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Development of Epoxy Matrix Nanocomposites Reinforced with Coconut Shell Nanocellulose

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As a strategy to reduce socio-environmental impacts, the incorporation of waste or natural fibers from various sources into the formulation of composites has proven to be an attractive and sustainable alternative. In the development of nanocomposites specifically, the use of coconut fiber waste at the nanoscale has been explored both for its ability to enhance the mechanical properties of materials and as a way to add value to an agro-industrial residue that would otherwise be discarded into the environment. In this context, the present work aimed to develop epoxy matrix nanocomposites using nanocellulose extracted from coconut husk fiber. The polymer matrix used was an epoxy system composed of diglycidyl ether of bisphenol A/diethylenetriamine (DGEBA/DETA), with a hardener-to-resin ratio of 16:100 (16 phr). The nanocellulose was extracted through chemical treatments of the coconut husk, using sodium hydroxide (NaOH) and sodium hypochlorite (NaClO) to remove lignin, hemicellulose, and other impurities. Subsequently, to eliminate the amorphous region of cellulose and obtain nanofibrils, the material underwent acid hydrolysis with sulfuric acid (H_2SO_4). The nanocomposites were prepared in an open mold by incorporating nanocellulose at weight fractions of 0, 1.25, 2.5, and 5% relative to the total mass of the epoxy system. The obtained materials were characterized through compression strength tests, Izod impact resistance, and thermal stability analysis (thermogravimetry). The results showed the influence of nanocellulose addition on the mechanical and thermal properties of the epoxy system, demonstrating the technical feasibility of applying this material as reinforcement in composites within the evaluated context.

Keywords: *Nanocomposites, nanocellulose, epoxy resin, coconut fiber*

1. Introduction

Due to the technological and economic development of new materials with specific properties, composites reinforced with synthetic fibers have been gaining ground in the automotive, aerospace, maritime, and sports, military, and energy generation industries[1,2]. Carbon fiber-reinforced composites are made of a polymer resin and carbon fibers as reinforcement to improve the mechanical properties of the composite, with the polymer acting as the matrix to bind the fibers and protect them from the environment. The interface between the matrix and reinforcement is a key component of composites, as it is directly related to the effective transfer and dispersion of loads between the matrix and the reinforcement. Consequently, it determines strength and toughness, in addition to offering high strength, modulus, and thermal stability[1,3-5].

Despite their attractive advantages, the use of synthetic fibers carries significant drawbacks due to high manufacturing costs, high energy consumption, the use of non-renewable raw materials, wear on processing equipment, and the environmental impact caused by their poor degradability[6]. As a sustainable alternative, natural fibers are being studied to replace synthetic ones, as they are renewable, biodegradable, and widely available, while also helping to minimize environmental impacts resulting from improperly discarded waste[7,8]. Moreover, they offer lower abrasiveness, low density, non-toxicity, and cost-effectiveness[3,9], making them suitable for use in various aerospace[10] and automotive[11] applications, among others.

The increasing demand for natural fiber substitutes as polymeric reinforcements in recent years includes plant fibers such as hemp, sisal, coconut fiber, jute, kenaf, and ramie[12-19]. Furthermore, the biodegradability of natural fibers ensures a lower environmental impact at the end of their life cycle, reducing waste and promoting sustainability in industrial practices[20]. Therefore, natural fiber reinforcement can contribute to the production of lightweight composite materials with excellent properties and abundant availability in nature. Among the various types of fiber, coconut fiber is one of the strongest natural fibers[21].

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The largest coconut producers in the world are Indonesia, the Philippines, India, and Brazil, with the latter producing 2.7 million tons annually[22,23]. The main solid byproduct is the husk, composed of coconut husk fibers and particles that can be used in the manufacture of new materials. However, only about 10% of coconut husks are currently utilized in Brazil, indicating a need for applications that add value to this waste to encourage its greater use[24].

