

**Obtenção, caracterização e aplicação de um compósito com Resina de Poliéster e Folha de Pó de Carnaúba**

**Obtaining, characterizing and applying a Composite with Polyester Resin and Carnauba Leaf Petioles Powder**

**Obtención, caracterización y aplicación de un Compuesto con Resina de Poliéster y Polvo de Hoja de Carnauba**

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**Resumo**

A carnaúba, a árvore da vida, é uma palmeira encontrada em todas as regiões do Brasil e tem diversas aplicações, da eletrônica à cosmética, com maior destaque para a cera. O objetivo desta pesquisa foi obter compósitos utilizando fibras de pecíolos de folhas de carnaúba e resina de poliéster. O pó foi obtido por meio da moagem da forragem e posterior peneiramento, gerando resíduos com diferentes tamanhos de grãos. Todas as granulometrias obtidas foram testadas e optou-se pela menor, devido a sua maior viabilidade de obtenção do compósito, devido a sua melhor processabilidade. Foram escolhidas três proporções de massa da mistura entre matriz e resíduos, 5%, 7,5% e 10%, visto que acima desse percentual houve um comprometimento na processabilidade e obtenção do compósito estudado. Foram realizadas caracterizações mecânicas, térmicas e ambientais, que demonstraram a viabilidade do compósito proposto. O pó de carnaúba estava presente no compósito como carga de

preenchimento. A melhor formulação, 10%, foi escolhida para fazer uma parábola de um fogão solar em concentração para produzir alimentos para cozinhar. O compósito também pode ser utilizado na fabricação de painéis decorativos para suporte de televisores, bem como na fabricação de móveis.

**Palavras-chave:** Pó dos pecíolos das folhas de carnaúba; Compósito poliéster; Caracterizações mecânica, térmica e ambiental; Fogão solar.

### **Abstract**

The carnauba, the tree of life, is a palm tree found in all regions of Brazil and has many applications, from electronics to cosmetics, with a greater emphasis on wax. The objective of this research was to obtain composites using petioles fibers from carnauba leaves and polyester resin. The powder was obtained using forage grinding and subsequent sieving, generating residues with different grain sizes. All the obtained granulometries were tested and the smaller one was chosen, due to its greater viability to obtain the composite, due to its better processability. Three mass proportions of the mixture between matrix and residues were chosen, 5%, 7.5% and 10%, since above that percentage there was a compromise in the processability and obtaining the composite studied. Mechanical, thermal and environmental characterizations were performed, which demonstrated the feasibility of the proposed composite. The carnauba powder was present in the composite as fill charge. The best formulation, 10%, was chosen to make a parabola of a solar cooker at concentration to produce cooking food. The composite can also be used in the manufacture of decorative panels for the support of televisions, as well as in the manufacture of furniture.

**Keywords:** Petiole powder from carnauba leaves; Polyester composite; Mechanical thermal and environmental characterizations; Solar Cooker.

### **Resumen**

La carnauba, el árbol de la vida, es una palmera que se encuentra en todas las regiones de Brasil y tiene muchas aplicaciones, desde la electrónica hasta la cosmética, con mayor énfasis en la cera. El objetivo de esta investigación fue obtener composites utilizando fibras de pecíolos de hojas de carnauba y resina de poliéster. El polvo se obtuvo mediante trituración del forraje y posterior tamizado, generando residuos con diferentes tamaños de grano. Se ensayaron todas las granulometrías obtenidas y se eligió la más pequeña, por su mayor viabilidad para la obtención del composite, por su mejor procesabilidad. Se eligieron tres proporciones de masa de la mezcla entre matriz y residuos, 5%, 7,5% y 10%, ya que por

encima de ese porcentaje hubo un compromiso en la procesabilidad y obtención del composite estudiado. Se realizaron caracterizaciones mecánicas, térmicas y ambientales que demostraron la viabilidad del compuesto propuesto. El polvo de carnauba estaba presente en el material compuesto como carga de relleno. Se eligió la mejor formulación, 10%, para hacer una parábola de una cocina solar en concentración para producir alimentos para cocinar. El composite también se puede utilizar en la fabricación de paneles decorativos para el soporte de televisores, así como en la fabricación de muebles.

**Palabras clave:** Polvo de pecíolo de hojas de carnaúba; Compuesto de Poliéster; Caracterizaciones mecánicas, térmicas y ambientales; Cocina Solar.

