for thermal insulation applications.

4. Conclusion

Natural rubber composites reinforced with curaua fibers were produced and thermally and mechanically characterized. FTIR and XRD analyses indicated that the interaction between the NR matrix and the fibers is predominantly physical, with no significant chemical changes in the composites. From a thermal point of view, thermogravimetric analyses revealed that the composites maintain thermal stability up to approximately 300°C, with minimal variations in the initial degradation temperature, and the lowest thermal conductivity was observed in samples with 2.5% treated fibers, representing a reduction of up to 34.2% in relation to the matrix.

In addition, composites with 2.5% treated fibers achieved a 161% increase in tensile strength, compared to NR, although higher fiber contents resulted in a decrease in this property due to inadequate dispersion of the reinforcing phase. In general, composites produced with treated fibers presented better interfacial adhesion and superior mechanical properties compared to those with untreated fibers. Thus, the results suggest that composites with 2.5% treated fibers offer the

best combination of thermal and mechanical properties, being the most promising for commercial and engineering applications.

5. Author's Contribution

- **Conceptualization** – João D'Anuzio Lima de Azevedo.
- **Data curation** – João D'Anuzio Lima de Azevedo.
- **Formal analysis** – João D'Anuzio Lima de Azevedo.
- **Funding acquisition** – NA.
- **Investigation** – João D'Anuzio Lima de Azevedo.
- **Methodology** – João D'Anuzio Lima de Azevedo; Virgínia Mansanares Giacon; José Roberto Ribeiro Bortoleto.
- **Project administration** – João D'Anuzio Lima de Azevedo.
- **Resources** – João D'Anuzio Lima de Azevedo; Virgínia Mansanares Giacon.
- **Software** – NA.
- **Supervision** – Virgínia Mansanares Giacon; José Roberto Ribeiro Bortoleto.
- **Validation** – Virgínia Mansanares Giacon; José Roberto Ribeiro Bortoleto.
- **Visualization** – João D'Anuzio Lima de Azevedo.
- **Writing – original draft** – João D'Anuzio Lima de Azevedo.
- **Writing – review & editing** – Virgínia Mansanares Giacon; José Roberto Ribeiro Bortoleto.

6. Acknowledgements

The authors would like to thank the Laboratory of Amazonian Materials and Composites (LaMAC/UFAM), the Multiuser Center for Analysis of Biomedical Phenomena (CMABio/UEA), and the Analytical Center of the Federal Institute of Education, Science and Technology of Amazonas, Campus Manaus Centro (CA-IFAM-CMC) for technical support. Among the authors, Azevedo specifically thanks the Federal University of Amazonas (UFAM) and the São Paulo State University (UNESP) for the opportunity to join and study the Interinstitutional Doctorate Program (DINTER) offered by the Postgraduate Program in Materials Science and Technology (POSMAT).

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Received: Dec. 03, 2024

Revised: Feb. 11, 2025

Accepted: Feb. 13, 2025

Associate Editor: José A. C. G. Covas