Coconut husk fiber is mainly composed of cellulose, lignin, and hemicellulose, with average proportions of approximately 44%, 33%, and 12%, respectively, and is characterized by a relatively low concentration of hemicellulose and lignin[25,26]. These components can weaken the bond between the matrix and the fiber in composites. Chemical treatments used to produce cellulose nanofibrils remove all interfering components, leaving only nanocellulose, which increases surface area, crystallinity, and cellulose content[27]. Its structure typically has a diameter between 1 and 100 nm and a length ranging from 500 to 2000 nm. It is composed of both amorphous and crystalline cellulose domains, distributed along long, flexible fibrils that resemble filaments or structured webs. This structure results in a large specific surface area, low density, low thermal expansion, and a high elastic modulus. Additionally, nanocellulose stands out for being non-toxic, non-abrasive, biodegradable, and renewable[19,25,28,29].

In contrast to fiber reinforcements, nanocellulose exhibits a higher degree of interaction with the matrix, which contributes to improved mechanical and thermal properties[19]. Epoxy resin is widely used in the fabrication of composites due to its thermal insulation properties, fast curing at low temperatures, easy application, and excellent chemical stability[30,31]. The use of nanomaterials, such as nanocellulose, enables a larger contact area between the reinforcement and the matrix, which facilitates the development of composites with optimized mechanical and thermal properties[32,33].

The objective of this research is to formulate epoxy-based polymeric nanocomposites reinforced with nanocellulose from coconut fiber, and to characterize their mechanical and thermal properties.

2. Materials and Methods

2.1. Materials

2.1.1. Epoxy resin

The epoxy system used as a matrix was Diglycidyl Ether Bisphenol A (DGEBA)/Diethylenetriamine (DETA), known on the market respectively as SQ2050 and SQ 3131 and marketed by the company AVIPOL. The ratio of epoxy resin adopted in this work was 16 parts hardener to 100 parts resin by mass.

2.1.2. Coconut fiber

The coconut fibers were collected in Morro do Coco, a municipality located in the northwestern region of Rio de Janeiro. The fibers were removed from the shell of the coconut fruit, with the mesocarp fibers being ground in a knife mill to obtain a fine particle size (200 mesh). The fibers were then washed with distilled water to remove impurities. After this, the fibers underwent an initial heat treatment with distilled water, adopting a ratio of 1:20 (mass/volume), i.e. 10 g of fibers for every 200 mL of water, under constant agitation at 80°C for 2 hours, in order to remove particulates. After treatment, the fibers were dried in an air circulation oven at 60ºC for 24 hours.

2.2. Preparation of Nanocellulose

Coconut fiber nanocellulose was obtained through a process divided into 4 stages, the first two of which were carried out twice and each of which was separated by washing repeatedly with distilled water until the pH was neutralized. In the first stage, 10g of fibers were placed in a 2M NaOH solution at 60ºC for 6 hours. In stage 2, the solution was poured into a 5% NaClO solution at 30ºC for 1 hour. In stage 3, the solution was poured into 64% (w/v) acid hydrolysis for 2 hours at room temperature. In stage 4, the solution was dried by freeze-drying, according to the methodology adapted from Rosa et al.[34] and Azizi Samir et al.[35].

2.3. Preparation of the nanocomposites

The proportions of 1.25%, 2.5% and 5% (m/m) were used to process the nanocomposites. The percentage of nanocellulose for each nanocomposite was dispersed in 90 ml of acetone under magnetic stirring for 24 hours to prevent agglomeration in a closed container so as not to evaporate, then 80 g of epoxy resin was added. The mixture was sonicated in an ice bath for 1 hour at an amplitude of 60% and then, with the nanocellulose already dispersed in the resin, it was left in an open container for 24 hours to allow the acetone in the mixture to evaporate. After this procedure, 12.8g of hardener was added and mixed to homogenize. The mixture was poured into an open silicone mold previously prepared with mold release agent. After the 24-hour period, the specimens were removed from the mold and, after curing at room temperature, were subjected to the post-curing process at a temperature of 70ºC for 3 hours.