## 1. Introduction

Composites are in great evidence in the field of materials, bringing a wide range of possible uses to replace conventional materials, for combining non-existent properties in a single material. Before a material was only considered composite if the load on the matrix was reinforced and predominated the synthetic fibers of glass, carbon fiber and Kevlar. With the diversity of Brazilian fauna and flora, with a multitude of vegetal specimens, the natural fibers appear as a great possibility of using such species as filling load in composite materials. Another possibility of use in composite materials came mainly from the ecological side, with the withdrawal of solid waste environment from industrial processes ((Souza, 2018), (Rodríguez Millán, Moreno, Marco, Santiuste, & Miguélez, 2016), (Khurshid, Hengstermann, Hasan, Abdkader, & Cherif, 2020), (Zaini, Azaman, Jamali, & Ismail, 2020), (Dong, 2018), (Ali et al., 2018), (Thakur, Thakur, & Gupta, 2014), (Zhu, Zhu, Njuguna, & Abhyankar, 2013) e (Koyuncu, Karahan, Karahan, Shaker, & Nawab, 2016). The use of composite materials has shown a significant dimension in several industrial areas worldwide. In the automotive, naval, aeronautical, ballistic and artisanal areas, such insertion has become a priority due to characteristics of good aesthetics, lightness and significant thermal and acoustic resistance ((Annamalai & Ramasubbu, 2018), (Bodur, Englund, & Bakkal, 2017), (Sathishkumar, Naveen, Navaneethakrishnan, Satheeshkumar, & Rajini, 2017) and (Singh, Kumar, Kumar, & Mohapatra, 2017).

The carnauba, whose scientific name in Brazil is *Copernicia Prunifera*, is a palm tree that inhabits the riverbanks of the Northeast region and produces a wax of high economic and social value. It called the Tree of Life, because it lends itself to an infinity of uses to man. Carnauba is used in more than 60 products, from electronics to cosmetics. Because it is a

native plant, carnauba does not require fertilization, agrochemicals, or agricultural mechanization. The greatest applicability of a by-product of carnauba is wax. Young leaf wax is used in the manufacture of candles, plastic and photographic films, varnishes, lubricants, phosphors, insulators, cosmetic ((Carvalho, 2011), (Freitas, 2011),(Düşünceli, Akyüz, Şahin, & Duru, 2019) and (Batista, 2014)). Wax production using carnauba is the most widespread academic topic in the use of carnauba. The use of carnauba leaf fibers to obtain composite materials has only been found in a few articles, only in Brazil, in the North and Northeast regions ((Carvalho, 2011), (Freitas, 2011), (Marques, 2012), (Costa, 2015), (Costa et al, 2013) and (Melo, Carvalho, Medeiros, Souto, & Paskocimas, 2012)).

(Marques, 2016) studied a biodegradable composite with carnauba straw powder and chitosan matrix. It demonstrated that the carnauba powder had a filling load function, as there was a decrease in the mechanical properties, caused by the low matrix adhesion - carnauba powder. (Costa, 2015), studied a composite composed of kaolin and leaf powder of the carnauba. The carnauba powder was added in three mass proportions, 10, 30 and 50%. The good thermal viability of the composite was demonstrated as a thermal insulator for temperatures up to 150°C, its economic viability and its limited applicability. (Costa, et al., 2013) studied a composite of carnauba fibers and a polyester resin matrix. They analyzed the influence of the length of the carnauba fibers and found that tensile strength, although lower than that of the matrix, presented better results for the longer fibers. (Melo, Carvalho, Medeiros, Souto, & Paskocimas, 2012) used carnauba fibers for the obtaining of a biodegradable composite was evaluated. Fiber treatments to improve interfacial bonding were carried out by alkali, peroxide, potassium permanganate and acetylation. Biodegradable composites were prepared using carnauba fibers and polyhydroxybutyrate (PHB) as matrix. The tensile strength of the composites made from peroxide treated fibers was superior. SEM observations on the fracture surface of the composites suggest improved fiber–matrix adhesion after peroxide treatment.

The objective of this research was to obtain composites using petioles fibers from carnauba leaves and polyester resin. The resin was chosen due to its easy processability at low temperatures, low cost and easy acquisition ((Souza, 2018), (Gomes et. al, 2015), (Saltan & Akat, 2019) and (Djeghader & Redjel, 2019)). Fibers of different thicknesses and sizes were obtained, reaching a fiber type practically powder, which was the granulometry of better performance in the processability of the composite. Preliminary tests showed the saturation of the mass of the fibers in relation to the resin, above 15%. Therefore, three formulations to be tested were chosen: 5.0%, 10% and 15%. As applicability of the composite was manufactured

a parabola, which represents the main element of a solar cooker at the concentration that was manufactured and tested to provide food cooking ((Filho, 2013), (Marques, 2016) and (Júnior, 2016)).

The main innovations are the use of the fiber of the petioles of the leaves of the carnation and a thermo mechanical characterization of the formulations studied, not found in the literature specialized in composite materials, besides the applicability in the manufacture of a solar prototype of great viability of use and great social outreach

## **2. Materials and Methods**

The materials and methods used to obtain the composite and fabrication of the test specimens and the solar prototype are presented, as well as the characterization of the proposed composite, obtained and studied. Subsequently, tests were carried out to characterize the composite formulations, following the relevant standards ((Souza, 2018), (Carvalho, 2011), (Düşünceli, Akyüz, Şahin, & Duru, 2019), (Batista, 2014), (Marques, 2012), (Melo et al., 2012), (Gomes, De Souza, De Souza Filho, & Santos, 2015), (Filho, 2013), (Marques, 2016), (Gomes, Godoi, De Souza, & De Souza, 2017), (Sathishkumar et al., 2017), and (Standards ASTM)).

### **2.1. Materials**

The steps to obtain the petiole powder of the carnauba leaves consisted of removing the leaves of the carnauba tree; removing the long fibers from the leaves; drying the fibers in a solar dryer; grinding the forage fibers, sifting the fibers and consequently obtaining the powder, with 2.0mm granulometry.

