

# Effects of replacing Carbon Black with Wood Fibers in wood-rubber composites

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#### Abstract

The objective of this research was to develop a more sustainable composite and still keep its characteristics. Fibers treated with NaOH at 5% w/w for 2 hours were incorporated into natural rubber in different proportions (0 phr, 24 phr, 36 phr, 48 phr and 60 phr). The mechanical properties of the composites suffered changes, increasing the hardness, young's modulus and decreasing its tensile strength. All the physical properties were not statistically different. The coloration became less dark in the WF60/CB0 treatment, and the electrical properties presented better resistivity with the increase in the concentration of fibers in the composite. This presents a possibility of using WF for the production of wood-rubber composites for the production of rubber artifacts which do not require high rolling resistance. Based on the results from this research, we recommended the WF24/CB36 mix to produce antistatic floors.

Keywords: carbon black, composite, composite properties, natural rubber, wood fibers.

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# 1. Introduction

Composites consist of a material divided into two phases: Matrix and Reinforcement; generally, the matrix is involved by the reinforcement. The properties of composite materials are a function of the properties of the matrix and the dispersion of the dispersed phase. Composite materials can be classified according to their matrix into three types: Ceramic Matrix, Polymeric Matrix (which included the elastomers) and Metal Matrix<sup>[1-3]</sup>.

In order to improve the physical-mechanical properties and reduce material costs in the manufacture of rubber composites, many particles are used by the rubber industry and this addition generally leads to a gain in mechanical properties, abrasion and tear resistance<sup>[4]</sup>.

Carbon black (CB) is the common reinforcing filler used in the rubber industry. The properties of carbon black reinforced rubber composites mainly depend on its 'structure'. CB has the capability to alter the rubber vulcanization chemistry and present both chemical and catalytic activity. Even though CB provides good physical, mechanical, thermal and barrier properties to the rubber composites, it has some disadvantages. CB is manufactured by the incomplete combustion of heavy petroleum products<sup>[5,6]</sup>.

Nowadays, there is a great environmental pressure relieving products that are more aggressive to the environment from industrial production. This is where lignocellulosic fibers come in as an alternative for the production of sustainable composites.

Cellulosic fibers are basically composed of cellulose, hemicellulose and lignin, which correspond to approximately 90% of the dry weight of these fibers, whereas the remaining 10% correspond to components such as pectins, waxes, extracts and proteins<sup>[7]</sup>.

The structure of the wood fibers (WF) confers to this wood byproduct excellent mechanical properties, such as high strength in relation to its weight. This material can be obtained from wood by different techniques, such as chemical, mechanical, biological and many low-cost combined  $processes^{[8]}.$ 

There are several papers reporting good responses to the addition of lignocellulosic fibers in the formulation of the rubber composite<sup>[6]</sup>: nanocellulose<sup>[9]</sup>, different fillers<sup>[10]</sup>, sugarcane bagasse<sup>[11]</sup>, jute fibers<sup>[12]</sup>, cereal straw. However, there are few reports about the replacement of CB by these fibers and the physical-mechanical behavior of the composite.

The objective of this research was to produce rubberwood composites and test them for physical-mechanical behavior, looking at sustainability, in order to develop anti-static floors.

# 2. Materials and Methods

### 2.1 Wood-rubber composite manufacture

The materials used is this research were: *Pinus* fiber, without classification about species, used in the manufacture of MDF panels provided by the company ARAUCO and Carbon Black N550 provided by the company Elastrobras. The Carbon Black N550 was used due to its classification as semi-reinforcing with high structure, provides easy and good dispersion, and can be used for calendered rubbers.