2.4. Characterization of nanocellulose

Initially, coconut fiber and nanocellulose were compared using a SHIMADZU IRTracer-100 Fourier Transform Infrared Spectrometer (FTIR). The wavelength used was between 4000 cm-1 and 500 cm-1.

Subsequently, the crystallinity index (CI) of the nanocellulose produced was determined using equation (1), based on measuring the intensities of the peaks corresponding to the crystalline and amorphous regions, obtained by X-ray diffraction (XRD)[36,37]. The analyses were carried out on a PROTO Manufacturing X-ray diffractometer, model AXRD Benchtop, scanning from 10 to 70° (2θ) at 2°/min.

$$CI(\%) = \frac{(Ip - Iam)}{Ip} x100\%$$

Where: CI: crystallinity index

- Ip: peak indices
- Iam: amorphous indices

2.5. Mechanical characterization

The compression test followed the ASTM D695 standard[38], using prismatic samples measuring 12.5 × 12.5 × 6.0 mm

(height × width × thickness). The equipment used was an Instron universal testing machine, model 5582, with a speed of 1.3 mm/min and a 100 kN load cell.

The impact test was carried out in the Izod configuration, using a PANTEC impact pendulum, model PAN 50J. The test was carried out using a 22 J hammer on specimens with dimensions of 12.5x12.5x60 mm, in accordance with ASTM D256[39].

The significance of the effects of adding nanocellulose on the mechanical properties was assessed using an analysis of variance (ANOVA), with a significance level of 5%. The procedure was carried out in Microsoft Excel, using the function described by Velasco et al. for calculating the p-value[40].

Finally, the fracture surfaces of the impact specimens were analyzed by High Resolution Electron Microscopy (SEM-FEG), using a Tescan model MIRA4. For this analysis, the samples were metallized with gold.

2.6. Thermal characterization

Thermogravimetric analysis (TGA) was carried out using thermogravimetric equipment SDT650 from TA Instruments. The analysis was carried out under a nitrogen atmosphere with a flow rate of 100 mL/min and a heating rate of 10ºC/min, between 0ºC and 1000ºC.

The Differential Scanning Calorimetry (DSC) analysis was carried out on TA Instruments' DSC 25 equipment. The test took place under a nitrogen atmosphere, with a flow rate of 100 mL/min, using a heating rate of 10 °C/min and covering the range from 0 to 1000 °C, in two runs.

3. Results and Discussions

3.1. Nanocellulose characterization

Figure 1 shows the spectra of coconut fibers and nanocellulose obtained by chemical treatment. Both spectra show no significant differences, indicating that the chemical reactions did not produce any new substances. The final product was cellulose itself and not a cellulose derivative[41].

In the first region, peaks at 3331 cm^{-1} and 2895 cm^{-1} can be observed, corresponding respectively to the stretching vibrations of the OH bonds from hydroxyl groups in the α-cellulose structure, and the same type of vibration in the C-H bonds of methylene and methyl groups in the cellulose chain. In the second region, the peaks at 1728 cm^{-1}, 1639 cm^{-1}, 1321 cm^{-1}, and 1018 cm^{-1} correspond respectively to C=O stretching, deformation vibrations due to residual moisture in the nanocellulose (which is hydrophilic in nature), deformation vibrations of the C-H and O-H bonds from CH_2 and OH groups in cellulose, and the stretching of C-O-C bonds in the glycopyranose rings, characteristic of cellulose. In the third region, two notable peaks are at 1018 cm^{-1} and 894 cm^{-1}, corresponding respectively to the stretching of C-O bonds in primary alcohol groups (C_6–OH) present in cellulose and the out-of-plane deformation of C-H bonds in the glycopyranose ring, associated with the β-glycosidic configuration of cellulose[42-45].