The chosen matrix was a polyester resin because it is easy to make, low cost, ideal to be worked at room temperature, easy to process and low risk of non-handling. To accelerate the curing process for resin, 2.0% of Butanox M-50 was used, relative to the mass of the matrix. The catalyst (MEK-P) was used to accelerate the curing process of the resin and the carnauba wax as a release agent to obtain better finished surfaces and facilitate the removal of the composite plates from the molds.

For the manufacture of composite plates for mechanical tests, two metal molds with dimensions according to specific standards were used. The mold 01 for traction and flexion, with 200 mm x 200 mm x 8 mm and the mold 02 for the impact with 120 mm x 120 mm x 10

mm. For the thermal tests, the metal mold 03 with the dimensions of 50 mm of internal diameter and 50 mm of height was used. For the compression of the molds, a hydraulic press with a capacity of up to 15 tons was used.

## 2.2. Methods

The obtaining of the composites was tested to determine the mass quantities of resin and carnauba leaf petioles powder (CLPP), that is, the maximum amount of CLPP mixed with the resin, which would guarantee a viable processability by the manual process of mixing between the components. Above 15% by weight of the petioles powder in relation to the resin, the processability of the composite was not viable due to the difficulties in homogenizing the material and its fluency when being deposited in the molds.

Therefore, four formulations were chosen, three of the composites and one of the polyester resin. The numerical nomenclature used is related to the amount of CLPP contained in the resin. The specimens were manufactured with the CLPP mass proportions of 5%, 7.5% and 10%, referring to the polyester resin matrix. Table 1 shows the formulations of the composites and polyester resin.

**Table 1** - Nomenclature for polyester resin and composites.

Matrix/Composites	Formulations
Resin	1.0 PR + 0.0 CLPP
C <sub>1</sub>	1.0 PR + 0.05 CLPP
C <sub>2</sub>	1.0 PR + 0.075 CLPP
C <sub>3</sub>	1.0 PR + 0.10 CLPP

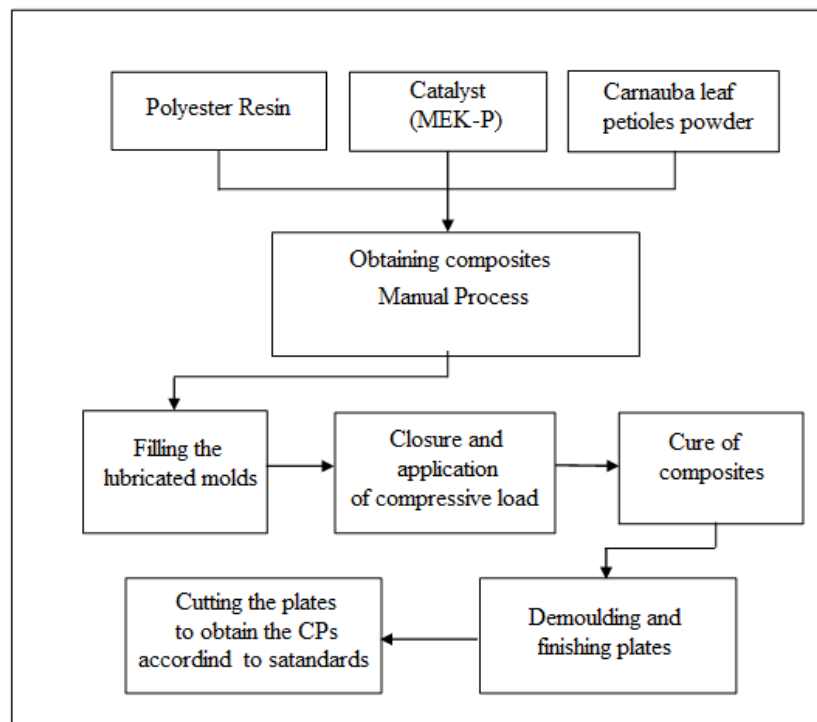
Source: Authors (2020).

Depending on the volume of the molds, the mass quantity of resin and petioles powder was defined using a precision scale. The manufacturing process used in this work was that of closed molding by cold wet compression. Then, the carnauba wax was applied to the mold and it was expected to dry. Soon after, the resin, the petiole powder and the catalyst were weighed on a precision scale.

In a disposable container, 2% by mass of the catalyst (MEK-P) was added. This solution was stirred manually, and then CFPP was added in the proportions of each composition. After homogenization, the molds were filled, waiting between 10 to 15 minutes until the mixture reached the ideal viscosity, to close the molds and apply a compressive load

of two tons. After 24 hours under pressure, the composite plates were demoulded and burrs and protrusions were removed using sandpaper and the specimens were cut with a bench saw, meeting the measures required by each standard. The flowchart of Fig. 01 presents the main stages of the processes for obtaining and manufacturing the samples of the polyester resin and CLPP composites for the characterization tests.

**Figure 1.** Steps in the manufacturing process of the specimens.



Source: Authors (2020).

