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# STRUCTURAL CHARACTERISTICS AND PROPERTIES OF EPOXY MATRIX COMPOSITES REINFORCED WITH POLYPROPYLENE MICROFIBER AND GRAPHENE OXIDE (GO)

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***Abstract.*** *The development of new composites with high mechanical properties receives great attention from the automotive, aeronautical and civil construction industries, aiming at low density inputs of these materials, low cost and innovation to solve the most different problems. In this work, the mechanical behavior of the polymer composite, reinforced with graphene oxide and polypropylene microfiber, with an epoxy resin matrix, was evaluated. The characterization and effects of graphene oxide (GO) on the workability of nanocomposite properties were evaluated in conjunction with polypropylene microfiber in the epoxy-based matrix.*

***Keywords:*** *Composites, polypropylene microfibers, mechanical behavior, epoxy resin, graphene oxide (GO)*

## 1. INTRODUCTION

The market for composite materials has grown steadily over the past decade and today includes diverse applications in aerospace, biomedical, automotive, and civil infrastructure. In these and several other sectors, many professionals have felt the need to design products with these new ones.

Although the association of the term composite is linked to the so-called cutting-edge technologies, in which parts and devices derived from this material are used in components used in satellites, aircraft, helicopters, orthopedic implants, biocompatible dental implants, Formula 1 vehicles, maritime platforms, oil, bridges, telescopes, musical instruments and intelligent structures in general (Hull et al., 1996), the origin of this important class of materials goes back countless thousands of years.

In recent years, polymers reinforced with nanometer-sized materials, known as nanocomposites, the reduced particle size and greater specific surface area can provide significant improvements in the mechanical properties of coatings, as well as promoting better barrier performance against the diffusion of corrosive electrolytes. compared to conventional loads. Polymers reinforced with nanometer-sized materials such as graphene nanosheets (Figure 1), known as nanocomposites. A variety of epoxy-based composites with different particles, such as silica, clay and carbon nanotubes, have been successfully prepared and their properties have been well explored (Ribeiro and Helio, 2015).

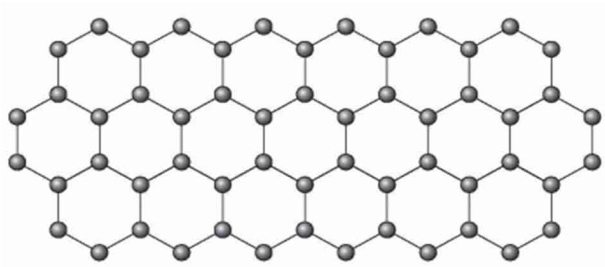

Figure 1 - Schematic representation of a graphene nanosheet (Cury and Ribeiro, 2018)

The definition of a composite material is a set of two or more different materials, combined on a macroscopic scale, to work as a unit, aiming to obtain a set of properties that none of the components individually presents (Neto et al., 2006). It is important to emphasize that as a definition of a general form of composite materials, it is very clear, however it is important to emphasize that the components appear on a macroscopic scale in the composite. This is to be able to differentiate from alloys, for example, in which different materials are combined, such as manganese in steel; however, on an atomic scale. In composite materials, the scale of inclusion dimensions is typically on the order of a micrometer or greater. Another definition is that a composite material is formed by joining two materials of different natures, resulting in a material with superior performance to that of its components taken separately.

Polymer matrix composites are divided into thermosets (epoxy, polyamide, polyester) and thermoplastics (poly-ether-ketone, poly-sulfone, polyetherimide) reinforced with fiberglass, carbon, aramid (klevar) or boron. Polymer composites for engineering applications primarily those made with epoxy resin due to low shrinkage during curing are excellent in adhesion to a variety of surfaces, good dimensional stability, low moisture absorption, good thermal and electrical properties, excellent chemical resistance and weather conditions with a high strength/weight ratio. Polymer composites have a low specific weight (Aquino et al., 2008). Making its use viable in the aerospace industry, where carbon, boron, quartz fibers present to designer's properties of improvement in structural projects, thus allowing greater in-flight performance of aircraft and reentry vehicles.

To obtain desirable characteristics of composite materials two or more materials are combined. Polypropylene fiber composite in polymer matrix is a typical example. The polypropylene fiber provides mechanical strength, while the polymer matrix is responsible for the flexibility of the composite. In fact, the matrix can be polymeric, metallic or ceramic. Another outstanding feature of composites is their versatility in terms of the broad spectrum of physical, chemical and mechanical properties that can be obtained by combining different types of matrix and by the various reinforcement options (Allen et al., 1999). The limit of combinations to obtain a composite tends to several possibilities according to the materials that can be combined.

The most used are epoxy resins (80% of all reinforced plastics), extremely versatile, epoxy resin based coatings have wide application in the industry due to their unique rigidity, dimensional stability, chemical resistance and strong adhesion to metallic substrates (Thomas et al., 2013). Epoxy resins are the most used resins in high-quality compounds, fundamentally because they have better physical and mechanical properties than polyester and vinyl ester resins. Furthermore, if we combine its good adhesion capacity on a large number of reinforcement materials, the result will be laminates with a high fiber content. However, the final characteristics of the resin will depend on the type of epoxide and the crosslinking agent (thermal resistance, hardening mode and ductility).