Figure 2 presents the XRD (X-ray diffraction) results, where the diffraction peaks of the coconut fiber nanocellulose can be seen at 2θ angles of 15.90º, 22.44º, and 34.25º, corresponding to the (110), (020), and (004) planes, respectively[46]. These peaks indicate that the nanocellulose has a type I structure[47-49]. The type I structure is characterized by microfibrillar organization composed of highly ordered cellulose chains interconnected by hydrogen bonds. It is predominantly allotropic in its crystalline organization—specifically cellulose Iβ—which is considered more stable and organized. With chains arranged in parallel, this structure exhibits high crystallinity with dispersed amorphous regions[50].

The crystallinity index observed was 77.8%, which is considered to be high, in line with both the previous assumptions about the type of nanocellulose and the values reported in the literature[51,52]. Due to its high crystallinity, the nanocellulose produced is expected to have a high modulus of elasticity and specific strength, indicating a potential use as a reinforcing material in composites[53,54].

3.2. Mechanical characterization

Table 1 and Figure 3 show that the use of nanocellulose reduces the impact resistance of the composites. The analysis of variance (ANOVA) indicates that this reduction was significant in all formulations.

Although significant according to ANOVA, this reduction is between 8 and 12%, and is in line with what is observed in

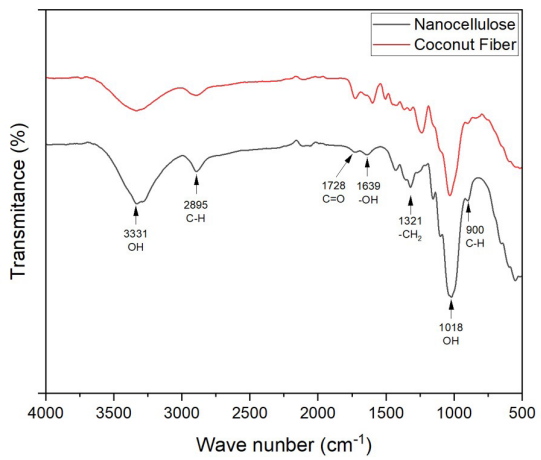

Figure 1. FTIR spectra of coconut fiber and nanocellulose.

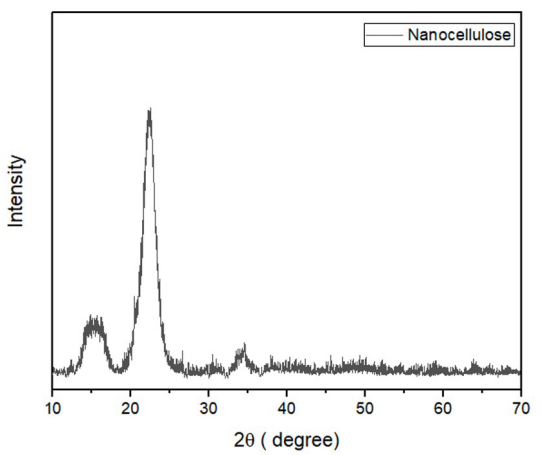

Figure 2. Diffraction of coconut fiber nanocellulose.

Table 1. Impact results (p-values in relation to the 0% formulation).

Amount of NC	Impact resistance (kJ/m^2)	Standard Deviation	Value-P
0.00%	2.91	0.07	-
1.25%	2.687	0.21	2.10%
2.50%	2.562	0.13	0.00%
5.00%	2.58	0.21	0.20%