For the characterization of composites, the tests of traction, flexion, impact, density, water absorption, thermal properties, aging, thermogravimetric analysis (TGA) and scanning electron microscopy (SEM) were performed, where each parameter was analyzed according to the standards pertinent. The performance of each test followed the guidelines of the corresponding ASTM standard. All tests were performed with controlled temperature and humidity. Six specimens were generally used for each test.

In the tensile and flexion tests, a SHIMADZU universal machine model AGS-X 300 was used. The tensile test was performed according to ASTM D3039 / D3039M-17 and the flexion test ASTM D7264 / D7264M-15. In the Sharp impact test, an analog pendulum of the LEIPZIG machine of 0.937 kg, length of the rod 220 mm was used, with the methodology

adopted by ASTM D6110-18.

The density test used was a digital densimeter Gehaka model DSL 910. The CPs were 25 mm X 25 mm X 8 mm thick. The test followed the ASTM D792-13 standard. The water absorption test was carried out for distilled and sea water. The CPs were 25 mm X 25 mm X 8 mm. CPs were weighed before immersion and every week for 60 days. The standard followed was ASTM D570-98.

In the thermal properties test, a portable analyzer kit from Decagon Devices model KD2 Pro was used. The CPs were 50 mm in diameter and 50 mm high. The test followed the ASTM D5930-17 (2017) 27 standard. At TGA, a simultaneous thermogravimetric and calorimetric analyzer from TA Instruments model SDT Q600 was used. The test was carried out in an atmosphere of synthetic air at 50 ml/min, using a platinum crucible, heating rate of 10°C/min and a final temperature of 700°C. It was performed for a sample of the matrix resin and a sample of the composite with the highest proportion of CLPP, in samples with 5 mm X 5 mm X 3 mm. The procedure for TGA followed ASTM E2550-17.

In the aging test, one specimen of resin and one of each composite were positioned in a location with no obstacles to sun exposure and with zero inclination, on a covering slab and were exposed to the conditions of natural aging for one year. The CPs were cut with 200 m X 25 mm and 8 mm. Before and after exposure the PCs were weighed and photographed to analyze the degradation. The test followed the ASTM D1435-13 standard.

In the scanning electron microscopy (SEM) test, the Hitachi Scanning Electron Microscope model TM 3000 was used. The fracture surfaces of the composite samples were analyzed after tensile testing, in order to observe the influence of the manufacturing process the quality of the resin/CLPP interface and, consequently, the distribution (homogeneity) of the CLPP particles after mixing, in addition to assessing the formation and propagation of damage in the rupture section. The test followed the ASTM E1382-97 standard.

### **2.3. Composite Applicability**

The parable of a concentration solar cooker was manufactured. The resin used to obtain the composite was of the orthophthalic unsaturated polyester type. The petioles of the carnauba leaves were used and a manual mixing technique was used. After mixing the resin, the spill occurred in the mold, a parabola of 1.14 m in diameter and 1.0 m<sup>2</sup> of area ((Filho, 2013), (Marques, 2016) and (Junior, 2016)). Figure 2 shows some steps in the process of manufacturing the parabola of the solar cooker.



**Figure 2.** Steps in the process of manufacturing the parable.



Source: Authors (2020).

The composite used was C3 (1.0R + 0.10 CLPP), seeking a greater homogeneity in the mixture and taking care that the viscosity of the composite did not bring great difficulties in its application in the mold. The mirrored surface of the parabola was obtained using plastic mirror segments, which provided the advantage of being able to work with segments of high dimensions. In the tests, the temperature data at the bottom of the pan (Focus Temperature) and the water/food temperature were measured with a chromel-alumel thermocouple coupled to a digital thermometer (Minipa MT-306). Figure 3 shows the solar cooker in operation to produce the cooking of food.

**Figure 3.** Concentration solar cooker in operation.



Source: Authors (2020).

### 3. Results and Discussions

The results and discussions resulting from the characterization process of the studied composite formulations and the polyester matrix resin are presented.

#### 3.1. Mechanical tests

Table 2 presents the results of the mechanical strength tests of the composite and polyester resin formulations.

**Table 2.** Results of the mechanical strength tests for composites and matrix.

Matrix/Composites	Tensile strength (MPa)	Flexural strength (MPa)	Impact resistance (J/cm <sup>2</sup> )
Resin	19.625 ± 0.525	73.802 ± 0.963	0.29 ± 0.017
C <sub>1</sub>	14.272 ± 0.465	35.323 ± 0.4397	0.29 ± 0.012
C <sub>2</sub>	13.115 ± 0.423	32.573 ± 0.385	0.30 ± 0.017
C <sub>3</sub>	12.912 ± 0.332	27.525 ± 0.288	0.30 ± 0.015

Source: Authors (2020).

As the quantity of CLPP increased, the tensile strength decreased, showing fragility, obtaining the worst result for C<sub>3</sub>, with a 34.3% decrease in relation resin. In the analysis between composites, it was noticed that the biggest drop occurred between C<sub>1</sub> and C<sub>3</sub>, corresponding to 20.0%. Therefore, there was a minor loss of tensile strength, since the amount of CLPP doubled.

