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RHEOLOGICAL BEHAVIOR OF GRAPHENE OXIDE SUSPENSIONS IN POLY(ETHYELENE GLYCOL)

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Abstract. *The main objective of this work is to characterize the rheological behavior of the suspensions of graphene oxide (GO) dispersed in poly(ethylene) glycol (PEG), through the rotational rheometer MCR501 and MCR301. Graphene oxide was obtained through Hummers' method and characterized by various techniques. This method is based on the chemical exfoliation of graphite through strong acids such as sulfuric acid, thus generating the graphite oxide (GrO), which is exfoliated in an ultrasonic bath in PEG for the formation of the suspension with graphene oxide. The rheological characterization is fulfilled through measurements in steady shear flow, transient shear flow and oscillatory shear flow. The characterization of GO was performed using techniques such as: X-Ray Diffraction (XRD), Raman Spectroscopy, Fourier Transform Infrared (FTIR), Atomic Force Microscopy (AFM) and Thermogravimetric Analysis (TGA). The results will serve as a basis for understanding the interaction between the GO and the polymer, and the rheological behavior of GO-based polymer nanofluids.*

Keywords: *polyethylene glycol, graphene oxide, characterization, rheological behavior.*

1. INTRODUCTION

Nanotechnology marked the evolution of a new scientific area in recent years by the simple fact that the properties of the materials also depend on its shape and size and not only on its composition and structure. Such technology can be applied in several scientific areas such as physics, mathematics, medicine, communication, among many other areas where it has great prominence (Bordoni, 2014).

Nanometric particles, when compared to the same materials at larger scales, have unique physical and chemical properties and the manufacture of these materials has been allowed due to new technologies that have emerged (Yu et al., 2007).

Among the variety of the most researched materials between academic papers and industries, we have polymer solutions reinforced with nanometric scale loads, with emphasis on carbon-based materials. The interest behind studying these materials also called nanofluids is due to the fact they tend to have better properties when compared with conventional suspensions. The properties of the material are reinforced, since they have a higher interaction between matrix/reinforcement when it comes to nanometric scale (Botan, 2011).

According to Witharama (2003), nanofluid is defined as a suspension of nanometer-sized particles (from 1 to 100 nm) in a base-fluid and this concept emerges from the field of nanotechnology. Several materials such as ceramic oxides, metal oxides (CuO, ZnO, TiO_2, SiO_2), metals (Cu, Ag, Au, Fe), carbon nanotubes (single and multiple wall) and graphene are used for the production of nanofluids (Oliveira, 2014).

Dynamic viscosity, thermal conductivity and specific heat are considered the main thermophysical properties of nanofluids and their determination depends not only on the base fluid used, but also on the particles that make up this fluid. And some of the factors that may interfere on these properties are volumetric concentration, particle size, morphology (size, shape, fractal surface), additives employed, production form, pH, among others (Noble, 2017).

In 2004, two Russian researchers discovered graphene through a simple process of exfoliation: using a duct tape, they wore a graphite plate out until obtain the graphene. Graphene is a flat monolayer composed of carbon atoms with sp^2 hybridization linked together, forming a two-dimensional network. Besides being an extremely resistant material, flexible, impermeable and with termical conductivity a thousand times greater than the one of copper, according to Vianna (2017), the material also presents high mobility of loads, optical transparency, vast superficial area and flexibility, combined with an enormous mechanical robustness and environmental stability. Figure 1 shows several crystalline structures composed of carbon atoms with sp^2 hybridization, which are: a) Graphite; b) diamond; c) fullerene; d) single-walled carbon nanotube; e) multiple wall carbon nanotube; f) graphene.

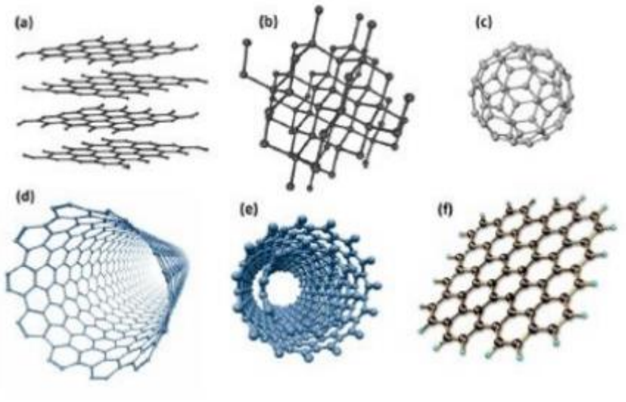

Figure 1.1- Carbon-based structures. (Zarbin and Oliveira, 2013)

Although graphene has excellent properties and many industrial applications, there is a big technological challenge to obtain it in large amounts, and with a low cost of production. There is a great deal of interest in developing preparation routes that are practically feasible with high yield and purity and good structural quality (Mehl et al., 2014).

The most promising approach to produce graphene on a large scale in ourdays, it is to from graphite through chemical methods. This can result in the rapid development of new technologies and subsequent introduction into the market (Chua, 2014; Castrillón, 2015).

