25th ABCM International Congress of Mechanical Engineering
October 20-25, 2019, Uberlândia, MG, Brazil

COB-2019-0931

RHEOLOGY OF GRAPHENE OXIDE SUSPENSIONS ON YIELD STRESS FLUID

Lorena R. da C. Moraes
Monica F. Naccache
Departament of Mechanical Engineering, Pontifícia Universidade Católica do Rio de Janeiro (PUC-Rio). Rio de Janeiro - RJ, 22451-900 - Brazil.
lenarcm@hotmail.com and naccache@puc-rio.br

Ricardo Jorge E. Andrade
Mackgraphe, Universidade Presbiteriana Mackenzie. São Paulo – SP, 01302-907 – Brazil.
ricardo.andrade@mackenzie.br

Abstract. *In this work we investigate the rheology of graphene oxide (GO) suspensions on a yield stress model fluid. Aqueous solution of Carbopol® Ultrez 10 was the dispersant used, and it is a transparent, non-toxic yield stress fluid with elasticity. The rheology of the suspensions was analyzed in relation to the dispersion method applied and concentration of GO. Tests with shear flow in permanent and oscillatory regimes were performed. For the different dispersion methods applied there was no change in the rheology of the suspensions. The increase in GO concentration leads to a decrease in elasticity, viscosity, and yield stress. pH measurements indicate the suspensions become more acidic with increasing GO concentration, although there is no strict relationship between the pH and yield stress values.*

Keywords: *Rheology, Yield stress, Carbopol, Graphene Oxide, Suspensions.*

1. INTRODUCTION

The oil industry continuously demands the development of complex fluids that perform multiple functions, such as drilling fluids. During the well drilling operation, the fluids are used to suspend and transport the shale produced to the surface, control the hydrostatic pressure of the well, stabilize the exposed formation, provide cooling and lubrication for the drill (Nasser et al., 2013; Aftab et al., 2016; Thomas, 2001). These functions must be performed consistently throughout the operation and are purely dependent on the drilling fluids properties, especially the rheological ones, mainly the viscosity, density and gel strength.

Each well presents specific conditions, such as the depth of the reservoir rock, the formations that precede it, and if it is offshore, the current legislation implemented other factors that influence the drilling operation. Therefore, the drilling fluids are extremely complex, designed to operate efficiently in each specification, which it is usual the use of different types of fluids on the various drilling stages in a same well (Caenn et al., 2017).

The use of nanomaterials to develop new fluids has been a subject of interest to many scientists around the world (Sadeghinezhad et al., 2016). Nanoparticles have improved physio-chemical properties compared to macro and micro-scalable materials, which can be attributed to their nanometric size along with their extremely high surface-to-volume ratio.

Among the several nanomaterials that have been the object of studies, carbon nanomaterials have been the most approached, and the most recent is graphene (Zarbin and Oliveira, 2013). Graphene is a two-dimensional (2D) material with a single layer of one atom thickness and consisting of carbon atoms bonded together in a hexagonal structure with double bonds (sp^2) (Ray, 2015). This structure of graphene gives it unique properties.

However, the difficulty of dispersing large flakes of graphene in aqueous media creates problems in water-based fluids. Instead, graphene oxide (the graphene sheet functionalized with oxygen groups) provides a more stable material for aqueous dispersions and maintains sheet-like morphology (Kosynkin et al., 2011) besides its synthesis being simpler.

Many researches have been done on the use of graphene oxide in fluids. Most of them use water or other Newtonian solvents as base fluid. There are also works on suspensions of GO in drilling fluids. In this case, the issue is a wide range of different materials that are used on the drilling fluids composition. In addition to the base fluid - which may be water, nonaqueous or pneumatic - clays, polymers and many other additives are used to give the fluid the properties required for its use in the well (Caenn et al., 2017). Consequently, the results of these works are specific for that particular drilling fluid used. Besides that, these works do not present a rheological the study of such fluids.

Based on that, there is a lack in the literature on how the interactions between GO and simpler non-Newtonian fluids can affect their rheology. Having this knowledge in advance will contribute to a research aimed to design better efficient fluids.