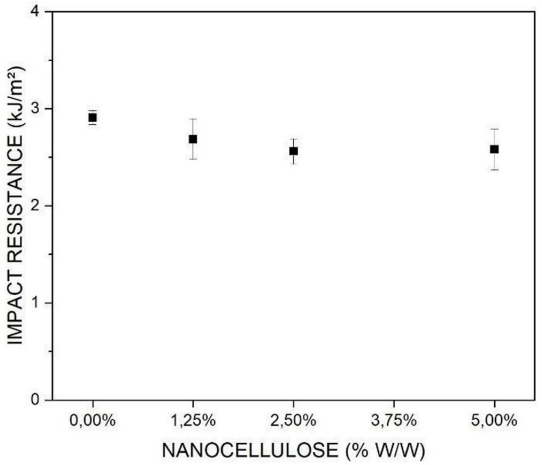

Figure 3. Impact results as a function of the amount of nanocellulose.

the literature, being due to the agglomeration of nanocellulose, as seen in Figure 4[55,56]. Agglomeration is due to the high specific surface area of the reinforcement, which favors its agglomeration in order to reduce surface energy[57]. In addition, the hydrophilic surface of the nanocellulose also favors its agglomeration[58,59].

Figure 5 shows the SEMs of the fracture surfaces of all the formulations, and it is possible to see river marks in all of them, characterizing brittle behaviour[60,61]. When comparing Figure 5 (a), the epoxy system without reinforcement, with the others, it can be seen that with the addition of nanocellulose, the surface of the sample is slightly more ductile. This indicates a tendency to increase impact resistance if the nanocellulose was not agglomerated, explaining why this result is in line with some of the literature and differs from the behavior observed by other authors[55,59].

The influence of the addition of cellulose on compressive strength varies according to its composition, size, shape, interface with the matrix, as well as its dispersion and distribution in the composites, indicating that different mechanisms can act to favor or reduce the performance of the composite[53,62-64]. The results of the compression test (Table 2) indicate that with the addition of nanocellulose in a proportion of 1.25%, there was a reduction in strength and modulus of elasticity, only the latter being significant according to the analysis of variance. Furthermore, with the addition of 2.5%, the reduction in both properties was significant, as can be seen in Figure 6. This is similar to the behavior observed by Véliz and collaborators[63], who observed that the addition of nanocellulose facilitated compression.