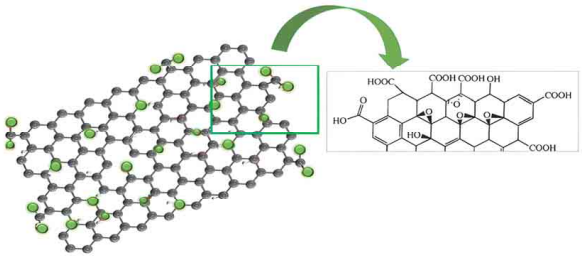


Figure 2 - Schematic structure of graphene oxide (Cury and Ribeiro, 2018)

Graphene oxide (Figure 2) is a carbon nanomaterial made up of oxygenated graphene units. Over the years, models proposed in relation to the structure of GO suggest the presence of several oxygenated functional groups in graphene nanosheets, being identified mostly in the form of hydroxyl and epoxy groups in the basal plane, smaller amounts of carbonyl and carboxylic groups, lactones, phenols and quinones on leaf edges (Cury and Ribeiro, 2018). The functional groups on the surface of graphene oxide make GO highly hydrophilic and dispersible, allowing exfoliation of monolayers in many common polar solvents, including water.

The addition of nanofillers to multiscale hybrid compounds brings with it a series of advantages and opportunities for innovation. Firstly, the presence of nanofillers makes it possible to adjust and improve the mechanical, electrical and thermal properties of the compound, providing superior performance compared to conventional compounds (Clyne et al., 2019). Furthermore, nanofillers can act as localized reinforcements, increasing the material's resistance and stress at critical points. Another important aspect is the possibility of developing composite materials with additional functionalities, such as electrical conduction capacity, resistance to high temperatures or barrier properties. This opens the way for innovative applications in various areas, such as aerospace, automotive, electronics, renewable energy, among others.

However, it is essential to highlight that the incorporation of nanofillers into multiscale hybrid composites also presents challenges. The dispersion of nanofillers in the matrix and the effective interaction between different scales are critical aspects that must be considered during the manufacturing process (Szeluga et al., 2015). Furthermore, it is necessary to carefully evaluate the cost of these additions, taking into account factors such as the availability and cost of nanometric materials. One of the most promising trends in the area of composites is the development of multiscale hybrid composites, by incorporating nanofillers, such as nanofibers, nanoparticles and nanotubes, and a micrometer-scale filler, such as fabrics or unidirectional fibers. These nanofillers have unique characteristics due to their restricted size on the nanometer scale, which confers different properties to the material composite (Anandhan et al., 2012).

The volumetric fraction of nanomaterials used in the polymer increment process depends on the strength of the interactions between the nanostructures and the polymer matrix. These interactions are strongly dependent on the polymer composition, the nature of the carbon nanostructure and also on the way the nanocomposite is prepared. The efficiency of the final product is dependent on the interaction between the polymer matrix and the nanostructure. The basic

prerequisites to guarantee a good interaction between the polymer and the nanostructure are the homogeneous dispersion of the isolated nanostructures and the establishment of a good chemical affinity (covalent or not) with the polymer matrix. The application of chemical treatments to nanostructures aims to generate increasingly stable dispersions, increasing the chemical affinity between them, generating high performance nanocomposites (Castro and Gomide, 2017).

## 2. MATERIALS AND THEORETICAL CHARACTERIZATION

Structural characterization techniques are extremely important and of great interest, as they are intended to provide mechanical, electrical, vibrational and morphological information on the materials under study. Scanning Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR) and Raman Spectroscopy analysis techniques are usually employed in the characterization of graphene, graphite oxide, carbon nanotubes, and are also employed in polymeric composites composed of it.

The diversity of processes involved in the manufacture and analysis of nanocomposites is capable of influencing their final characteristics according to the intended final application. Among some objectives related to the development of polymeric nanocomposites, there is the search for improvements in their mechanical properties, such as mechanical strength, tenacity, hardness, but also properties such as electrical and thermal conductivity, resistance to corrosion and wear, low friction, biodegradability, biocompatibility et al (Ventura, 2009).

### 2.1 Graphene oxide

Graphene oxide, commonly synthesized from the chemical oxidation of natural graphite and subsequent exfoliation of graphite oxide (GrO), has excellent mechanical and chemical properties and high functionalization capacity, in addition to having an extremely large surface area.

Considering epoxy-based composites, amine-type functional groups can stabilize graphene dispersion, due to the strong interaction generated with the polymeric matrix. Therefore, this reflects the increase in polarity and the possibility of reaction between the amino groups and the epoxy group of the resin (Castro and Gomide, 2017). All this influences the dispersion of graphene due to the emergence of stable covalent bonds between the amine, present in the structure of functionalized graphene, and the epoxy group, present in the polymeric matrix.