Due to environmental requirements imposed by governments and regulatory agencies, most of drilling fluids currently used today are water-based. An aqueous-based fluid with non-Newtonian behavior similar to the drilling fluids, and widely used among rheologists, is the aqueous solution of polyacrylic acid or aqueous solution of Carbopol® (trade name of polyacrylic acid produced by the Lubrizol company). Just three elements are used in this fluid, which is water, Carbopol, and a base to neutralize it. Other attributes of Carbopol include transparency, lack of toxicity and ease of handling. These characteristics make the aqueous solution of Carbopol an ideal model fluid for this study.

2. MATERIALS AND METHODS

2.1 Carbopol®

The polymer used to make the model fluid was Carbopol® Ultrez 10, a product supplied by "The Lubrizol Corporation". Carbopol® Ultrez 10 is a crosslinked carbomer, supplied as a white powder with a slightly acetic odor. Its molecular structure is presented in Figure 1.

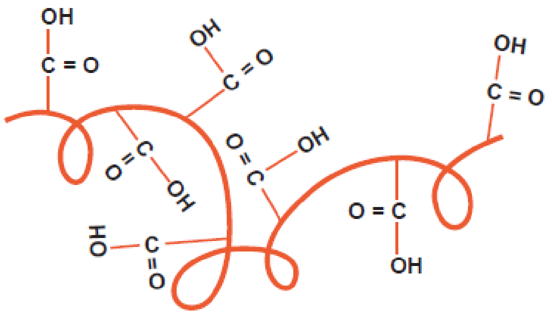

Figure 1. Schematic depicting molecule of Carbopol® polymer (Corporation, 2009).

2.2 Synthesis of Graphite Oxide (GrO)

For the synthesis of graphite oxide, the Hummers modified method with oxidation time of 2 hours was used (Oliveira et al., 2019). The materials used in the process were: graphite (graphite powder 95%, ≤ 45 µm - Sigma-Aldrich), H_2SO_4 (Sigma-Aldrich - 99%), $KMnO_4$ (Sigma-Aldrich); deionized water, H_2O_2 aqueous solution (Synth - 30%); 10% aqueous solution of HCl (Synth – 36,5-38%), ethanol (Sigma-Aldrich). With the exfoliation of GrO it is possible to obtain the graphene oxide sheets. A schematic model of the graphene structure is presented in Figure 2.

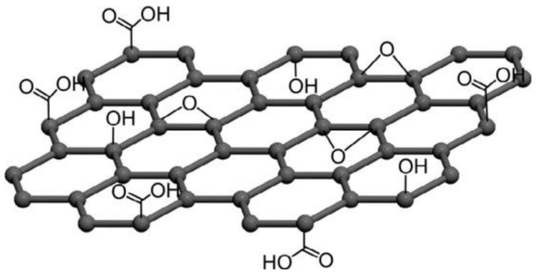

Figure 2. Molecular model of graphene oxide (Sadeghinezhad et al., 2016).

2.3 Preparation of Base Fluid

The base fluids were prepared with a fixed concentration of 0.15wt% Carbopol Ultrez 10, the objective of that concentration was to obtain a fluid with relevant yield stress (Varges et al., 2018). For the dispersion and neutralization of Carbopol were adopted recommendations of the supplier (Corporation, 2002; Corporation 2009). The neutralizing agent used was an aqueous solution of 0.18wt% sodium hydroxide (NaOH). The amount of NaOH required to neutralize the dispersion consisted of 2.3 times the amount of Carbopol. Carbopol was added to deionized water, where it remained at rest for 5 minutes. Then the mixture was stirred at 300 rpm for 1 hour. Finally, NaOH was added and the fluid continued to be stirred at 300 rpm for a further 3 days.

2.4 Preparation of Graphene Oxide Suspensions in Aqueous Carbopol® Dispersion

The suspensions were prepared in 3 different ways of preparation. Preparation mode 1 consisted of first performing the GrO exfoliation directly into the base fluid by ultrasonic bath for 2 hours at a master concentration of 10 mg/ml and thus obtaining a GO suspension. Subsequently, dilutions of the suspension from the master concentration in the base fluid were prepared.