From the 21st century, researchers and scientists have begun to carry out measurements and tests with graphene suspensions and until nowadays few studies have been carried out on the rheological properties of these suspensions, although there are many articles that deal with the thermal and electrical properties of these nanofluids (Giudice and Shen, 2017).

Numerous operations in the industries use complex fluids with non-Newtonian behavior. Non-Newtonian fluids are important for technological advancement in many sectors and therefore should be studied and understood so that their applicability in the market increases. Regarding the rheological characterization, the complete study of the mechanical behavior and the nature of each of these fluids, as in the present case is the suspension of polyethylene glycol with graphene oxide, it is necessary in order to determine its applications in certain procedures and to be able to evaluate and improve their properties, thus optimizing the processes where they are inserted.

Because of their excellent electrical, mechanical properties and thermal stability, many researchers have been interested in studying graphene/polymer nanocomposites, although they face some challenges during their processing such as the homogeneous dispersion of graphene in the polymer matrix (Shu et al., 2016). Up to now, studies have been conducted on the rheological behavior of graphene/polymer nanocomposites focusing on graphene processed from graphene oxide (GO) dispersed in some common solvents such as water. However, there are few reports that aimed at the study of obtaining graphene oxide dispersed directly in a liquid polymeric matrix, as will be the focus of this work.

In the area of nanotechnology, the determination of the polymer in which GO will be applied after obtaining the charge of nanofluid is quite important. The present work studies the incorporation of GO in polyethylene glycol with molecular weight (M_w) of 400g / mol (PEG 400). PEG, in generally, is a water-soluble nonionic polymer, but PEG 400 is a liquid polymer and which is used as a kind of model polymer that has facility to interact with nanoparticles and it would assist in the study of GO with different oxidation levels. Futhermore, although not the main reason for this work, this polymer is very important for the pharmaceutical industry, especially nowadays, for the newer controlled drug release systems. It has also been widely used in several sectors, among them, the cosmetic and the dental area by virtue of its fantastic chemical properties.

The amount of oxygenated functional groups that are inserted into the graphite structure is what determines the oxidation level of graphene oxide. Some factors such as the oxidation method, the graphite purity, the oxidizing agent used and the amount added, the reaction time are able to influence the degree of oxidation during GO synthesis. The degree of oxidation influences its morphology and GO properties. The size of graphene oxide sheets is related to the amount of C-O groups inserted in the structure, and the size of the GO is fundamental to determine its applications (Ferreira and Silva, 2018).

In the present study, the influence of different oxidation levels of GO on a model polymer (PEG 400) and the effects of GO concentrations were investigated, because it is necessary to investigate the rheological behavior of GO suspensions in some polymer once the polymeric matrix has a rheological response dependent on the particle-particle interactions with the polymer chain., so that it is possible to understand the effect in flow processes, that is, during industrial processes.

2. EXPERIMENTAL

2.1 Materials

Table 1 show all products and reagents used, such as the supplier company for the graphene oxide production:

Table 1. Products, reagents and the supplier company for the graphene oxide production.

Products/reagents	Supplier company
Graphite powder <45µm, ≥99.99% trace metal basis	Sigma-Aldrich
H2SO4 (99%)	Sigma-Aldrich
KMnO4	Sigma-Aldrich
Poly(ethylene glycol) M_w 400g/mol	Sigma-Aldrich
C_2H_5OH	Synth
HCl (ACS reagent, 37%)	Synth
H_2O_2 (30% m/m)	Synth
Deionized water	PUC

2.2 Sample preparation method

Graphite oxide was obtained by modified hummers' method and used in samples preparation of different concentrations. This method consists in the addition of 1g of graphite and 60mL of sulfuric acid (H_2SO_4) in a 500mL round bottom flask. This reaction is performed under low temperature (emerged in an ice bath) and under agitation at a frequency of 600 Hz (a medium goldfish is placed inside the flask and through a magnetic stirrer provides such agitation) for 15 minutes. Thereafter, 3.5mg of potassium permanganate ($KMnO_4$) is added slowly for 15 minutes. It is important to mention that the addition of permanganate must be done slowly due to the fact that an extremely exothermic reaction occurs and with a very high kinetic velocity. These reagents are added with the intention of expanding the structure and oxidizing the surface of the graphite sheet due to the presence of oxygenated functional groups. At the end, the system was removed from the ice bath and allowed to stir under the desired oxidation time. For this work the oxidation times used were 2h and 96h.

After the oxidation step, the system returned to the ice bath and 200mL of deionized water was slowly added to stop the reaction. At the end of the dilution, a portion of aqueous solution of hydrogen peroxide (H_2O_2) was added until it stopped bubbling because the reagent neutralized the ions presents in the solution. At the end of the synthesis, the flask was removed from the ice bath and allowed to stand for 12 hours to allow the decantation to occur.

The generated product was filtered with a vacuum pump to speed up the filtration process and washed in the following order: 500 mL of deionized water, 250 mL of a 10% aqueous hydrochloric acid solution, 500 mL of ethanol and finally 250mL of deionized water again. The deionized water was responsible for the elimination of the salts, the hydrochloric acid for the removal of the metal ions and the ethanol for the removal of the organic residues.