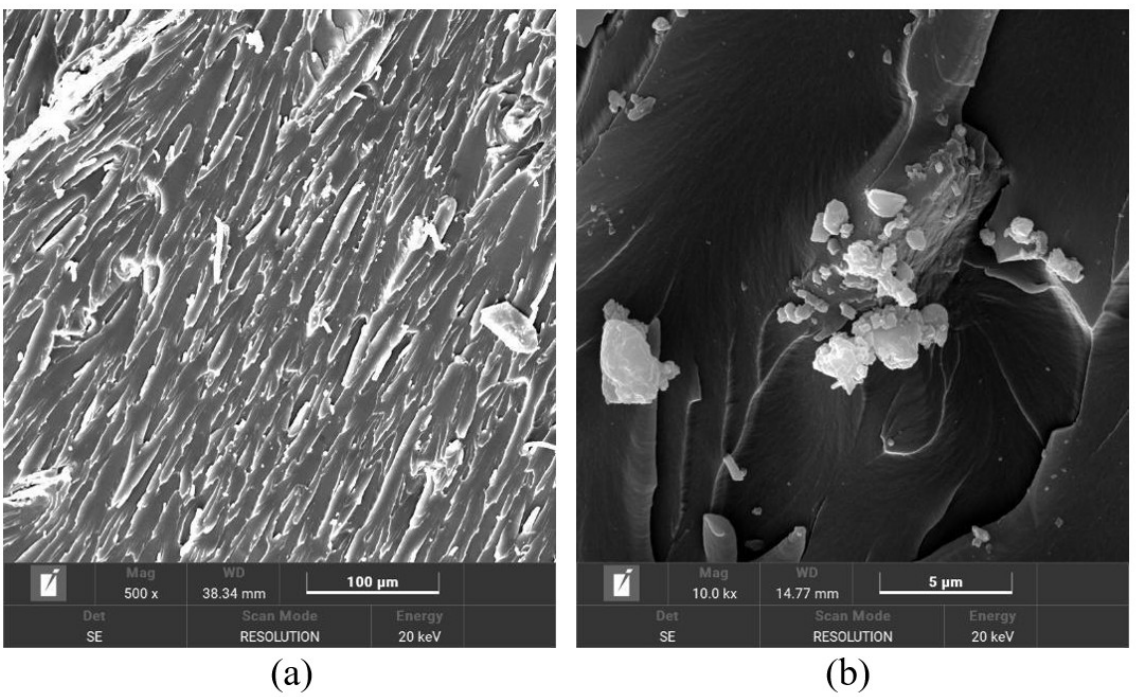

Figure 4. Fracture of the specimens of the formulation with 2.5% nanocellulose: (a) fracture surface (500x magnification); (b) nanocellulose agglomerate (10,000x magnification).

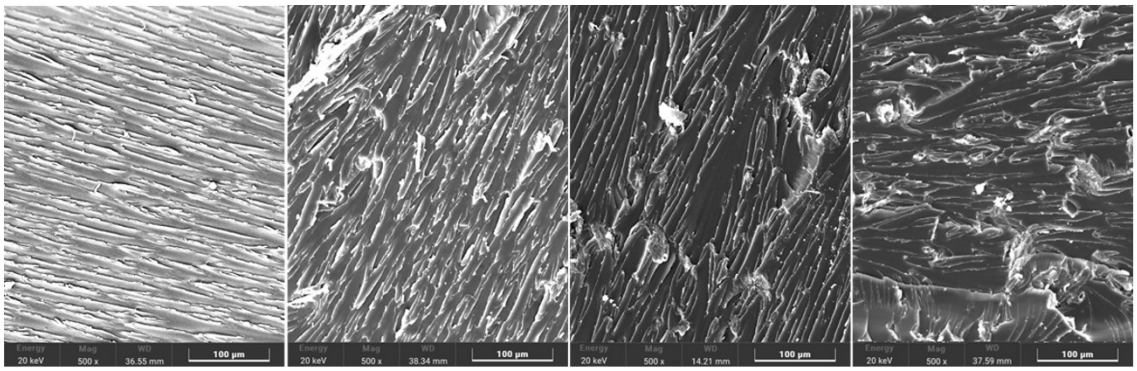

Figure 5. Fracture of the specimens: (a) without reinforcement; (b) with 1.25% nanocellulose; (c) 2.5% nanocellulose; (d) 5% nanocellulose.