For Preparation mode 2 the base fluid used to exfoliate the graphite oxide was modified. The amount of NaOH added to the dispersion was doubled (amount of NaOH 4.6 times the quantity of Carbopol) in order to obtain a basic pH dispersion with the attempt to obtain the final suspension with higher pH then the other ones. The master suspension was made by the exfoliation of the GrO in ultrasonic bath for 2 hours in this modified base fluid. Subsequently, the master suspension was diluted in the normal base fluid.

In preparation mode 3, prior to preparing the base fluid, a concentration of 1 mg/ml of graphite oxide was exfoliated in deionized water through the ultrasonic bath by 2 hours. Subsequently, the Carbopol dispersion was carried out in the suspensions following the procedure described in the preparation of the base fluid.

2.5 Rheological characterization

For the rheological tests the stress-controlled rotational rheometer AR-G2, of TA Instruments, was used. The geometry used in the tests was a cross-hatched parallel-plate, with 60 mm diameter, and the distance of 1mm was defined as gap. All tests were conducted at room temperature (25 °C) and before each measurement the suspensions were sonicated in ultrasonic bath for 16 minutes to ensure homogeneity. In order to verify the results obtained, all the measurements of each sample were repeated, trying to eliminate any errors that may have occurred in the measurement by some factor.

Steady-state flow tests were performed with the objective of obtaining a flow curve. The shear rate was varied from 1000 s^{-1} to 0.01 s^{-1}. The rheometer was programmed to record 5 points per decade. And, the maximum time established for the equilibrium of the point to be measured was 20 minutes.

Oscillatory tests were also performed by stress sweep. This test consists to analyze the behavior of storage module (G') and viscous module (G") with variation of stress. The purpose of this test is to verify the presence of elasticity in the fluid. For the oscillatory test, a stress variation between 0.1 Pa and 100 Pa was defined with 5 points per decade. The frequency was fixed at 1 Hz.

2.6 pH measurements

The pH of the suspensions was also evaluated. Suspensions had the pH measurements performed through a Quimis pH meter.

3. RESULTS AND DISCUSSION

The rheology of the base fluid, or pure Carbopol dispersion, will be used as a reference for analysis of the GO suspensions. Figure 3 shows the shear stress (τ) vs. shear rate ($\dot{\gamma}$) curve for the Carbopol dispersion. It is observed that as the shear rate approaches zero the curve tends to form a plateau in which the shear stress approaches a constant value τ_0 different from zero, that is, the fluid presents a yield stress. Above this stress ($\tau > \tau_0$), the relationship between shear stress and shear rate is nonlinear, characteristic of non-Newtonian fluids, and is well represented by the Power-Law function. Therefore, it can be concluded that the fluid is well represented by the Herschel-Bulkley model, described by Eq. 1.

$$\tau = \tau_0 + k\dot{\gamma}^n \tag{1}$$

The Herschel-Bulkley model characterizes the flow of non-Newtonian fluids from three parameters: the yield stress (τ_0), the consistency index (k) and the flow index (n) (Machado, 2002).

In Figure 4, the apparent viscosity (η) vs. shear rate ($\dot{\gamma}$) of the base fluid is presented. It is observed that as the shear rate increases the apparent viscosity decreases, therefore, when in flow, this dispersion is a pseudoplastic fluid.