The deformation behavior was identical to that of the tensile strength, presenting lower values than matrix and with a decrease with the increase of the CLPP load, with a corresponding increase in the elasticity module. The modulus of elasticity was greater than that of the matrix resin, for all formulations, demonstrating an increase in the fragility of the composite, as the percentage of CLPP increased. The composite with the highest CLPP load, C<sub>3</sub>, presented an elastic modulus of 0.754GPa. Considering that the price of CLPP in relation to that of resin is insignificant, the increase in the load of CLPP brought greater economic viability for the composite, without significantly compromising its tensile strength.

There was a decrease in the flexural strength of the composites in relation to the matrix. The composite with the highest percentage of CLPP, C<sub>3</sub>, had a loss of 62.8%. The composite with the greatest flexural strength was C<sub>1</sub>, 22.1% higher than C<sub>3</sub>. The reduction in tensile and flexural strength was already expected, since particulates, as shown in the

literature, decrease the mechanical strength of composites, when compared to the matrix. Despite the good miscibility of the CLPP particles, the discontinuity generated in the matrix caused a decrease in strength, due to the increased fragility of the composite, mainly for composites with a higher percentage of CLPP load. Even with the good homogeneity of the mixture of polyester resin and CLPP, as the processability was manual, the occurrence of voids and agglomerates of CLPP, verified in the SEM images, brought fragility to the composites, when compared to matrix resin.

Regarding impact resistance, the composites showed results very close to those of polyester resin, demonstrating the fragility of the composites produced. Once again, the filling function of CLPP in the composites was proven. The energy absorption capacity remained practically constant with the increase in the amount of CLPP. The increase in the absorption capacity was insignificant, in the same order as the precision of the equipment used, and can even be considered as a parallax error when reading it.

### 3.2. Apparently Density

Table 3 presents the results of the apparent density tests of the composites and polyester resin.

**Table 3.** Results of apparent density of composites and polyester resin.

Matrix / Composites	Apparently density (g/cm <sup>3</sup> )
Resin	1.241 ± 0.09
C <sub>1</sub>	1.179 ± 0.07
C <sub>2</sub>	1.147 ± 0.05
C <sub>3</sub>	1.143 ± 0.05

Source: Authors (2020).

The apparent density of the composites was always lower than that of polyester resin. The lower density of CLPP corresponding to 0.33g/cm<sup>3</sup> caused this property to drop in composites, in relation to resin. A decrease in density occurred as the proportion of CLPP in the resin increased. The composite with the lowest amount of CLPP, C<sub>1</sub>, showed a density 5.0% lower in relation to the polyester resin and the highest amount, C<sub>3</sub>, of 7.9%. Despite the decrease in the density of composites being an important characteristic in the search for lighter materials, the levels were not significant, requiring a much higher mass ratio of CLPP, which would make the processability of the composite difficult, making manual mixing

between resin and CLPP impossible. However, the density decrease represents an important factor to make the applicability of polyester and CLPP composites viable.

The decrease in apparent density in composites was due to the lower density of CLPP in relation to resin, supplanting the small effect of volume retraction when mixing CLPP and resin, which occurred mainly for the composite with the highest percentage of CLPP, C3. The good diffusion of CLPP in the resin produced a mixture of good miscibility, quite homogeneous, mainly due to its low particle size.

### 3.3. Water Absorption

Table 4 presents the results of the distilled water and seawater absorption test of the composites and polyester resin.

**Table 4.** Results of the distilled water and seawater absorption test for composites and polyester resin.

Matrix/Composites	Absorption of distilled water (%)	Absorption of marine water (%)
Resin	0.35 ± 0.02	0.48 ± 0.03
C <sub>1</sub>	5.01 ± 0.12	5.17 ± 0.14
C <sub>2</sub>	5.42 ± 0.14	5.59 ± 0.16
C <sub>3</sub>	5.72 ± 0.18	5.96 ± 0.19

Source: Authors (2020).

The composites showed an absorption capacity much higher than that of polyester resin. This behavior of greater absorption was due to CLPP is hygroscopic, as it has 41.5% cellulose, 28.9% hemicellulose and 14% lignin, substances that are well compatible with water. Due to the greater water absorption capacity of CLPP, when compared to resin, there was an increase proportional to the growth of the mass proportion of CFPP, with greater absorption for C3, for both aqueous media. This behavior was already expected, but the water absorption capacity for the two aqueous media studied was low, reaching a maximum of 5.72% for distilled water and 5.96% for sea water.

The results indicated that all the composites had distilled water absorption capacity much greater than that of the polyester resin. Despite the low CLPP load on the resin, there was a very significant increase in the absorption capacity of the composites, with a better result for the one with the lower CLPP load, C1. Among the composites with the lowest and highest CLPP load, C1 and C3, the increase in absorption capacity corresponded to 14.2% in

distilled water and 15.5% in sea water. The absorption capacity in sea water was greater for composites and polyester resin in relation to the absorption of distilled water, caused by the greater number of chemical elements in salt water, generating a greater ionization force than distilled water, producing greater interactions with the resin, and causing increased water absorption. However, this increase was not significant, reaching a maximum of 4.2%.

### 3.4. Thermal Tests

Table 5 presents the results of the thermophysical properties of thermal conductivity, volumetric thermal capacity, thermal diffusivity and thermal resistance (resistivity), of composites and polyester resin.