The formulation used in the preparation of NR-CB/WF vulcanizates are in Table 1. The wood fiber was dried in an oven for 24 hours at 65°C then it was used directly as filler in NR-CB/WF composites. The mixing was performed in two-roll mills with varying amounts of WF (0, 24, 36, 48 and 60 phr) and CB (0, 60, 36, 24, 12 phr). The treatment control does not have any WF and CB. The ingredients were added in the following order: zinc oxide, stearic acid, WF (treated with sodium hydroxide 15% for 2 hours), CB (when present) and 24 hours later we added MBTS, TMTD and sulfur. The sheets were vulcanized at 150°C under a pressure of 165 Kgf/cm<sup>2</sup> in a hydraulic press. 5 sheets with 15x15 cm were made.

# 2.2 Wood-rubber composite characterization

The rheological properties were obtained using an oscillating disc rheometer in order to determine the t90 and the influence on torque of the replacement of CB by WF.

The mechanical physical properties were obtained according to the ASTM standard for each analysis. The cure characterization was determined by a rheometer with an oscillating disk according to the ASTM D 5289. The water absorption was obtained according to ASTM D 570. Stress-strain test was performed on an EMIC Model DL 2000 Universal Testing Machine as per ASTM D 412. The composite hardness was measured according to ASTM D 2240 using the Shore A type durometer. The composite density was obtained by the Archimedes Method and the crosslink density as per the Flory-Renner equation<sup>[12]</sup>. Abrasion tests were made using a MAQTEST rotating cylinder according to DIN 53516.

The morphological properties tested were Colorimetry, which was made using Konica Minolta CM-5 spectrophotometer in a computer with the SpectraMagic NX program. The wettability was obtained using the tensiometer OCA 25 with DDE/3 by the sessile drop method. The superficial roughness was determined using the profilometer Talysurf CCI model 2. Thermogravimetric analysis was performed on Thermogravimetric Analyzer STA 449F3 Jupiter with heating rate of 10°C/min under nitrogen atmosphere and the DSC was performed on DSC 200F3 MAIA following the temperature range (-100 to 300°C), using nitrogen as carrier gas with 30ml/min flow.

The resistivity analysis was carried out in an Agilent 4339B resistor with model 16008B resistivity cell, using a 26 mm diameter electrode with a pressure set at 5 Kgf/cm<sup>2</sup>. For each sample, 5 surface resistivity and 5 volumetric measurements were obtained. For all the treatments, a voltage was used that allowed the evaluation without the equipment being subject to error.

SEM was used to evaluate the morphological characteristics of the composites, as well as the identification of weak bonds, foreign bodies and possible non-incorporation of reinforcements. Before being submitted to image capture, the composites were metallized with gold particles by the plasma spray system for 90 seconds with a current of 30 mA.

# 3. Results and Discussions

# 3.1 Rheology of wood-rubber composite

The cure characteristic of all the composite formulations is given in Table 2.

The lowest torque value was found in the control treatment which also presents better fluidity. This was expected due to the absence of additives (CB and WF) in its formulation, which demands a lower torque for disc movement. The maximum torque increased following the increase in charges, as a result of the increase in existing cross-links, that is, growth in the number of linked chains and a strong link between the charges and the matrix<sup>[13]</sup>.

Table 1. Formulation in phr for the wood-rubber composite.

Components	Control	WF0/CB60	WF24/CB36	WF36/CB24	WF48/CB12	WF60/CB0	Î
Rubber	100	100	100	100	100	100	ĺ
Wood Fiber*	0	0	24	36	48	60	
Zinc Oxide	5	5	5	5	5	5	
Stearic Acid	2	2	2	2	2	2	
MBTS	1.2	1.2	1.2	1.2	1.2	1.2	
TMTD	0.4	0.4	0.4	0.4	0.4	0.4	
Sulfur	2.5	2.5	2.5	2.5	2.5	2.5	
Carbon Black	0	60	36	24	12	0	

MBTS - MBTS - benzothiazole disulfide; TMTD - Tetramethyl thiuram disulfide. \*Pinus spp. Fiber.