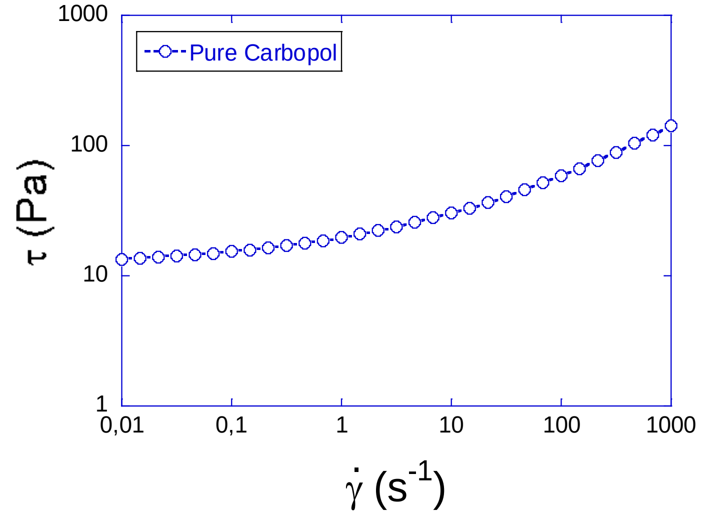

Figure 3. Shear stress x shear rate of Carbopol aqueous dispersion.

From Figure 4 it is worth noting how much the viscosity increases as the shear rate decreases, at a shear rate of 0.1 s^{-1} the viscosity reaches 100 Pa.s while for 0.01 s^{-1} the viscosity reaches 1000 Pa.s, in other words, at low shear rates the viscosity grows more sharply. The Herschel-Bulkley model determines that viscosity is infinite if ($\tau < \tau_0$), therefore such behavior confirms this is the better model to represent the base fluid.

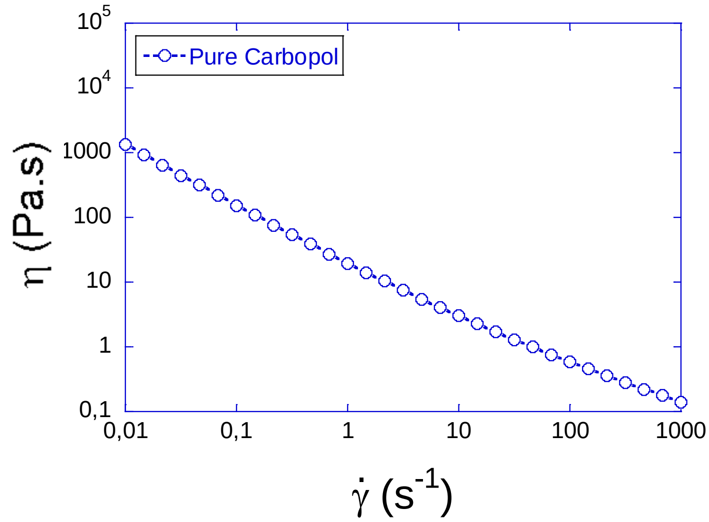

Figure 4. Apparent viscosity (η) vs. shear rate ($\dot{\gamma}$) of the base fluid.

In general, the pseudoplastic systems, although homogeneous in appearance, are in fact dispersed particles with irregular shapes, such as filaments, planar plates and droplets, are dispersions or emulsions. At rest, these materials maintain a certain irregular internal order, and are characterized, therefore, by a high internal resistance against the flow, that is, their viscosity. With increasing shear rate, many dispersed, linear or laminar particles align in the direction of the force that sustains it. This alignment between particles (or molecules) facilitates flow, reducing viscosity. The "thinning" of many liquid materials manifests reversibly, i.e., they recover their high original viscosity when shear is reduced or ceased (Machado, 2002).

These characteristics found in the Carbopol dispersion are also found in many drilling fluids. Among their functions, drilling fluids need to keep the shale produced in suspension when the drilling operation is interrupted. It is the yield stress that enables this behavior. In the oil industry, this characteristic is related to the gel strength, which is in fact related to the yield stress. During the operation, the fluid must be pumped easily, and such performance is made possible by the pseudoplastic behavior of the fluid.

Figure 5 shows the results of the oscillatory test by stress sweep for Carbopol dispersion with concentration of 0.15 wt%. From these tests the values of the storage modulus (G') - which refers to the elastic character of the material, i.e. the energy stored during deformation - and the loss modulus (G") - refers to the viscous behavior, i.e. the energy dissipated during the flow - are measured.