**Table 5.** Results of thermal properties for composites and polyester resin.

<b>Matrix / Composites</b>	<b>Conductivity K (W/m.K)</b>	<b>Resistivity R (K.m/W)</b>	<b>Thermal Capacity C (MJ/m<sup>3</sup>.K)</b>	<b>Diffusivity D (mm<sup>2</sup>/s)</b>
Resin	0.174	5.75	1.748	0.099
C <sub>1</sub>	0.160	6.27	1.675	0.096
C <sub>2</sub>	0.158	6.33	1.655	0.095
C <sub>3</sub>	0.136	7.35	1.465	0.093

Source: Authors (2020).

The thermal behavior of composites was superior to that of polyester resin, for thermal insulators, due to the conductivity of CLPP, 0.12 W/m.K, being inferior. There was an improvement in the thermal properties analyzed, K, C and D, as the amount of CLPP in the polyester resin increased. The composite with the lowest thermal conductivity was C3, 21.8% lower than the polyester resin and the one with the highest thermal conductivity was C1, with an 8.0% decrease.

The behavior of resistivity, resistance to the passage of the thermal wave, was inversely proportional to that of thermal conductivity, increasing as the amount of CLPP in the composite increased. The volumetric thermal capacity decreased with the increase for CLPP in the resin. The composite with the lowest volumetric thermal capacity was C3, with a decrease of 4.2% in relation to the resin and 12.5% in relation to with the highest volumetric thermal capacity, C1. Once again, the quantity of CLPP had a great influence on the thermal properties of the composites obtained. The same behavior occurred in thermal diffusivity, with little significant decrease as the amount of CLPP increased, with a maximum variation between C3 and C1 of 3.2%. Regarding polyester resin, the variation in diffusivity was also

not very significant, with a maximum variation of 6.0%.

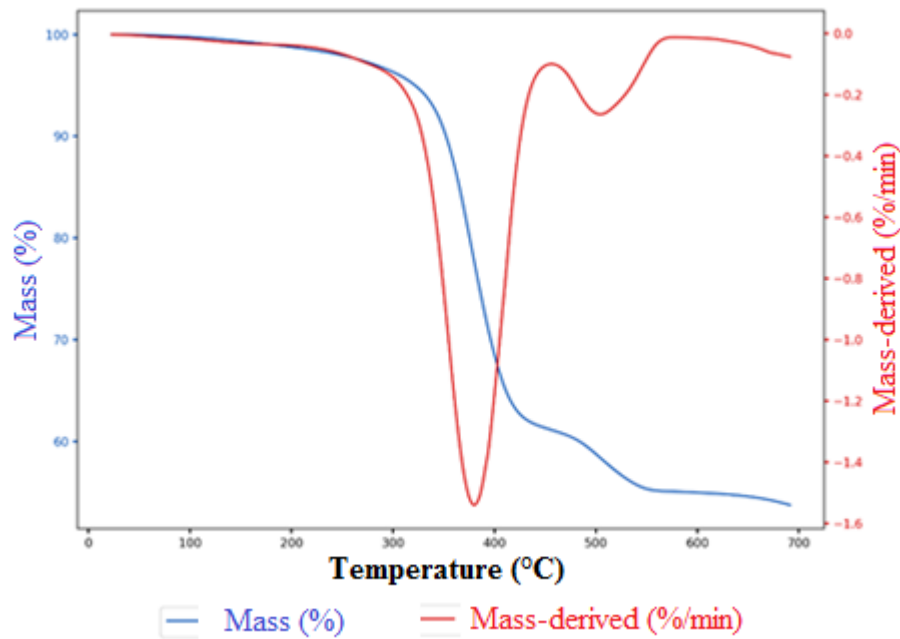
The good thermal behavior of the composites possibilities of using the composites with CLPP for applications as thermal insulator, since it presented thermal conductivities below 0.21 W/m.K. Few composites already obtained and studied have this level of thermal conductivity. The improvement in the thermal insulation properties of the composites in relation to the resin was significant, and can be used, for example, for the manufacture of tubes for conducting fluid at low temperatures.

### **3.5. Thermogravimetric Analysis (TGA)**

Figures 4 and 5 show the results of the thermogravimetry test performed with a sample of the composite and the polyester resin. The graphs contain the sample mass curve as a function of temperature (TGA curve - thermogravimetry) and the derivative of that curve (DTG curve - derived thermogravimetry), which provides information on the first derivative of the variation in mass as a function of temperature and allows to identify in how many steps a thermal degradation occurs.