The obtained product was placed in the oven at 60 ° C to dry for a period of 12 hours. After being removed from the greenhouse, the material was macerated and dispersed in the polyethylene glycol with the intention of preparing suspensions with concentrations: 0.1, 1, 10, 20, 40 and 80mg / mL. These colloidal suspensions of graphite oxide were exfoliated in an ultrasonic bath at 40 ° C for a period of 4 hours, and then due to the exfoliation, graphene oxide (GO) was obtained.

2.3 Rheological measumerements

Before starting any measurement, the suspensions were kept for constant stirring on a magnetic stirrer at a frequency of 1000Hz for 15 minutes for homogeneity of the samples. The rheological tests of these suspensions of different concentrations were performed on a Physica MCR501 rheometer using the cone and plate geometry with a diameter of 60mm and with a gap of 0.057 mm. The other geometry also used to perform some tests despite being in another rheometer (Physica MCR301) to obtain comparative data was the double-gap. Both are stress-controlled rheometer. A protective cap is used above the system to avoid evaporation of the solvent. Each test was repeated at least three times to confirm its reproducibility.

Before starting the measurements, the samples were allowed to achieve the temperature equilibrium of 20°C for 5 minutes. Then, the rotational test is performed in order to obtain the flow curves of the suspension, that is, the evaluation of the behavior of the shear stress and the viscosity as a function of the shear rate, in steady shear flow. These properties were monitored by ranging the shear rate from 1000 s^{-1} to $0.1s^{-1}$.

In these measurements, the flow curve was evaluated decreasing the shear rates from the highest to the lowest, because in this way the time to reach the steady state is lower, since the sample undergoes a pre-shear. Tests were also performed starting from the lowest to the highest rates in order to verify the hysteresis of the curves, but for these tests it is necessary to make the pre-shear setting the shear rate.

Regarding the oscillatory tests, it was performed the dynamic strain sweep at a fixed frequency of 1Hz ranging the strain from 10% to 10,000%. This result shows the variation of the storage modulus (G ') and the lost modulus (G '') as a function of the strain in order to define the linear viscoelasticity region of the suspension, where the modulus (G 'and G '') are independent of the amplitude of the deformation. It is also possible to evaluate the fluid behavior in this region, by verifying the predominance of viscous or elastic effects.

Then, for the dynamic frequency sweep, a fixed shear stress value obtained from the linear viscoelasticity region (LVR) was applied, and the frequency was varied from 0.1 Hz to 100 Hz, to investigate the structure of the suspensions.

2.4 Characterization

The Raman spectrum of graphene oxide were obtained by the Witec Alpha 300R Raman Spectrometer. The equipment was calibrated using a silicon waffer (Si), with a grating refraction index of 600 g/mm (grades per millimeters), integration time of 0.25 seconds, 50x objective lens and 532nm laser. The sample was prepared from the dilution of 1 mg GO in 1mL of deionized H_2O, dropping one drop of the solution onto silicon oxide substrate.

In order to determine, by AFM, the sheets dimensions and thickness of the GO flakes obtained, the Bruker Model Icon Dimension Microscope with probe with tip covered with Si was used. A resolution of 512 lines with 512 points was used in the areas of each image, captured with Scan Asyst mode. The samples were prepared from the dilution of 1mg GO in 1mL of deionized H_2O, and from this solution a further 1: 200 dilution with a total volume of 1mL was prepared.

The diffraction measurements by X-Ray Diffraction were performed on the Rigaku MiniFlex II diffractometer, varying the scanning at angles of 5^o to 50^o with a rate of 2^o per minute using λCuKα and monochromator radiation and fixed slots with 30 kV, 15 mA, with the samples powdered at room temperature. From the Bragg Law, $2d\sin\theta=\lambda$, where λ is the wavelength of incident radiation, d is the distance between atomic planes and θ is the angle of incidence in relation to the plane considered it was possible to calculate the interlamellar distances.

The equipment used for the thermogravimetric analysis of graphite oxide powder was the SDT-Q600 of TA Instrument. The temperature range used in the analysis was 25^oC to 1000^oC, with a variation rate of 10^oC per minute and in a synthetic N_2 air environment.

The absorption spectrum was obtained from dilute solutions of graphene oxides prepared with a concentration of 1 mg / mL in deionized H_2O and pH> 5. The solution was dripped on a silicon oxide (SiO_2) wafer. The equipment used to obtain these results was the Bruker Vertex 70 Fourier Transform Infrared Spectrometer and it was recorded in the wavenumber range of 750-3500cm^{-1}.

The software used for almost all characterization was OriginPro 8.5, except for AFM that used for image/histogram processing, the Gwyddion.