Treatment	t90 (min)	M <sub>190</sub> (lb.in)	M <sub>H</sub> (lb.in)	M <sub>L</sub> (lb.in)
CONTROL	5' 19"	15.40	17.00	2.00
WF0/CB60	2' 58"	37.00	40.50	3.50
WF24/CB36	2' 55"	39.70	43.90	2.40
WF36/CB24	3' 30"	40.40	44.60	2.60
WF48/CB12	3' 58"	42.70	47.10	2.80
WF60/CB0	4' 36"	40.00	44.10	2.50

Table 2. Rheometrics parameters of NR compared to composites of NR with CB and WF.

Where:  $t_{90}$  – Cure time; M  $t_{90}$  – Torque on  $t_{90}$ ; M<sub>H</sub> – Maximum torque; M<sub>L</sub> – Minimum torque.

Analyzing the t90, it is noted that the vulcanization time is much longer in the control treatment, where there is no CB or WF. When it comes to replacing the CB with WF, there was an increase in time. This behavior suggests that carbon black acts in the vulcanization system, accelerating the formation of sulfide bonds, unlike wood fibers<sup>[14]</sup>.

The increase in t90 for treatments with the use of WF occurs due to the fact that the fibrous material absorbs the vulcanizing agents and curing accelerators due to its high contact area and hydrophilic character<sup>[6,15]</sup>. One of the possibilities of having a difficulty in the process of vulcanization of rubber is the hydroxyl groups present on the surface of the fibers<sup>[15]</sup>.

# 3.2 Physical and mechanical properties of wood-rubber composite

#### 3.2.1 Water absorption

The water absorption results are in Table 3.

A low WA through the Control and one treatment confirms the hydrophobic behavior of both natural rubber and CB<sup>[16-18]</sup>. The control showed a higher water absorption compared to the WF0/CB60 due to the absence of carbon black and fibers in its composition. In this case, carbon black reduces the empty spaces left by the rubber at the time of its vulcanization, that is, the presence of carbon black in the WF0/CB60 treatment contributed to the reduction of water absorption by the sample<sup>[19]</sup>.

In treatments with substitution of CB for WF, greater water absorption was noted; this behavior presented was similar to that found by<sup>[20]</sup>, who, evaluating the water absorption with different fillers, concluded that lower presence of CB influences the water absorption by composites.

Generally, the WA of a given material requires the presence of pores or some permeable channel in its microstructure in the form of open cells to create a way for water<sup>[21]</sup>. The low WA by the treatment with a greater presence of CB, is the result of the small number of pores that the microstructure of the composite presents and the high-water absorption by the treatments as there is a replacement occurs by the lignocellulosic material presenting active hydroxyl groups leading to the formation of hydrogen bridges with water particles<sup>[22,23]</sup>.

#### 3.2.2 Density

The basic density results are in Table 4.

According to the results presented in Table 4, there were no statistically significant differences regarding the density of the composite. The presence of fillers in rubber composites

Table 3.	Water	absor	otion	of the	wood-rubber	composite.

1	1	
Treatment	Water Absorption (%)	
CONTROL	2.04 b	
	(27.75%)	
WF0/CB60	0.48 a	
	(1.07%)	
WF24/CB36	4.00 e	
	(10.80%)	
WF36/CB24	4.96 f	
	(7.44%)	
WF48/CB12	6.66 g	
	(15.43%)	
WF60/CB0	7.41 h	
	(5.55%)	
		1

Mean values followed by the same letter in the column showed no statistical difference by Tukey's test (p=>0.05). Values in parentheses correspond to the Coefficient of Variation.

Table 4. Basic density of wood-rubber composite.

Treatment	Basic Density (g/cm <sup>3</sup> )
CONTROL	1.112 a
	(6.46%)
WF0/CB60	0.991 a
	(2.14%)
WF24/CB36	1.103 a
	(15.27%)
WF36/CB24	1.080 a
	(12.87%)
WF48/CB12	0.999 a
	(1.24%)
WF60/CB0	0.998 a
	(0.93%)

Mean values followed by the same letter in the column showed no statistical difference by Tukey's test (p=>0.05). Values in parentheses correspond to the Coefficient of Variation.

does not influence the basic density of the composite, regardless of the nature of this filler<sup>[24,25]</sup>. The basic density of a composite is directly associated with the hardness of the material, that is, as there is a greater number of additives, there is also an increase in the hardness of the material and its basic density<sup>[26]</sup>.