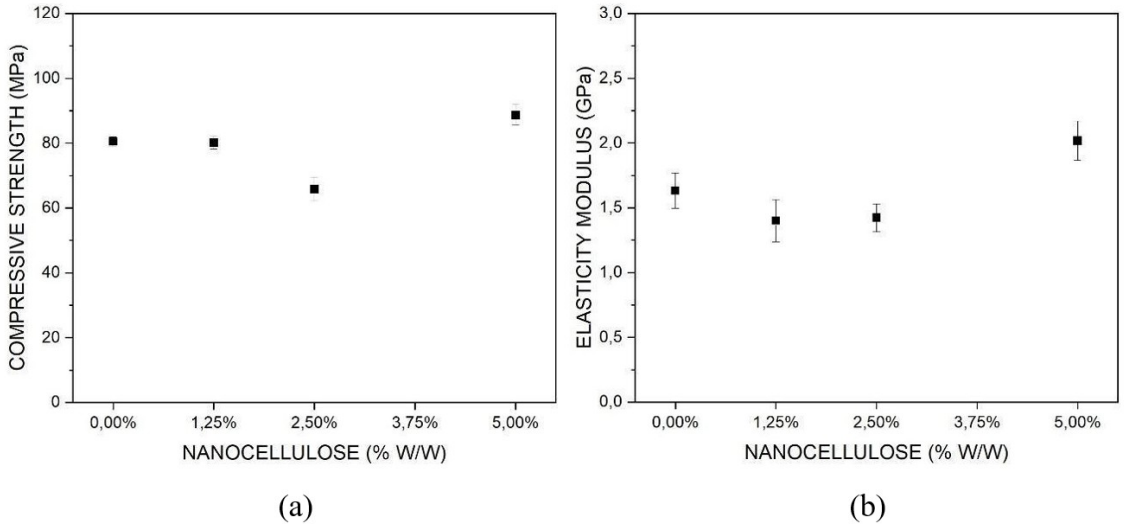

Figure 6. Results of the compression test according to the amount of nanocellulose: (a) compressive strength; (b) modulus of elasticity.

However, considering the properties of nanocellulose, a different behavior was expected, which was only observed in the formulation with the highest amount of nanocellulose (5%), where there was a significant increase in compressive strength and modulus of elasticity[53]. This is attributed to the agglomeration discussed earlier. This favors poor distribution, and this effect is mitigated by using larger quantities of nanocellulose[65]. This result is in line with the literature, which has found it difficult to obtain experimental results close to the theoretical ones due to these challenges[55].

3.3. Thermal characterization

Thermogravimetric analysis (TGA) of the nanocomposites, as shown in Figure 7, showed a change in their behavior as the percentage of nanocellulose increased. It can be seen in better detail in its derivative (DTG), that with an increase in the percentage of nanocellulose there is a shift favoring the thermal capacity of the material and the appearance of the nanocellulose degradation peak.

Initially, it can be observed that the nanocellulose shows an initial weight loss of 7%, related to surface moisture, due to its highly hygroscopic behavior[66], followed by degradation

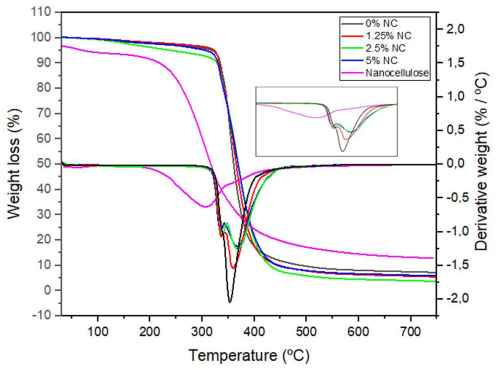

Figure 7. Thermogravimetric analysis of the compounds produced.

Table 2. Compression test results

Q. NC	R. Comp.	Standard Deviation	Value-P
0.00%	80.5	1.4	-
1.25%	80.1	2.0	69.40%
2.50%	65.8	3.7	0.00%
5.00%	88.7	3.2	0.00%