Graphite oxide (GrO) was prepared by the modified Hummers method (Castro and Gomide, 2017). GrO was produced via a synthesis and exfoliation process in an ultrasonic bath, using natural graphite to produce graphite oxide. Graphite was slowly added to sulfuric acid in a volumetric flask placed in an ice bath until a homogeneous mixture was obtained. Afterwards, 2.0 g of graphite, 1.75 g of NaNO3 and 80 ml of H2SO4 were added to the beaker. After inserting the three reagents, the mixture was stirred for 20 minutes until all were completely dissolved. While still stirring, KMnO4 was gradually added to the beaker. After 30 minutes, 210 ml of H2O4 and 550 ml of distilled water (cold) were slowly added to the mixture, stirring for another 15 minutes. After stirring, the beaker was removed from the box, plasticized (film) and rested for 24 hours in order to obtain the precipitation of the oxide and graphite (GO). The GrO was washed extensively with deionized water by centrifugation, the neutralized material was exfoliated with deionized water in an ultrasound bath, originating graphene oxide (GO). The mixture was centrifuged to separate the GO contained in the supernatant, which was subsequently frozen and subjected to drying.

In the characterization of graphene oxide, scanning electron microscopy was used the scanning electron microscopy (MEV) technique allows obtaining an enlarged and three-dimensional image of the sample from the interaction of an electron beam with the material, provided that it is not transparent to electrons. The beam of electrons (primary electrons) generated by thermo-ionic effect is accelerated through a potential difference and collimated through an optical-electronic column, being led to the chamber that contains the sample. Figure 3 shows the controlled deposition of graphite oxide performed layer by layer, repeatedly, which causes the films to become progressively thicker. The SEM images indicate that the preparation of graphene films by this method results in uniform films with small irregularities on its surface.

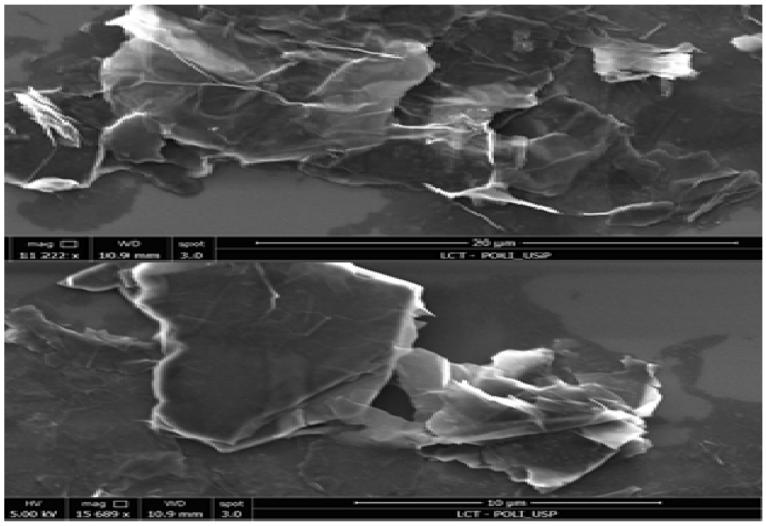

Figure 3 - Scanning electron microscopy (MEV) rGPU (Source: authors)

When this electron beam focuses on a sample point, it generates signals that are captured and amplified, providing an electrical signal that generates the image. As the beam sweeps the area under analysis, a virtual image is formed point by point.

An essential tool in the characterization of carbon-based materials is Raman spectroscopy, and this technique is sensitive to changes in the structure of these materials. The Raman analyzes were performed with the WITEC equipment, Confocal Raman Microscope Alpha300 R, using an argon 22 lasers with a wavelength of 532 nm (green). The laser was focused using a Leica optical microscope (ZEISS) coupled to the device with a total optical magnification of 500x. As a parameter for the acquisition, the region of 300 - 3200 cm-1 was used for the evaluation of the D and G bands. Measurements of punctual spectra were carried out at 5 different points and in all measurements 3 accumulations of the spectra were made with 30 to 60 seconds of acquisition time.

Raman analyzes were carried out with the WITEC equipment, Confocal Raman Microscope Alpha300 R, using an argon laser with a wavelength of 532 nm (green) from the Nano Sensors Laboratory at USP Polytechnic.

In the Raman spectra, figure 4 shows the commercial graphene and the graphene synthesized in the FGA nanotechnology laboratory. All spectra exhibit characteristic peaks in the region between 1000 to 3000 cm-1 for visible excitation energy. The bands found in the interval are known as the D bands (1200 to 1400 cm-1), the bands (1500 to 1600 cm-1) and the bands (2600 to 2700 cm-1).