In this work, there was no positive correlation between hardness and basic density, which is due to the characteristics intrinsic to WF. The WF are light, making the composite less dense. Following this observation, the potential of using WF as a substitute for CB facilitates handling, transportation and still maintains important characteristics from the technological point of view for the flooring industry.

#### 3.2.3 Crosslink density

The molecular transport of solvent molecules across the natural rubber matrix is a kinetic parameter that mainly depends on the free volume present in the material, the mobility of polymer chains and the size of the penetrant molecule<sup>[6]</sup>. The crosslink density results are in Table 5.

There is a significant difference between the values of treatments with a high CB content compared to those presenting WF. This drop is justified by the hydrophilic character of the WF, which makes it difficult to form a chemical bond (crosslinking) with the accelerators and activators, as well as the density of cross bonds<sup>[27]</sup>.

In vulcanized rubbers, swelling is directly related to the density of crosslinks that are effectively formed during the vulcanization process. These crosslinks are strong bonds between the chains and the crosslinking agent, which do not allow the complete dispersion of its molecules in toluene, thus restricting the deformation of the rubber<sup>[27]</sup>.

As there is an increase in the density of crosslinks, the diffusion of this composite in the solvent will be lower, reducing the swelling caused<sup>[11]</sup>. With the addition of reinforcement in the composite formulation, there is usually a decrease in swelling, this behavior is more pronounced when using reinforcing fillers that cause a chemical bond with the crosslinking agent, than inert fillers which originate a physical bond.

The high crosslink densities can directly contribute to the mechanical characteristics of the composite<sup>[28]</sup>. There is a tendency for a positive correlation between bond density, Shore A hardness and tensile strength, which is related to the amount of bonds formed between the matrix and the fillers.

There is also a relationship between cross-links and physical properties, such as water absorption and curing parameters. There is a greater difficulty in causing deformation to the composite (increased torque), when there is a greater crosslink density and the opposite occurs for water absorption, where there is a reduction on this characteristic<sup>[13,29]</sup>.

#### 3.2.4 Hardness

The hardness of a material is related to the amount of filler connections to the activators and accelerators, in this study, carbon black and wood fibers<sup>[9]</sup>. A greater number of fillers restricts the movement of the chain, making the composite more rigid, from there the composite becomes more resistant to penetrating forces.

The hardness of the material is directly linked to the amount of reinforcing material that is placed in the

Table 5. Crosslink density of wood-rubber composite.

Treatment	Crosslink Density (mol/cm <sup>3</sup> )
CONTROL	6.66E-04
WF0/CB60	1.28E-03
WF24/CB36	6.04E-04
WF36/CB24	4.69E-04
WF48/CB12	6.21E-04
WF60/CB0	5.50E-04

formulation, that is, the higher the phr of reinforcement used, the greater the hardness of the material, safeguarding the nature of the material<sup>[30]</sup>.

According to Figure 1, there was an increase in the hardness of the material when there was an incorporation of WF in the formulation, since the amount of filler used was greater, that is, in the WF0/CB60 treatment the result is lower due to the amount of filler in the formulation being smaller than in the other treatments, in volume of material. When we observe WF60/CB0, we notice a slight decrease in the hardness of the material, compared to the other treatments, this is due to the WF being softer and less hard<sup>[31]</sup>.

Based on rubber classification as to hardness, the control is classified as soft rubber, due the lack of fillers and the others as hard rubber. The rubber flooring companies related that the Shore A hardness of the materials vary from 60 to 85, depending on the application. However<sup>[30]</sup>, reports that products with Shore A hardness ranging from 70 to 90 can be used for engineering rubber parts.