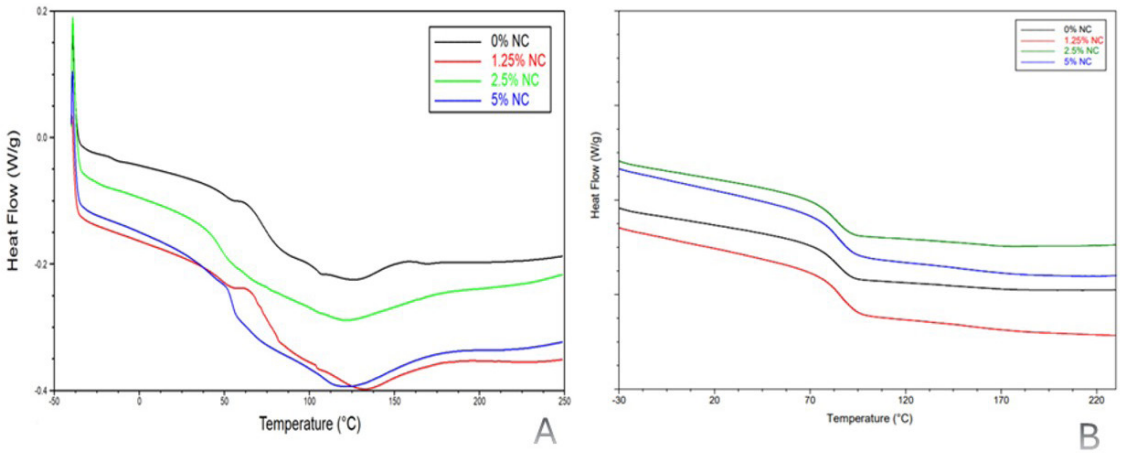

Figure 8. Differential scanning calorimetry analysis of the composites; (A) 1st run and (B) 2nd run.

Table 3. DSC temperatures of the nanocomposites.

Q. NC	T onset (ºC)	TG	T endset (ºC)
0.00%	74.80	84.20	91.39
1.25%	76.66	85.71	93.81
2.50%	73.81	83.72	90.88
5.00%	75.52	83.93	92.42

evidenced in the DTG curve with a peak at 306°C and a mass loss of 80.91%. Typically, degradation is observed in the range of 200–300°C, which may vary depending on equipment parameters or the chemical treatment used in its extraction[53].

The thermal decomposition of the epoxy resin exhibited a peak at 352°C, initially attributed to the degradation and decomposition of the resin's curing agent[67,68]. In the nanocomposites, a small initial mass loss was observed due to moisture loss, followed by degradation.

The DTG peaks for the nanocomposite formulations with 0%, 1.25%, 2.5%, and 5% nanocellulose showed maximum decomposition temperatures of 352.83°C, 362.86°C, 368.64°C, and 366.67°C, respectively, indicating a shift in temperature as the percentage of nanoscale reinforcement increases. Additionally, a new degradation peak appeared earlier, which is associated with the thermal mass loss event of the coconut nanocellulose[69].

As shown in Figure 8, the DSC curve exhibited similar behavior among the developed formulations. In the first graph, corresponding to the first heating run, a residual curing process was evident during the glass transition, with undefined peaks indicating that no significant curing-related phenomena occurred during the analysis of the material.

In the second graph, the glass transition temperatures of each composition are in the same temperature range, as shown in Table 3, with the temperature values of each phase. The percentage of nanometric reinforcement showed no significant changes in values and the resistance to thermal degradation showed a variation in relation to the percentage of reinforcement, which can be explained by the inefficiency of the dispersion.

4. Conclusions

The characterization results confirm that the chemical treatment by acid hydrolysis was successful and that the final product obtained from the coconut mesocarp fiber is nanocellulose—nanofibrils with a high degree of crystallinity, approximately 77.78%. The final yield was around 22% by weight relative to the initial dry mass, a value consistent with the literature, indicating strong potential as reinforcement for epoxy polymer matrices in open-mold processing.

A dispersion challenge was observed as the reinforcement content increased. This issue is already reported in the literature regarding the dispersion of nanoscale reinforcements. As a future suggestion, the inclusion of a dispersing agent is recommended to reduce the tendency of agglomeration.

The nanocomposites exhibited variations in their thermal behavior and an increase in strength relative to the reinforcement percentage. Further studies intend to increase the nanocellulose content to better analyze the behavior of the developed nanocomposites.

At low nanocellulose content, structural changes and increased surface roughness at the fracture surface were observed; however, there were no significant gains in material properties. In the fracture region, nanocellulose agglomeration was evident, which is directly related to inadequate dispersion of the nanoparticles within the matrix.

For the continuation of the research, suggestions involving the surface modification of nanocellulose or adjustments in the nanocomposite processing method are already being considered. The results of these improvements will later be compared to the data obtained in this study to assess potential enhancements in mechanical and thermal properties.

5. Acknowledgments

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Data Availability

The authors confirm that the data supporting the findings of this study are available within the article and are also available from the corresponding author, upon reasonable request.