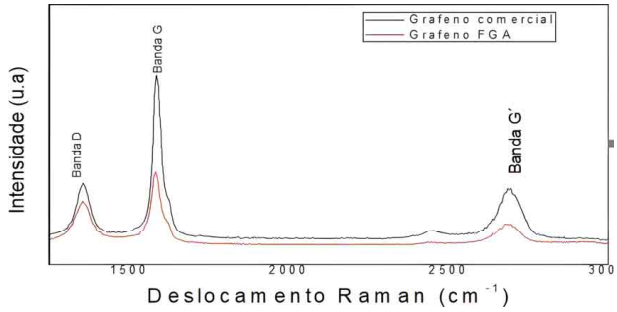


Figure 4 - Raman spectrum for commercial graphene and functionalized graphene

It is known from the literature (Wang et al., 2009) that the G band can be related to the C-C vibrations of graphite, which are present in carbonaceous materials, and is associated with defects in the graphene structure (sp2 bonds) as well as edge effects, that is, the presence of incomplete connections that can make this band more intense. On the other hand, the D band is related to the disorder of the graphite hexagonal structure. The D band is associated with poor graphitization, it is known as the “disorder” band or amorphous band, in which it is attributed to the C=C stretching modes of the graphene structure. The 2D or G' band, is found around 41 of 2700 cm-1 and is related to the structural organization in the two-dimensional plane of graphene (Wei et al., 2015).

Raman spectra shown in figure 4 shows a difference in the peak intensity and in the width of the mid-height of the D and G bands. This difference is related to the disorganization in the structure of the material, since the presence of defects significantly alters the vibrations of the spectrum Raman, thus increasing the contribution of peak D. On the other hand,

the Raman signal of the D band referring to graphene in relation to graphene oxide is more intense indicating a material structurally more disorganized.

### 2.2 Polypropylene microfiber

PP is a linear hydrocarbon polymerized from propene (or propylene) monomer, an unsaturated organic compound. It is a polymer similar to polyethylene (PE) in many properties. However, the presence of the methyl group attached to each alternative carbon atom of the main chain is the factor that differentiates it and, consequently, changes its properties in several ways.

After its polymerization, PP can form three basic chain structures, depending on the position adopted by the methyl groups: isotactic (in which the side groups are arranged on the same side of the chain), syndiotactic (groups arranged regularly on alternate sides) and atactic (in which the side groups are arranged randomly, without order). Due to the spatial distribution of the lateral group on the same side, isotactic PP (sometimes called iPP in different literatures) has a higher degree of crystallinity (up to 60%) and, consequently, higher rigidity indices and higher melting temperature.

Furthermore, the presence of the regularly positioned side group forces the crystalline phase chains to pack into a helical spatial conformation. A peculiarity of PP due to this conformation is its high index of resistance to bending fatigue, sometimes called the “hinge effect”, which makes it ideal for applications in flip-top integral hinges such as lids and glasses cases. This effect happens as long as the part is oriented (literally bent) quickly after injection so that the directional orientation of the chains occurs in the direction of movement.

The polypropylene microfiber is produced from multifilaments indicated for the reinforcement of inputs in order to generate a homogeneous compound and control cracking due to retraction or stretching and to increase the mechanical properties. Table 01 contains data on the propylene microfiber that will be used.

Table 1. Physical Properties

| | | |
|---|---|---|
| Diameter | µm | 18 |
| Section | | Circular |
| Length | mm | 6 |
| Stretching | % | 80 |
| Feedstock | | polypropylene |
| Specific weight | g/cm$^3$ | 0,91 |

### 2.3 Epoxy resin

The Epoxy Resin with Hardener system is one of the most versatile systems on the market, and can be used in the most diverse applications, from laminations with special fibers, encapsulation, adhesives, putties, liquid porcelain tiles, industrial floors, resin tables (river table) and handicrafts. in general. It has transparency and good color retention allowing mixtures of paste pigments, powder pigments and dyes. The system has excellent acceptance of fillers and additives used for other specific purposes in the most diverse applications. This epoxy system is recommended for use in solvent-free formulations that require transparency or good color retention. Systems cured with this hardener do not show an oily surface ("blushing") even when used at room temperature, whether or not in a film.

Recommended ratio is 100% Resin to 50% Hardener by MASS/WEIGHT. The stoichiometric ratio is one of the fundamental parts of a quality application. In general a smaller amount of hardener decreases the criss-cross density, therefore the physical properties of this polymer are inferior. In the same way that a much smaller amount of epoxy leads to the formation of material with a more linear structure whose physical properties will be much lower, and the consequence of this is a material that is sensitive to humidity and CO 2 (increasing the blushing index or other surface defects).

The system's base epoxy resin is a liquid product, resulting from the reaction of Epichlorohydrin with Bisphenol-A, its characteristics are in table 02.

Table 2. Characteristics of Epoxy Resin components

| | **Resin 2004** | **Hardener 3154** |
|---|---|---|
| Appearance | Colorless viscous liquid | Slightly yellowish liquid |
| Viscosity, 20°C, cPs | 600 a 900 | 200 max. |
| Specific weight, 20°C, g/cm³ | 1,12 +/- 0,01 | 1,005 +/- 0,015 |

The modified polyamine-based hardener promotes better adhesion with good thermal, chemical and mechanical resistance with low exotherm, having good solubilization in the resin with non-critical proportions, allowing homogeneous curing with good speed and very controllable final properties, depending on the properties of the system cured, presented in table 03.