3. RESULTS AND DISCUSSION

3.1 Structure characterization of graphene oxide

In the Raman spectrum, according to figure 3, characteristic peaks such as the D-band between 1300 and 1500cm^{-1}, which corresponds to the edge defects and the defects in the graphite layers that arise during the oxidation due to the addition of functional groups. This growth is due to the emergence of incomplete bonds, heteroatoms attached to the sheets structure and change of the sp^2 to sp^3 bond type in some carbon atoms. The small peak in the D-band of graphite facilitates the oxidation process by the modified Hummers method.

It is also possible to observe the predominance of the G band between 1500-1750cm^{-1} in the graphite spectrum, which is related to the vibrations created by the elongation of the sp^2 bonds between the carbons and which means that its crystalline structure presents a small amount of structural disorder. With the oxidation process, there was a widening of the G band indicating a greater disorder in the graphical structure and a greater heterogeneity.

The 2D band between 2500-2750cm^{-1} is related to the two-dimensional structure of the material and provides information on the existence of multilayers in the sample appearing only in the structure of the graphite.

The I_D/I_G intensity ratio of the D and G bands allows an estimation of the structure disorder or defects. For the GO with 96hrs of oxidation, I_D/I_G = 1.109. Comparing with the GO with 2 hours of oxidation, which has an I_D/I_G = 1.066, it can be concluded that the graphene oxide produced with a greater number of hours of oxidation had more defects introduced in its structure, defects produced by the introduction of a greater number of functional groups in the structure. As the oxidation time increased, there was an increase in the D-band peak and a decrease in the 2D-band peak, indicating that there was damage in the carbonaceous structure and the disintegration of the graphene layers composing the graphite.

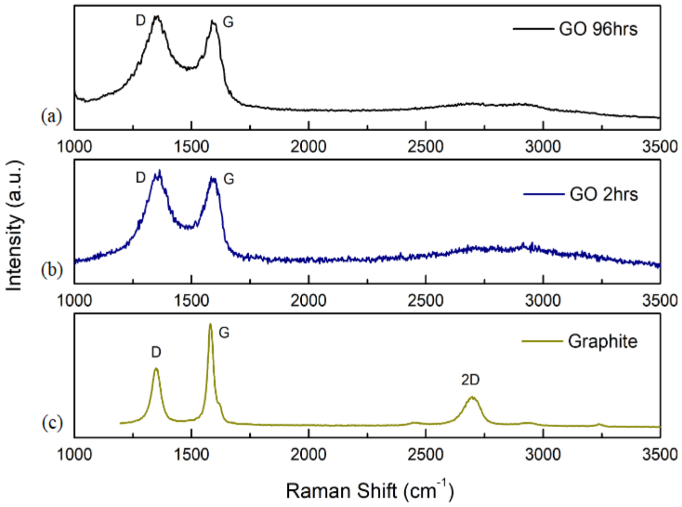

Figure 3. Raman spectrum of (a) GO with 96h, (b) 2h of oxidation and (c) graphite.

It is possible to observe in figure 4 that the two reactions had a great difference in the crystallographic distance of the material. According to Bragg's Law, the material with 96 hours of oxidation obtained a greater spacing between the basal planes of 9.066 angstroms (Å), than in the material with 2 hours of oxidation and the graphite. The graphite showed a peak at the 2θ of 26.2° relative to the plane, which indicates a distance between layers of 3.12 angstroms. In the GO of 2h, the peak occurred at 2θ of 9.9°, which shows that the spacing between layers became 8.935 angstroms. The possible cause of the greater distance in the GO of 96hrs is the availability of time that the material had for the entrance of more functional groups in its structure, besides the water adsorbed during the process.

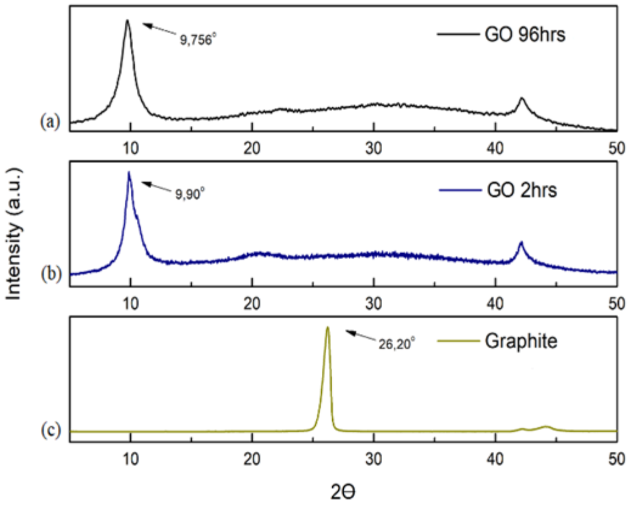

Figure 4. XRD interference planes for (a) GO 96hrs (b) GO 2hrs and (c) Graphite

The thermogravimetric curve represents the loss of mass with increasing temperature. According to figure 5, in the structure of the graphite it is possible to observe the beginning of the loss of mass of the graphite crystals around 808°C, while for the sample of GO with 2hrs of oxidation, first it is possible to perceive an initial mass loss due to the loss of residual moisture after the oxidation process, and then a significant mass loss occurs between 100 and 300°C, where 66.11% of the GO mass evaporates. From approximately 140°C up to around 200°C, the 96hrs GO has nearly 20% reduction in its mass resulting from the loss of residual moisture from the exfoliation process. From 200 to 600°C the GO obtained with 96 hours of oxidation, has the reduction of mass of 62% due to the loss of oxides like CO_2 present in the structure of the material. These values indicate that the system with higher oxidation time obtained a greater amount of functional groups introduced in the graphite structure, so the GO will be more easily dispersed in aqueous suspensions.