The WF80/CB0 stands out, presenting hardness within the standards for rubber sold as flooring material. The research demonstrated the efficiency of replacing CB with WF, making the material more sustainable, replacing a non-organic material by an organic one, and yet maintaining the characteristics required by the industry.

#### 3.2.5 Tensile properties

The tensile properties results are in Table 6.

Due to increase in WF, the composite becomes more rigid and a tendency to decrease the maximum strength and deformation that the material can withstand until rupture is noticed. Due to this increase in hardness, the material breaks with less deformation, a characteristic present in composites of hybrid loads<sup>[32]</sup>. This result corroborates the Shore A hardness result (Figure 1), which tends to increase hardness with increasing loads.

The behavior of increasing tensile strength with the incorporation of CB in the composition (control for WF0/CB60) and the subsequent reduction in other treatments was



Figure 1. Hardness of wood-rubber composite.

<b>Fable 6.</b> Tensile properties of wood-rubber	composite.
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Treatment	Tensile Strength (MPa)	Elongation to break (%)	Young Modulus (MPa)
CONTROL	14.15 b	$1772.09 \pm$	2.04 d
	(10.67%)	97.34	(14.96%)
WF0/CB60	20.29 a	$575.54\pm25.83$	3.93 cd
	(7.66%)		(4.48%)
WF24/CB36	8.33 de	$445.04 \pm 11.43$	7.63 bc
	(5.14%)		(25.96%)
WF36/CB24	7.13 e	$496.75 \pm 30.17$	8.63 abc
	(5.98%)		(28.25%)
WF48/CB12	4.19 f	$407.45 \pm 81.25$	13.62 a
	(10.21%)		(34.55%)
WF60/CB0	3.01 f	$406.53 \pm 83.91$	10.03 ab
	(10.92%)		(11.33%)

Mean values followed by the same letter in the column showed no statistical difference by Tukey's test (p=>0.05). Values in parentheses correspond to the Coefficient of Variation.

observed by<sup>[32,33]</sup> in their pure PALF, carbon black + PALF and carbon black + silica composites, respectively.

According to the authors, there are two reasons for the occurrence of this behavior: one is that the leveling stress of rubber with CB is lower than rubber without CB or with addition of WF. Also, the hybrid composites with CB present a good recovery rate when exposed to high stress levels.

For the Young modulus, a behavior that is inversely proportional to the tensile strength is noted, so it can be said that to exert a certain deformation, a greater effort is required to deform the rubber-WF composite as the charges increase.

In the treatments WF36/CB24, WF48/CB12 and WF60/CB0 there were no statistical differences among them, which coincides with the greatest replacements, proving that the less elastic the material, the greater its hardness and the greater the effort for the same degree of deformation of the composite<sup>[1]</sup>.

This statistical similarity among treatments further highlights the advantage of using WF60/CB0 for flooring production, as Young's modulus was statistically similar to the other treatments mentioned above even with Shore A hardness and lower maximum tensile strength.

#### 3.2.6 Abrasion

The mass loss results are in Figure 2.

Figure 2 shows the increase in mass loss through abrasion in treatments where there was addition of wood fibers, with the best result in treatment Control and WF0/CB60. The increase in mass loss with the addition of wood fiber was associated with the possible creation of aggregates, due to the difficulty of dispersing wood fibers in the rubber matrix, causing the decrease in abrasion resistance. One possible cause of this negative effect on this addition can be the natural fiber size used<sup>[6]</sup>.

Another factor that can be considered to explain this increase is the acid character of the wood fiber. This causes a reduction in the action of accelerating and vulcanizing agents, which have a basic character, causing a reduction in the cross-link, that is, the fibers have a low compatibility



Figure 2. Abrasion loss of wood-rubber composite.

with the matrix causing less interaction in these treatments, thus recording the resistance to abrasion<sup>[1,34]</sup>. In the WF0/CB60 treatment, there was a small decrease in mass loss by abrasion, which can be explained by the ease with which the CB disperses in the rubber matrix.