Table 3. Cured system properties

| | |
|---|---|
| Thermal Deflection Temperature (HDT) (ºC) | 70 – 80 |
| Compression Force (psi) | 4000 |
| Tension Strength (psi) | 3000 – 6200 |
| Elongation (%) | 4,9 |
| Izod impact (ft lb/in) | 0,3 – 0,6 |
| Barcol hardness | Min. 10 |

## 3. EXPERIMENTAL

The polymer composite presented in table 04 consists of an epoxy matrix reinforced with 5% polypropylene microfibers and 0.5% graphene oxide (OG) analyzing the mechanical behavior and results. Bisphenol A diglycidyl ether (DGEBA) type epoxy resin will be used, properly mixed with the hardener triethylene tetramine (TETA) in stoichiometric proportions, 6mm polypropylene microfiber and processed graphene oxide.

Table 4. Sample identification and descriptions

| Conditions | Description | Simple Identification |
|---|---|---|
| I | Epoxy matrix | ME |
| II | Epoxy matrix + 5% by weight of microfibers | MEM500GL |
| III | Epoxy matrix + 0.5% by weight Graphene oxide | MEG50GL |
| IV | Epoxy matrix + 0.5% by weight of Graphene oxide + 5% by weight of microfibers | MEGM50+500GL |

The simple mixing method involves dispersing the nanostructures in polymeric solution (Figure 5), thus resulting in the formation of an intercalated structure. This method has the advantage of being simple, but often does not allow the preparation of nanocomposites with controlled composition and structure. The adopted strategy consists of pre-mixing the graphene nanostructures, polypropylene microfibers and the epoxy resin polymer and then mixing the components. In general, the simple mixing synthesis methods require the optimization of the process parameters, performing the calculation of the determined weight proportions, and control in the mixing process in order to avoid disorderly agglomeration of the components and to allow a strong interaction between the components. nanostructures, microfibers and the polymeric matrix.

The composite components, after proportion calculations, are weighed on a precision scale and unified in the mixer.

Figure 5 - Unification of materials with the polymeric matrix / Samples of MEM500GL and MEGM50+500GL (table 4)

Molds were made for the samples (Table 5) of MEM500GL and MEGM50+500GL, the dimensions of the molds followed the measurements of 260mm x 26mm x 26mm in order to extract the specimens, as determined by ASTM D3039

Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials, for mechanical analysis in the Instron 8801 equipment, and fragments for the other characterizations.

The tensile test was carried out in order to obtain the maximum tensile strength, the reference parameter to establish the fatigue test. By using the D3039/D3039M-14, standard, it is possible to obtain the axial properties of maximum tensile strength and deformation, axial modulus of elasticity and Poisson coefficient. In addition to stress vs strain curve profiles.

Table 5. Description of the specimens used

| Type | Size CxLxe (mm) | Standard |
|---|---|---|
| Pattern without hole | 250x25x2.5 | ASTM D3039 |

The tests follow the procedures described in the ASTM D 3039/D3039M-14 standard using Instron 8801 equipment and a 100 kN load cell, with controlled displacement of 2 mm/min. Two tests (Figure 6) were carried out for the specimens without notches and 4 for the OHT standard. The specimens were fixed by claws with a length of 30 mm with a hydraulic system and the pressure in the claw used was approximately 100 kgf. Load and displacement were monitored until material fracture using Instron's Bluehill software.

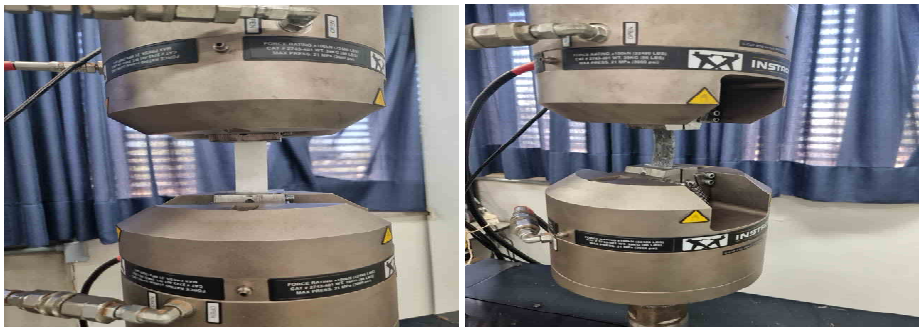

Figure 6 - Specimens of matrices of MEM500GL and MEGM50+500GL (table 4)

The investigation of the chemical composition of nanocomposites is essential for evaluating the types of chemical interactions established between the different phases and the chemical reactivity of the components, whose characteristics interfere in their final application (Wang et al., 2009). Among the most used chemical analyzes are spectroscopic techniques such as absorption spectroscopy in the infrared region with Fourier transforms (Fourier transform infrared spectroscopy – FTIR), energy dispersive spectroscopy (energy dispersive spectroscopy – EDS), visible ultraviolet (UV-Vis) absorption, X-ray diffraction, energy dispersive X-ray spectroscopy (EDX), among others.