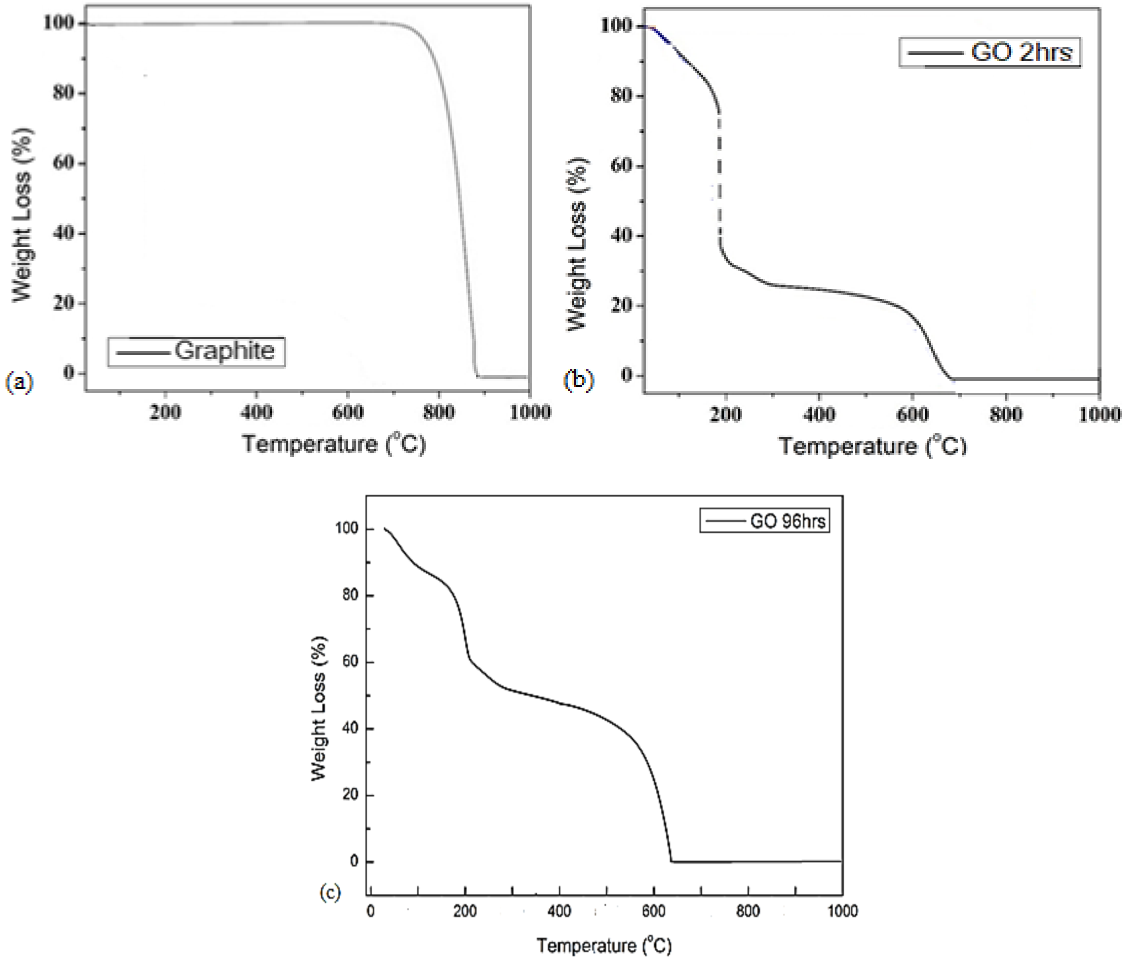

Figure 5. Thermogravimetric curves for (a) graphite (b) GO 2 hrs and (c) GO 96 hrs oxidized

Infrared Vibrational Spectroscopy (FTIR) is based on absorption spectroscopy that identifies a compound through the infrared region, and investigates the composition of a sample or provides evidence of the presence of functional groups. To obtain the measurements of a sample, the amount of energy transmitted by a beam of infrared radiation passing through the sample is recorded. A molecule passes into a state of excited energy when it absorbs radiation and this absorption occurs when the radiation has the same frequency as the vibration of the bond, thus increasing the amplitude of the vibration

Figure 6 shows the FTIR spectrum of the oxidized GO 96 hour. It can be observed a strong absorption between 3400 and 2400 cm^{-1}, which normally overlaps the CH stretch, because of the presence of the OH group. Analyzing an absorption peak at approximately 1710 cm^{-1} for C = O stretching vibrations, it is possible to detect the presence of functional groups such as carboxylic acid, ether, ester (these three groups can be estimated when taken into account also the presence of absorption at 1093 cm^{-1}), ketone and aldehyde (a weak peak is observed at 2900 ~ 2800 cm^{-1}). The absorption with media intensity indicated between 1418 ~ 1352 cm $^{-1}$ takes into account the torsion vibration of carbon-hydrogen bonds in the GO structure. The FTIR spectrum for the graphite and the GO of 2 hours of oxidation were not performed.