Even with the results for treatment with the addition of fibers as reinforcement being greater than the Control, it is noted that the WF24/CB36 treatment obtained results lower than 250mm<sup>3</sup>, which is the requirement for the production of rubber floors<sup>[27]</sup>.

#### 3.3 Morphological properties of wood-rubber composite

#### 3.3.1 Differential scanning calorimetry of wood-rubber composite

DSC analysis was performed with the objective of verifying the phase transitions of the composite. The glass transition temperature (Tg) provides information on how the mobility of the chain can be changed with the addition of cellulosic elements or demonstrates the change in the matrix/load interaction<sup>[34]</sup>.

Tg is the temperature at which the material changes phase, leaving the glassy (more rigid) state to a rubberier (elastomeric) state. Below this temperature there is little movement of the molecules (freezing of the chain segments) and above that temperature there is an increase in the amplitude of this vibration<sup>[35]</sup>. With Tg ranging from -60.7 to -61.8°C (Table 7) it is compatible with that found in the literature for poly-cis-isoprene (natural rubber) naturally used or in composites.

The Tg of the treatments that use WF presented a slight decrease when compared to the Control, which may be attributed to the higher molecular weight and a smoother molecular chain of the composites<sup>[36]</sup>. The same authors report that this result can be attributed to the wrapping that rubber makes on different fillers, which can be proven in the density of cross-links and may influence the mechanical results of composites. This small difference between treatments is related to the properties inherent to the equipment, which can make this technique without enough sensitivity to detect Tg changes in this type of material<sup>[37]</sup>.

#### 3.3.2 Colorimetry

Rubber floors usually come in dark colors due to the environment where they are normally used in. The colorimetric evaluation is necessary to detect, even if not visually noticeable, the color difference between the formulations in this study. The results of the colorimetric parameters are in Table 8.

The luminosity  $(L^*)$  is the tendency of a material to present a shade closer to white (+) or closer to black (-). However, some factors may interfere in this parameter, where the materials roughness stands out<sup>[38]</sup>. The correlation between surface roughness and material color change is responsible for explaining the increase in the value of L\* in treatment WF0/CB60, even though it has a lighter shade than the Control treatment, but for presenting a greater roughness according to Table 9, it also presented a higher superficial brightness due to the incidence of light in the grooves which reflect more intensely.

Evaluating the parameter, a\* (red-green axis coordinate), the addition of the CB modified the color of the composite, making its color greener than the Control treatment. Unlike the total addition of WF (WF60CB0), which also changed the color, but giving a more reddish hue to the composite.

Table 7. Glass transition temperature of wood-rubber composite.

Treatment	Glass Transition Temperature (°C)
CONTROL	-60.7
WF0/CB60	-60.8
WF24/CB36	-61.4
WF36/CB24	-61.3
WF48/CB12	-61.8
WF60/CB0	-61.8

 Table 8. Colorimetric parameters and total roughness of wood-rubber composite.

Treatment	L	a*	b*
CONTROL	22.72	0.43	0.97
WF0/CB60	24.65	-0.29	-1.99
WF24/CB36	22.09	-0.24	-1.08
WF36/CB24	23.53	-0.25	-1.27
WF48/CB12	22.92	-0.14	-0.53
WF60/CB0	23.82	2.64	3.46

Legend: L-Luminosity; a\* - Green - red axis; b\* - Blue - yellow axis.

 Table 9. Superficial roughness parameters of wood-rubber composite.