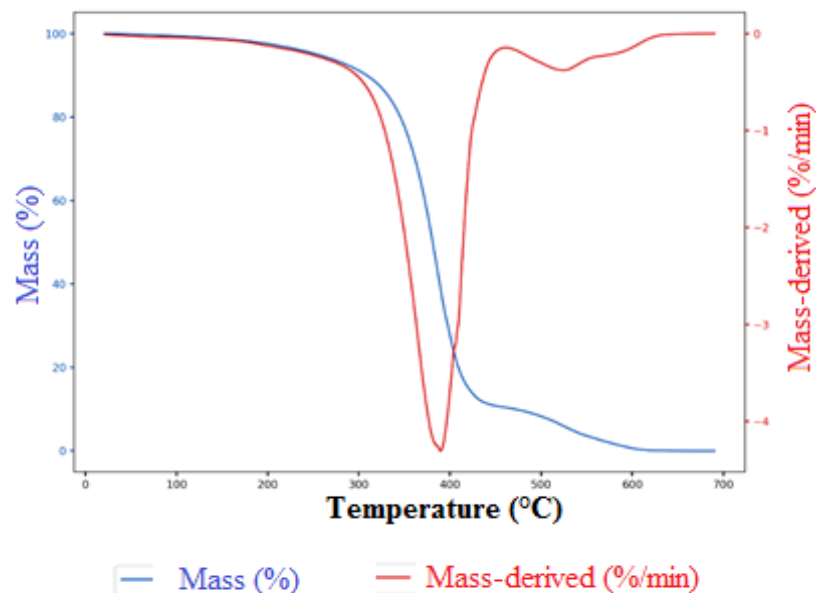
This test was carried out to check the thermal stability and degradation of the resin, 1.0 R + 0.0 CFPP and 1.0R + 0.10 CLPP (C3). This technique determines important properties, allowing the evaluation of possible applications of a material.

**Figure 4.** Thermogravimetric curve of polyester resin.



Source: Authors (2020).

**Figure 5.** Thermogravimetric curve of composite C3.



Source: Authors (2020).

There were no significant differences in mass loss between the matrix and composite C3. CLPP did not have a greater heat resistance capacity. The residual mass after 600°C, for

matrix and composite, was less than 1.0%. Table 6 presents the values of mass loss for matrix and composite C3 as a function of temperature in the TGA test.

**Table 6.** Percentage values of mass loss for matrix and C3 composite in the TGA test.

Temperature (°C)	RESIN (%)	C <sub>3</sub> (%)
100	0.0	0.5
300	7.1	8.3
350	17.2	23.3
400	74.1	72.2
500	92.0	91.7
600	99.1	99.4
690	99.3	99.9

Source: Authors (2020).

As the process of obtaining the composite took place at room temperature, by cold pressing, the loss of mass was insignificant, not causing major concerns of loss of mass when the applicability of the composite. Once again, the influence of the petioles load in relation to temperature resistance was evidenced.

According to the ASTM E2550-17 standard, it is necessary to determine the mass variation and the onset temperature (temperature of onset of loss of mass) of each reaction observed during the test. As the degradation of the resin and that of the carnauba fiber occurred in the same temperature range, it was not possible to distinguish them. Thus, the values required by the standard were calculated for the resulting degradation reaction. This calculation took place through a mathematical and geometric analysis of the data that make up the TGA and DTG curves. The values required by the standard, as well as the peak temperature, which corresponds to the maximum degradation temperature are shown in Table 7.

**Table 7.** Tonset, Tpeak and mass loss values for polyester resin and composite C3.

Matrix/Composite	T <sub>Onset</sub> (°C)	T <sub>Peak</sub> (°C)	Mass loss (%)
Resin	351.07	386.88	99.28
C <sub>3</sub>	346.2	389.3	99.9

Source: Authors (2020).

Regarding the temperature of onset of mass loss, it was identified that the resin needed a higher temperature to start the thermal degradation process, being more thermally stable. This is explained by the fact that the carnauba fibers degrade at lower temperatures than the



resin. The peak temperatures of the mass loss were practically equal and at the end of the test the mass loss was greater than 99% for matrix and formulation. It is important to note that for most applications, which are subjected to temperatures close to that of the environment, the composite will not undergo thermal degradation. For use in a temperature range that causes thermal degradation, additives must be used, which increase the thermal stability of the materials.

### 3.6. Aging Test

Table 8 presents the results of the variation of the mass of the specimens submitted to the aging test, in a natural environment of the composites and polyester resin.

**Table 8.** Results of the mass variation of the aging tests of composites and polyester resin.

Matrix / Composites	Initial mass (g)	Final mass (g)	Weight loss (%)
Resin	22.15	22.09	0.27
C <sub>1</sub>	20.62	20.34	1.36
C <sub>2</sub>	18.36	18.07	1.58
C <sub>3</sub>	19.66	19.28	1.93

Source: Authors (2020).

All composites showed mass loss percentages much higher than that of polyester resin, however not significant for one year of exposure, with losses below 2.0%. The loss of mass caused by the degradation related to exposure to bad weather showed an increasing behavior with the increase in the percentage of CLPP. The composite with the lowest percentage of loss was C1 and the highest percentage was C3.