The FTIR spectroscopy analyzes were performed using the Nicolet TM ISTM 10 FT-IR Spectrometer - Thermo Scientific equipment, from the N-TEC Laboratory (UNB-Gama). The device's accessories were used for analysis of solid and liquid samples.

Field emission scanning microscopy analyzes were carried out at the Electronic Microscopy Laboratory (LME-UNB), with the aim of morphological characterization. The equipment used in the analysis was the Field Emission Scanning Electron Microscope, brand Jeol JSM-7001F, operating at 1 kV and 9 μA. The samples went through the metallization process before analysis.

## 4. RESULTS AND DISCUSSION

The samples show similarities between the bands, observing the change in peak intensity, with emphasis on peaks with greater transmittance according to the chemical composition of the composite. The epoxy group is characterized by having three absorption bands in the region of about 1250 cm-1, 950-860 cm-1 and 865-785 cm-1 in the infrared region (Junkui, 2013). The first band located in the region close to 1250 cm-1 corresponds to the C-O bond, a second band appears around 916 cm-1, which is attributed to the asymmetric axial deformation of the ring, the bond is observed at 1600 cm-1 C=C of the benzene ring.

The strong bands at 3300 to 3400 cm-1 refer to the asymmetric folding of the O-H group of the triethylene tetramine hardener according to its chemical chain. At 700 to 850 cm-1, the band is medium and can be associated with stretching of the C–H bonds with rocking-type angular deformation. Finally, weak bands at 600 to 700 cm-1, associated with angular deformations of the wagging type for the C-H group, the presence of the hardener with the term “E” outside the parenthesis of the chemical structure (Figure 7) stands out, and the other chemical structures end up overlapping each other polypropylene (Zhao et al., 2008).

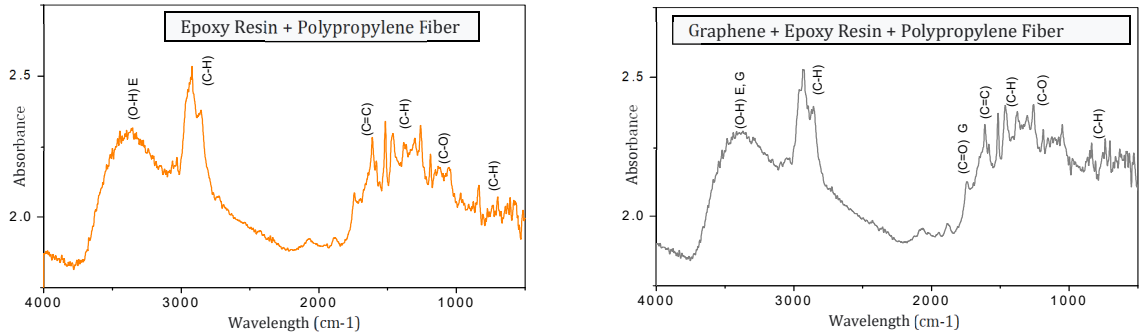


Figure 7 - Infrared spectrum for the epoxy matrix + 5% by weight of microfibers / Infrared spectrum for the epoxy matrix + 0.5% by weight of graphene oxide + 5% by weight of microfibers

In the composite with the polymeric matrix in the epoxy resin with + 0.5% by weight of graphene oxide + 5% by weight of microfibers, the infrared spectrum was analyzed and it was verified that the intensity of the band referring to the absorption of moisture was weaker in 3600 to 3700 cm-1 and no band appeared around 1640 cm-1 (Figure 7). Therefore, bands 3300 to 3400 cm-1 can be associated with the O-H group characteristic of the triethylene tetramine hardener, but also correlated to graphene, 2800 to 2900 cm-1 to axial stretching for the C-H group and to C–H bonds. Bands that are very weak at 1300 to 1400 cm-1 may be related to angular deformation of the C-O group and asymmetric stretching. Finally, the intensities at 700 to 800 cm-1 are related to the axial deformations of the existing C–H connections at the end of the plane.

It is noted that the intensity of this band for the epoxy resin spectrum is intense, it is also observed that the addition of reinforcements caused a slight increase in the intensity of this band, which may indicate that the reinforcements hinder the reaction of the epoxy groups. Other important bands in the interpretation of epoxy resin spectra are at 1200 to 1300 cm-1, attributed to the symmetric axial deformation in phase of the C–O and C–H bonds of epoxy rings, in which these bonds stretch and contract in phase, and at 700 to 800 cm-1, attributed to the asymmetric axial deformation of the epoxide ring, and these are linked to the other chemical structures of this composite.

In order to obtain the shear modulus in XY, the Iosipescu test was performed, and the response of the material under shear load in the transverse plane to the laminate is illustrated in Figure 8.