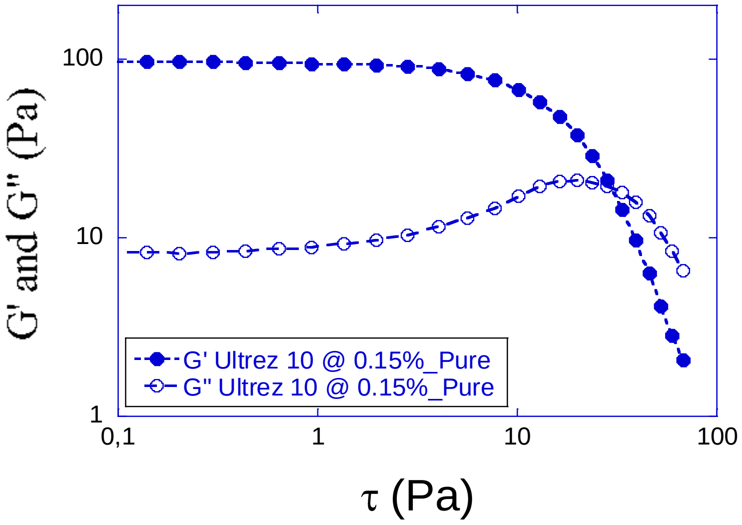

Figure 5. Stress sweep of Carbopol aqueous dispersion.

The graphs shown in Figure 5 can be divided into three regions, the linear viscoelastic region (LVR), the transition region and the nonlinear region. The LVR occurs in the range of low stress amplitude and is characterized by having constant modules (i.e., independent of the stress amplitude). In the LVR, the imposed stress amplitude is smaller compared to the strength of the bonds that support the microstructure, so its integrity is not affected. The microgels remain in their initial dispositions, that is, they can deform elastically, but do not move significantly in relation to each other. This microstructure confers a predominantly elastic behavior to the material, indicated by the value of G' to be greater than that of G". The elastic predominance indicates a low internal dissipation within the microgels (Piau, 2007).

Therefore, the Carbopol dispersion has an elastic behavior for low stresses. This result corroborates with the result of the flow curves which showed that the dispersion has a yield stress, below which we observe an elastic behavior. It is possible to observe, as the stress increases results in lower elasticity, as indicated by the decrease in G', suggesting alteration of the structuring. In the transition region, located in the middle range of the stress amplitude and around the crossover point between the modules, the stress amplitude is high enough to cause some structure failure, that is, some microgels leave their physical position and more relative movement occurs. The frictional forces arising due to the relative motion dissipate more mechanical energy, which explains the increase in G". Within this stress range the level of structuring is still high enough to retain a percolated microstructure and a considerable elastic response, as indicated by the high G' values found in this region (Varges et al., 2018).

The nonlinear region begins beyond the crossover point, where G" reaches its maximum and begins to decrease, and where G' begins to decrease much faster than G". The high stress amplitude found in this region causes a massive collapse of the bonds, so that the microstructure is no longer percolated. This imparts a net behavior to the material. The higher the stress amplitude the lower the structuring level, leading to a more liquid-like and less elastic behavior (Varges et al., 2018).

Described the rheological behavior of the Carbopol dispersion follows the analysis of GO suspensions. Initially it was necessary to evaluate if the preparation mode could affect the rheological behavior of the suspensions. From this analysis, a preparation mode was established to prepare the subsequent suspensions.

The preparation modes were elaborated to investigate two aspects explained in the following. Preparation modes 1 and 3 demonstrate whether GrO exfoliation can be affected and consequently affect rheological results. In preparation mode 1, the GrO is exfoliated for two hours only, while in preparation mode 3 besides to the ultrasonic bath exfoliation, the material is subjected to mechanical agitation for three days, due to the Carbopol dispersion protocol, which can provide higher levels of exfoliation.