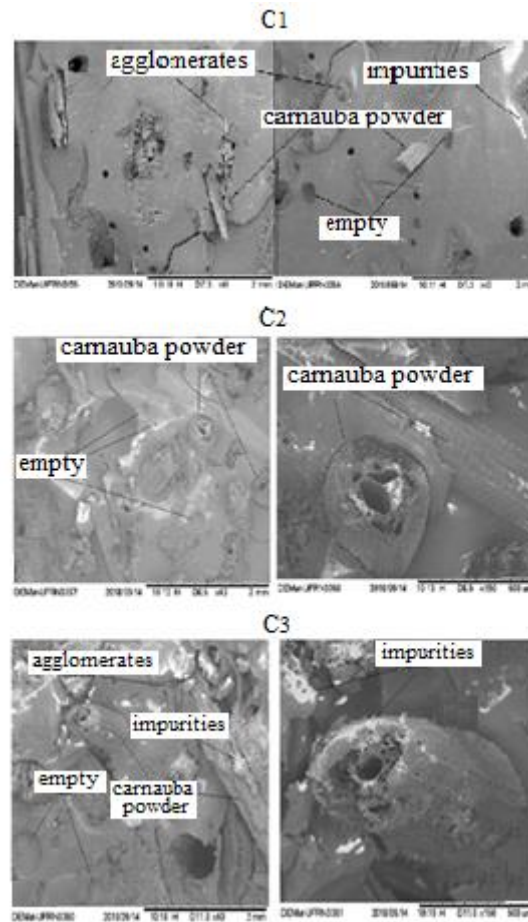
Therefore, the most viable composite for exposure to the weather and climatic variations, in all seasons, was C1, but of higher cost since it has a smaller amount of cement powder in the polyester resin. Combining technical and economic conditions, the most viable composite was C3. The main change in the samples before and after exposure, in the period of one year (365 days), occurred in the color, mainly in the polyester resin that acquired a more yellowish hue. The samples of the composite formulations acquired a darker tone.

### 3.7. Scanning Electron Microscopy (SEM)

Figure 6 shows the images of the scanning electron microscopy (SEM) test on the

fracture surfaces of samples subjected to traction for all composites.

**Figure 6.** The SEM of composites and highlighting the defects resulting from their manufacturing process.



Source: Authors (2020).

As the proposed composite material was particulate and the load used was not reinforcement, as the results of traction and flexion demonstrated, the main concern was the optimization of the process of obtaining and manufacturing the plates for the removal of the specimens for the tests of characterization.

It was tried to avoid the presence of contaminants that generate impurities, to obtain a very homogeneous mixture between the two constituents, minimizing the occurrence of voids and agglomerates, which could bring an even greater fragility to the composite, in relation to the polyester resin. In order to minimize these effects, some precautions were taken, using new disposable containers, a reed mixer never used, a clean, organized work environment and a processing room at room temperature.

SEM images after fracture in traction showed the occurrence of fragile fracture,

presence of impurities, a small number of voids, pullout of agglomerates generated by the concentration of carnauba powder, mainly for formulations with a higher filling load, C2 and C3. These defects did not compromise the processing of the composite, nor the structural part of the manufactured parabola, since such a prototype did not need to present significant mechanical resistance to traction and flexion.

### 3.8. Solar Cook Tests

The solar cooker that had a parabola manufactured with the composite obtained and studied produced the cooking of several foods. Figure 7 shows the cooker in operation in the cooking operation of some of the foods tested.

**Figure 7.** Food cooked in the solar cooker manufactured from the studied composite material.



Source: Authors (2020).

## 4. Conclusions and Suggestions

The feasibility of obtaining the proposed composites has been demonstrated. Although they present much lower mechanical resistance than the matrix resin, they can still be used for applications with low mechanical load requirements.

The carnauba leaf petioles powder reduced the density of composites, in relation to resin, making them lighter, a characteristic that can increase their possibilities of applicability and provided an increase in water absorption, with growth as the quantity of PPFC increased, with greater absorption for sea water.

The composites showed thermal behavior superior to that of the resin for thermal insulators and similar mass loss in the TGA, insignificant for temperatures below 300°C,

which brings great processing viability for the composite.

The composites showed a loss of mass greater than the resin in the aging, however little significant, and an increasing behavior with the increase of the percentage of CLPP.

The scanning electron microscopy showed the occurrence of voids, impurities and agglomerates and surfaces showing a fragile fracture, due to the deficiency of adhesion between the CLPP and resin, causing damage to the mechanical strength of the composites, showing that the manufacturing process of composite boards needs to be optimized, using a mechanized mixing.

The most economically viable composite was C3, which can be used to manufacture solar/wind panels, furniture and prototypes. The feasibility of practical use of the composite was demonstrated, and a parabola was manufactured for a solar cooker at a concentration of 1.0m<sup>2</sup>.

For the manufacture of the parable of the solar cooker, it would be important to use an additive that would increase the viscosity of the resin for a better distribution in the mold, in the process of pouring the composite, which could be plaster or sodium bicarbonate.

The low adhesion of fiber and matrix has been demonstrated, and in other works, it is necessary to produce the composite with other resins, for example, epoxy resin.

It would be important to perform a chemical treatment on the carnauba leaf petioles powder (CLPP) to improve adhesion with the polyester resin matrix

One could hybridize the studied composite with another natural fiber, jute, açai, sisal, piassava and characterize it, comparing its results with those obtained for the studied composite.

Due to the good aesthetics of the composite produced with the carnauba leaf petioles powder (CLPP), we intend to manufacture panels for TV sets and decorative furniture for living rooms, bedrooms and kitchens

The acoustic behavior of the composite produced with the carnauba leaf petioles powder (CLPP) could be evaluated.

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