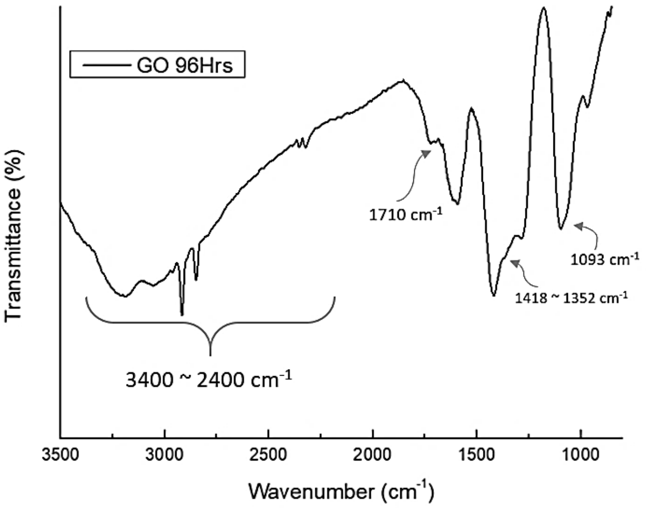

Figura 6. FTIR spectrum of GO with 96 hrs of oxidation.

The graphs shown in figure 7 show the sheets are still overlapping having several different thicknesses and that the oxidation level influenced the particle size, since the GO of 2hrs has particles ranging their size from 5 to 145 nanometers and the GO of 96hrs had particles with size from 0 to 60 nanometers, most of which are concentrated in the 0-40 nanometer range and other points exceeding 60 nm do not belong to the sample and may be external particles present at the time of the test. The AFM for the grafite was not performed because the particle is larger than 3 micrometres (has 45 micrometres) and is considered too large to be used in AFM.

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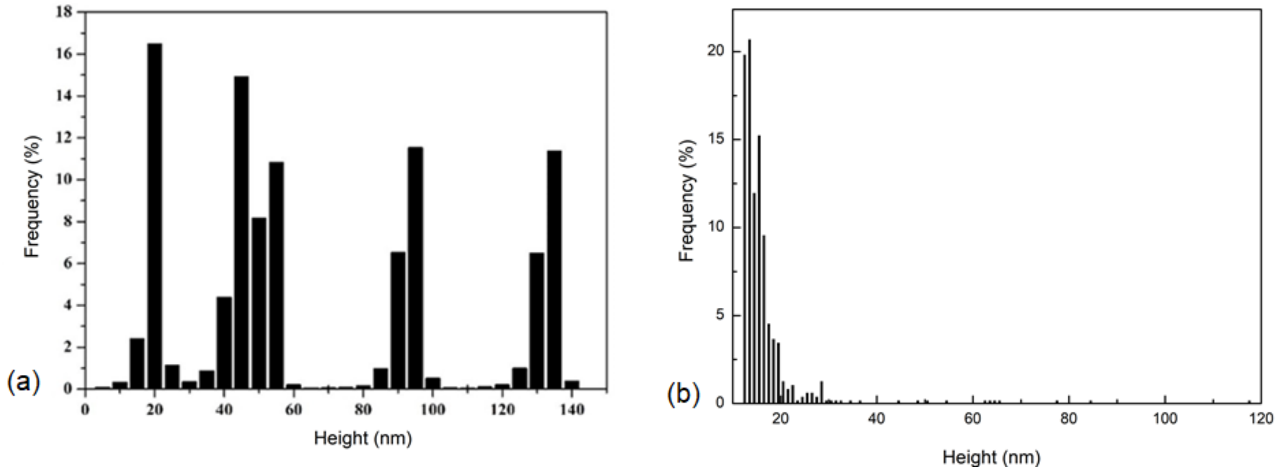

Figure 7. Frequency (%) of GO sheets of (a) 2 hrs of oxidation (b) 96hrs of oxidation as a function of their thickness.