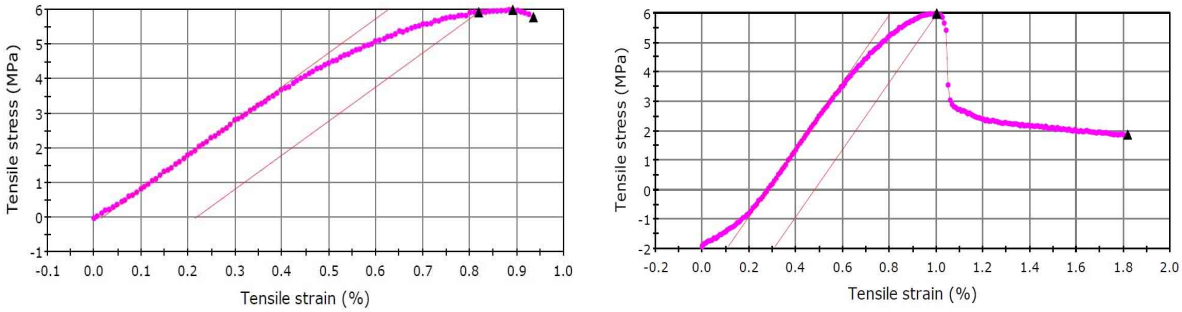


Figure 8 - Stress curves of shear tests in the transverse plane - Epoxy matrix + 5% by weight of microfibers

The data obtained in the graph (Figure 8) are the results of the application of load in the fatigue tests, caused by ruptures in the useful area and presenting values consistent with those received in the test specimens. In addition, the maximum load values reached in the fatigue tests were comparable to the values obtained in the specimens with resistance limit.

In the two specimens of epoxy matrix + 5% by weight of microfibers shown in the graph, it was possible to observe an abrupt rupture in one of the samples, suggesting the occurrence of some type of defect, such as the formation of bubbles in the specimen. This phenomenon may have contributed to the premature failure of the material during the translation test. The presence of bubbles or other internal imperfections in materials can significantly affect their resistance and mechanical behavior. These defects function as stress concentration points, given the material's ability to withstand the applied load. As a result, the sample may fail at a lower stress level than expected for a defect-free material.

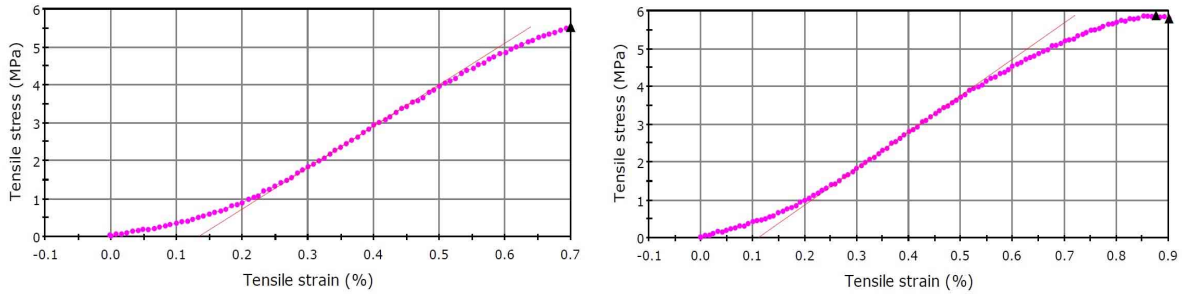


Figure 9 - Stress curves of shear tests in the transverse plane - Epoxy matrix + 0.5% by weight of Graphene oxide + 5% by weight of microfibers

In the two specimens of epoxy matrix + 5% by weight of microfibers presented in the graph illustrated in Figure 9, it was possible to observe an abrupt rupture in one of the samples, suggesting the occurrence of some type of defect, such as the formation of bubbles in the sample. This phenomenon may have contributed to the premature failure of the material during the translation test. The presence of bubbles or other internal imperfections in materials can significantly affect their resistance and mechanical behavior. These defects act as stress concentration points, given the material's ability to withstand the applied load. As a result, the sample may fail at a lower stress level than expected for a defect-free material.

In the SEM analysis (Figure 10), detailed observation of this material revealed adhesion characteristics of the polypropylene fiber, as well as disintegration in specific areas, mainly on the surface of the composite. It was evidenced that the polypropylene microfibers incorporated into the epoxy resin matrix had a good interaction between the fibers and the matrix, suggesting good adhesion. However, the analysis also identified areas where disaggregation of the composite occurred. These areas showed characteristics of separation and failures at the epoxy resin matrix interface.