Treatment	Ra	Rq	Rz
CONTROL	0.2165 a	0.2667 a	1.2951 a
	(24.76%)	(26.22%)	(28.08%)
WF0/CB60	0.2883 a	0.3561 a	1.3153 a
	(52.07%)	(53.30%)	(25.79%)
WF24/CB36	0.2600 a	0.3261 a	1.1000 a
	(65.17%)	(77.71%)	(66.57%)
WF36/CB24	0.2864 a	0.2840 a	1.1397 a
	(63.85%)	(40.04%)	(34.96%)
WF48/CB12	0.2717 a	0.3341 a	1.2449 a
	(44.70%)	(50.29%)	(38.31%)
WF60/CB0	0.2685 a	0.2693 a	1.1814 a
	(70.50%)	(24.19%)	(28.39%)

Where: Ra – Medium roughness; Rq – Quadratic roughness; Rz – Total roughness. Mean values followed by the same letter in the column showed no statistical difference by Tukey's test (p=>0.05). Values in parentheses correspond to the Coefficient of Variation. Different results were found to those presented in this research, where the increase in the charge of carbon nanotubes modified the color of the material making it more reddish; however, as the authors used  $CaCO_3$  as a charge, this may have influenced the change in relation to CB used along to wood fibers in this research<sup>[39]</sup>.

For parameter b\* (blue-yellow axis coordinate), the Control and WF60/CB0 presented positive values, that is, they presented a more yellowish color. The other treatments, which used CB, presented negative values, giving a characteristic closer to blue. The addition of carbon nanotubes caused the b\* coordinate to show lower values, which is compatible with that found in this work for CB, due to the dark coloration of CB and carbon nanotubes, these two factors contribute to the darker color of the composite<sup>[39]</sup>.

#### 3.3.3 SEM Images

The Micrographies of the rubber-wood composites from the treatments Control, WF0/CB60, WF24/CB36 and WF60/CB0 are shown in Figure 3.

The presence of sulfur can be noted on the surface of the composite (Figure 3A-3B), which can be characterized by the poor distribution of this reinforcement at the time of addition of this element in the mixing process. The presence of voids can be attributed to the bad formation of the rubber mass at the time of pressing, resulting in air bubbles.

In addition, the adhesion between wood and rubber fibers was weak, so it is possible to identify the presence of voids between the fibers and the matrix. This fact is undesirable, but predictable due to the difference between the nature of the fibers (hydrophilic) and rubber (hydrophobic) which causes this non-compatibility between reinforcement and matrix. Due to these facts, it can be said that the fiber treatment did not have the desired effect on the interfacial bond between wood fibers and rubber.

Another important factor worth highlighting is the surface roughness that can be seen in Figure 3A-3D, when there is an increase in the amount of wood fibers in proportion to the carbon black. In this situation, the surface area becomes less smooth, that is, there is the formation of agglomerates of fibers on the surface or even under the rubber.

Analyzing the SEM images, it is possible to clearly explain the high abrasion losses when there is the presence of a large amount of wood fibers in relation to carbon black. That happens as the agglomerates formed are pulled out with the effort of the grinding wheel, thus decreasing the mass in a much more substantial amount than when the agglomerates are not present.

#### 3.4 Electrical properties of wood-rubber composite

According to Table 10, it is noted that there was a decrease in surface and volumetric resistivity with the addition of carbon black and then there was an increase when CB was replaced by wood fiber. The most significant increase was in the treatment where there is no presence of carbon black.

We can classify the treatments Control, WF36/CB24, WF48/CB12 and WF60/CB0 as insulating composites, because they present resistivity in magnitude greater than 10<sup>8</sup>. The other treatments are considered antistatic



Figure 3. Micrographies of rubber-wood composites. A: Control; B: WF0/CB60; C: WF24/CB36 and D: WF60/CB0.

Treatment	Superficial Resistivity (Ω)	Volumetric Resistivity (Ω.cm)
CONTROL	3.1777E+13	7.533E+14
	(2.07)	(0.29)
WF0/CB60	3.5100E+05	6.2287E+05
	(0.52)	(1.09)
WF24/CB36	1.7333E+06	5.4908E+06
	(0.43)	(0.62)
WF36/CB24	4.3873E+09	2.7454E+11
	(0.73)	(0.55)
WF48/CB12	2.4850E+12	1.7681E+12
	(0.24)	(0.21)
WF60/CB0	4.1109E+12	2.0500E+12
	(0.58)	(0.15)

Table 10. Compared Superficial and volumetric resistivity for	the
composites of NR with CB and WF.	