3.2 Rheological effects of graphene oxide suspensions in poly(ethyelene glycol)

The tests performed on a steady state shear flow were used to investigate the flow properties of the material by recording the shear stress and viscosity at increasing shear rates. The GO tests with 2h of oxidation were performed with the cone-plate geometry and the GO tests with 96h of oxidation with the double-gap, with the intention of observing the behavior in a higher range of shear rate. Points not shown in the graphs showed lower torque values than the minimum reliable torque in the respective geometry. In Figure 8 it is possible to note that for both oxidation times there is an increase of the viscosity as the concentration is increased, the viscosity for the same concentration is higher in the GO suspension with 96h of oxidation than that of 2h. In the flow curve of the GO of 96h the change of the Newtonian behavior to a pseudoplastic behavior begins to be observed at suspensions with concentration of 40mg/ml, whereas in the tests made for concentrations of 0.1 to 40 mg/ml of GO of 2h all samples presented a Newtonian behavior in the shear rate range analyzed. This increased viscosity observed with increasing oxidation time can be explained by the fact that the longer the oxidation time, the smaller the particles and more oxygen groups are inserted into the graphite structure. It results in a greater interaction between the nanoparticles and the polymer, forming larger aggregates that lead to an increase in viscosity, which explains the so-called Bridging effect. Figure 9 shows the Bridging effect.

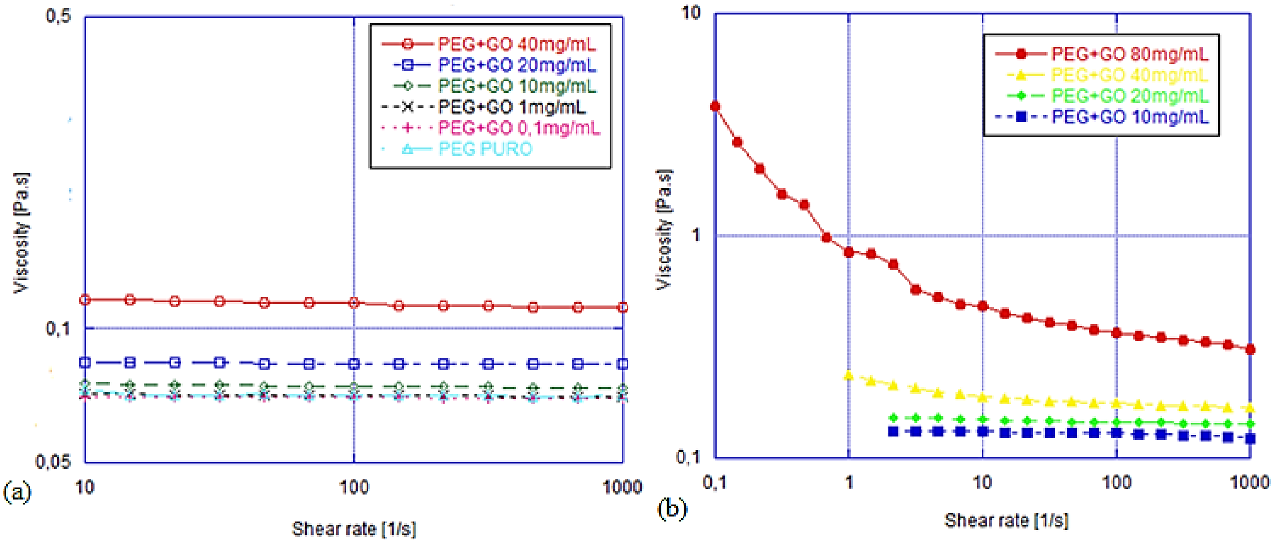

Figure 8. Steady shear rate flow behavior of the (a) GO of 2hrs (b) GO of 96hrs of oxidation

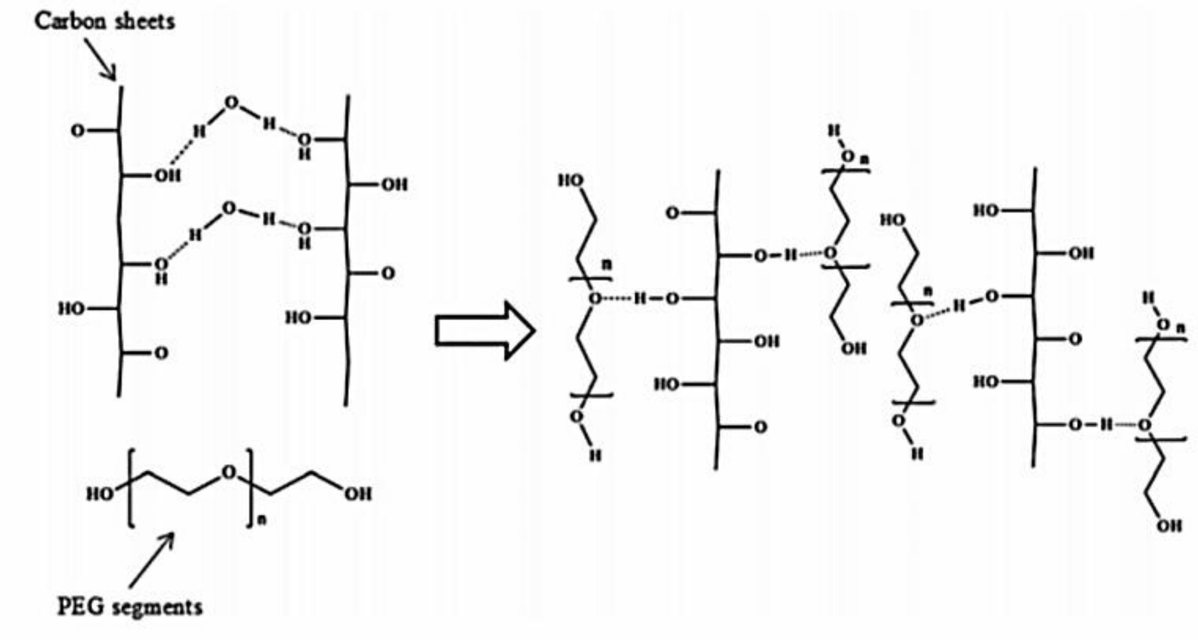

Figure 9. Bridging effect (Wang, 2012)