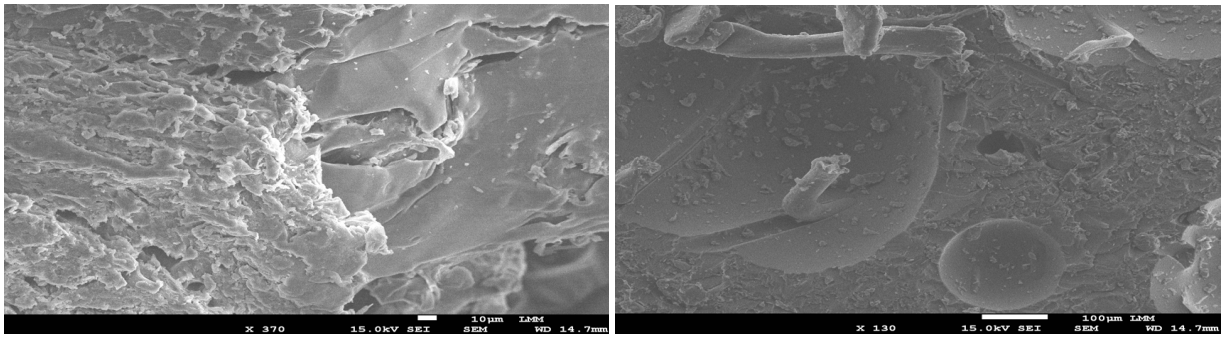

Figure 10 - Scanning electron microscopy (SEM) - Epoxy matrix + 5% by weight of microfibers

The inclusion of graphene oxide in the matrix resulted in fewer fractures and greater cohesion, which can be observed in Figure 11, demonstrating interaction between the polypropylene microfibers and the epoxy resin matrix, characteristics of an intercalated structure. The analysis also identified areas of roughness in the composite, similar to the previous version of the material.

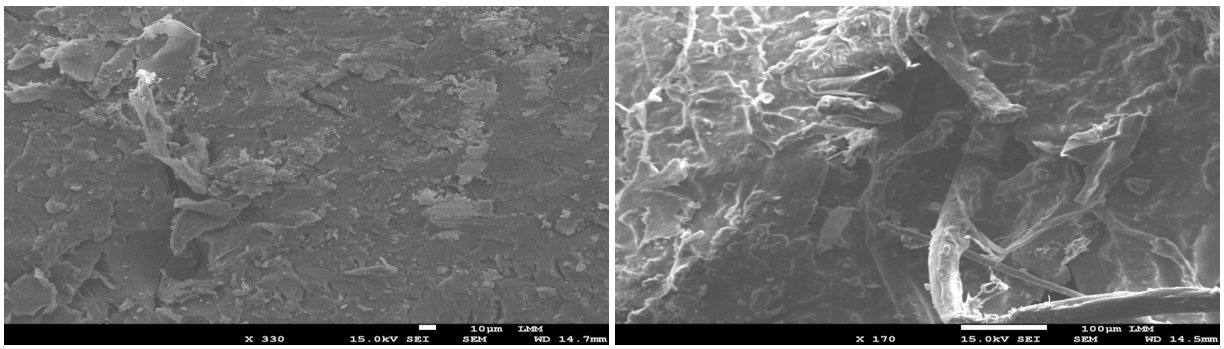

Figure 11 - Scanning electron microscopy (SEM) - Epoxy matrix + 0.5% by weight of Graphene oxide + 5% by weight of microfibers

## 5. CONCLUSION

In the analysis of the nanocomposite with epoxy matrix reinforced with 5% polypropylene microfibers and 0.5% graphene oxide (GO), in comparison with the other spectra, shifts of the spectral bands referring to the carbonyl groups were verified (-C Epoxy matrix + 0 .5 wt% graphene oxide + 5 wt% microfibers =O), and a small shift of the ether band (-O-C-O) for lower wavenumbers. These changes are evidence of the occurrence of strong intermolecular interactions between graphene and epoxy and they verified that this led to the shortening of carbon bonds, favoring interactions between polymeric chains, thus inducing greater crosslinking of the material.

Comparing the infrared spectra, it is not possible to observe large changes in the FTIR spectra of some samples, and this may be due to a low percentage of graphene oxide nanofillers. The interaction of the analytes with the nanocomposite generated changes in the optical properties of the material observed in scanning electron microscopy. From the analyzes described, it was possible to obtain information about graphene regarding its crystalline structure. FTIR analysis indicated expected functional groups, such as hydroxyl groups and carbon bonds, confirming graphene oxide.

Regarding the axial tensile tests carried out, results presented in the samples of epoxy matrix + 5% by weight of microfibers and epoxy matrix + 0.5% by weight of graphene oxide + 5% by weight of microfibers, the presence of of little delamination and cracking suggests that the material showed good adhesion and cohesion between the union of materials in the polymer composite, demonstrated during the tensile test, which is tolerated by the mechanical resistance of the material in engineering applications. On the other hand, the concentration of the fracture area in areas that did not show standardization may be an indication that the presence of bubbles or lack of homogeneity generates discontinuities in the material and may have been a contributing factor to the fracture without central standardization, and according to scanning electron microscopy analyses, these discontinuities act as stress concentration points, making the material more susceptible to failures in these regions.

Understanding these fracture changes and segregations is crucial in the context of material integrity and therefore the composite material in question. The ability to analyze the dynamics between the components of the composite makes it possible to make adjustments to manufacturing procedures. These adjustments aim to ensure the integrity and homogeneity of the material, directing it to the highest level of performance and, therefore, enabling consistent practical applications.

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## 7. RESPONSIBILITY NOTICE

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