Values in parentheses correspond to the Coefficient of Variation.

or semiconductors because they have resistivity in the magnitude range between  $10^4$  to  $10^{8}$  [<sup>38,40]</sup>.

The resistivity of a material can be influenced by some factors, such as: acid doping, matrix conductivity, material thickness, polymerization method and synthesis of both the reinforcement and the matrix and the dispersion form of the reinforcement<sup>[41,42]</sup>.

Other studies report that the temperature influences the resistivity of the material, as well as the moisture of the reinforcement<sup>[19,43,44]</sup>.

Note that as wood fibers are added, which have a resistivity of  $10^{10} \Omega$ , there is an increase in the surface and volumetric resistivity of the composite, which can be attributed to the effect of the insulating capacity of dry wood, which has resistivity at around  $10^{10} \Omega$  and even when it reaches the fiber saturation point (PSF) it presents values of  $10^7 \Omega$ , contributing to the insulating capacity of the material.

Another point to be observed is that the interfaces of the materials inserted in the composite have different electrical conductivities and therefore, the electrical charges that are inserted in the material with friction and walking can become trapped in these interfaces (traps), thus being able to store or not the electrons.

What can be noted is that this insulating capacity is gradual, that is, it grows with the amount of fiber inserted, and it can be seen that the FM24/NF36 formulation is what, according to the standard, can be classified as antistatic because it presents results of resistivity in the magnitude of  $10^6 \Omega$ , that is, this formulation that mixes wood fibers and carbon black has the best potential for use in antistatic floors.

# 4. Conclusions

For the physical tests, there was no statistical difference for the basic density, however, as the addition of a fiber makes the composite less hydrophobic, there was an increase in water absorption. For the swelling test, there was a decrease in the swelling rate in treatments where there was a substitution of CB for WF. For mechanical tests, the hardness increased at a considerable rate with the addition of WF as a filler, with the treatment WF48/CB12 being the one with the greatest hardness. The tensile strengths were reduced in the treatments with the use of WF in the formulation, which is caused by the increase in hardness causing a faster rupture in the composite. The abrasion resistance was the lowest for the WF60/ CB0 treatment, as the difficulty in mixing may have caused agglomerates that may have caused greater abrasion on the composite surface. For technological analysis, there was no difference in the glass transition (Tg) value, the values being all close to -60°C which is compatible with rubber. This means that WF did not cause a structural change in natural rubber. The color changed in the WF60/CB0 treatment, making the composite lighter. The resistivity of the composite increased with the substitution of CB for WF, proving the possibility of using the wood rubber composite in high electric charge situations. Therefore, the synergistic use of CB and WF is an advance in the search for a more sustainable production for rubber floors that are not exposed to a high abrasion load, concluding that there is a technical viability in the use of WF as a replacement of CB.

# 5. Author's Contribution

- Conceptualization Renan Zunta Raia.
- Data curation Renan Zunta Raia; Setsuo Iwakiri; Rosilani Trianoski; Alan Sulato de Andrade; Edemir Luiz Kowalski; Aldo Eloizo Job; Fábio Friol Guedes de Paiva.
- Formal analysis Renan Zunta Raia.
- Funding acquisitions NA.
- Investigation Renan Zunta Raia.
- Methodology Renan Zunta Raia.
- **Project administration** NA.
- **Resources** Coordenação de Aperfeiçoamento de Pessoal de Nível Superior.
- Software NA.
- Supervision Setsuo Iwakiri; Rosilani Trianoski; Alan Sulato de Andrade; Edemir Luiz Kowalski.
- Validation NA
- Visualization NA.
- Writing original draft Renan Zunta Raia.
- Writing review & editing Setsuo Iwakiri; Rosilani Trianoski; Alan Sulato de Andrade; Edemir Luiz Kowalski.

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