In linear oscillatory shear flow, strain sweep tests were performed in order to obtain the linear viscoelastic region that serves as input data for the frequency sweep test. When choosing a strain value of 10 Pa that is within the linear viscoelasticity range for all tests, this value was set, and a frequency sweep of 0.1 to 100Hz was done. Values below 0.1 Hz have been discarded because they have a very low torque value, and values above 100Hz are also discarded because the phase angle is not between 0 and 90°, and the material structure could be beginning to break. Figure 9 shows that for all concentrations the viscous modulus is much higher than the value of the elastic modulus. For GO of 2 hrs the elastic modulus is practically negligible, meaning that the material doesn't present elasticity. However, for GO of 96 hrs it can be observed that for the concentrations of 40 and 80 mg/ml the elastic modulus begins to increasing, indicating the presence of an elastic behavior. The viscous modulus in both cases were frequency dependent and the suspensions with GO of 2hrs behaved as a purely viscous liquid.

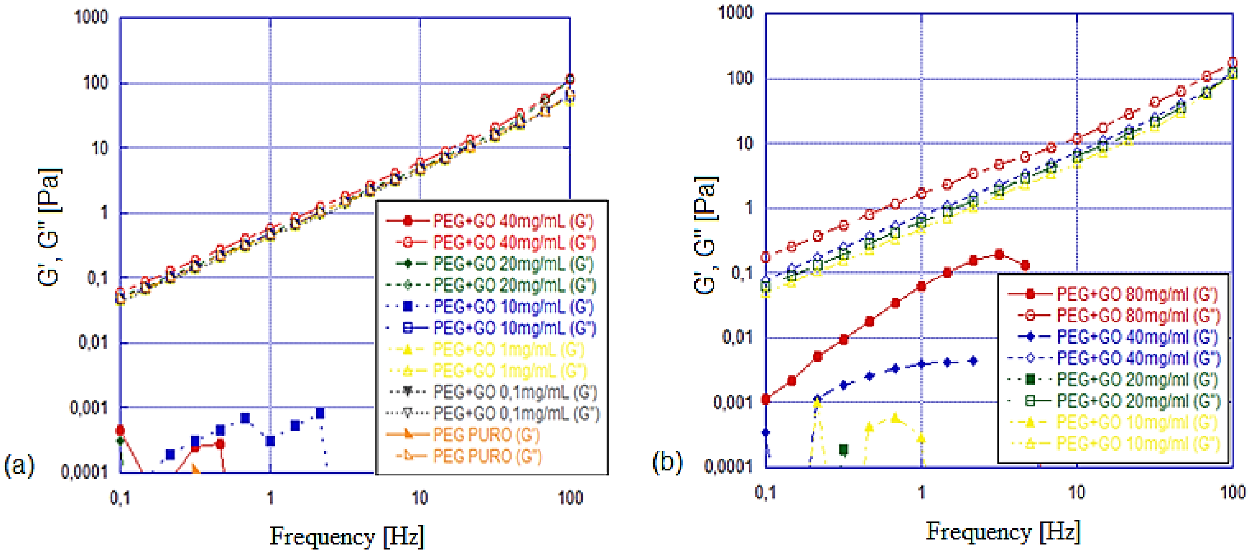

Figure 9 – frequency sweep for (a) GO of 2 hrs (b) GO of 96 hrs of oxidation

4. CONCLUSIONS

The objective of this work was to analyze the rheological behavior of graphene oxide suspensions in the presence of poly(ethyelene gycol), and to investigate the organizational structure of the suspension to observe how the sheets of graphene oxide interact with the polymer.

Through the characterization techniques it was possible to prove that the oxidation occurred, and that the longer the oxidation time, the greater the interplanar distances, the smaller the particle size and the greater number of functional groups introduced in the graphite structure.

It was possible to verify that, for all the suspensions, with the increase of the concentration of graphene oxide there was an increase in the viscosity. The samples of GO with 2 hrs of oxidation, from the pure PEG to the PEG with concentration up to 40mg/mL, presented a Newtonian behavior, while the suspensions of GO with 96 hrs of oxidation and concentrations above 20 mg/mL began to exhibit a pseudoplastic behavior. Moreover, through the oscillatory tests, it was possible to verify that only the more concentrated suspensions of GO with 96 hrs presented elasticity.

5. ACKNOWLEDGEMENTS

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