Preparation mode 2 evaluates the pH variation of the suspension. Carbopol is a polyelectrolyte with pendant carboxylic acid groups along the main chain. When this polymer is dispersed in water some of the hydroxyls deprotonate and the solution becomes acidic. The dispersion is then neutralized with NaOH and the remaining hydroxyls deprotonate as well. Thus, the polymer becomes filled with anions providing chain expansion and viscosity increase. The issue is the graphene oxide particles also have pendant hydroxyls and the addition of this material in the

Carbopol dispersion could cause a decay of the suspension pH, thus affecting its rheology. Therefore, the base fluid used to make the master suspension of preparation 2 is alkaline with an attempt to maintain the pH of the final suspension neutral.

For analysis of the influence of the preparation modes, suspensions were made with 1mg/ml GO concentration. Figure 6 shows the flow curves of the suspensions prepared by the three different preparation modes.

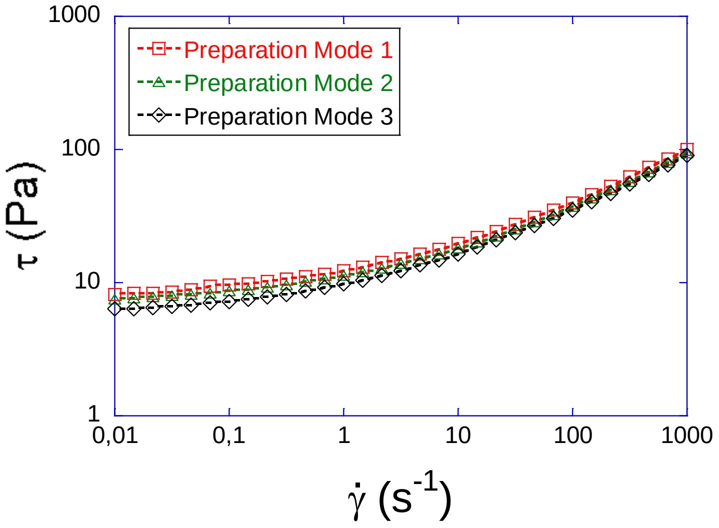

Figure 6. Flow Curves of the suspensions prepared by the three different preparation modes.

Analyzing Figure 6 it is possible to verify that the three suspensions have similar results. As the Carbopol dispersion, all curves present yield stress and they are nonlinear so, they are described by Herschel-Bulkley mode.

Table 1 shows the rheological parameters and pH values obtained for the three suspensions. The n values bellow 1 means that the suspensions are pseudoplastic. In the case of yield stress and consistency index, the differences can be considered rather small.

Table 1. Values of Herschel-Bulkley parameters and pH of the suspensions.

Parameters	Mode 1	Mode 2	Mode 3
τ_0 (Pa)	8.20	7.46	6.04
k	3.96	3.7	3.67
n	0.46	0.46	0.45
pH	5.2	7.4	5.7

On de other hand, the pH measurements show variations that can be considered significant. The variation of the pH values between the suspensions prepared by modes 1 and 3 reaches approximately 30%. pH variation like that one on the base fluid would cause considerable change in its yield stress, but this behavior was not observed in the suspensions. Therefore, there is no explicit relationship between the rheological parameter results and the pH measurements of the suspensions.

Figure 7 shows the results of the oscillatory tests by stress sweep of the suspensions made by the three different methods of preparation. The results show that all suspensions have elasticity in the linear viscoelastic region, since G' is greater than G", up to the crossover point, as well as the Carbopol dispersion. G' and G" values are very similar for the suspensions of preparation modes 1 and 2. The G' of the preparation mode 3 suspension has a lower value than the others, which means that the suspension has less ability to absorb energy and return to its initial condition when subjected to stresses that are within the LVR range. However, all suspensions present a crossover point between G’ and G” around the same stress value, in other words, for the three preparation modes, the stress amplitudes that break structures of the suspensions to the point of starting flow are close. This is an analogous behavior to that observed for the flow curves. The yield stress points - which is the stress required to start the steady-state flow - of the suspensions are also around the same value.

These results indicate that there is no significant rheological change for the suspensions prepared by the three different preparation modes. Thus, and due to its simplicity, preparation mode 1 was chosen as the standard preparation mode of the suspensions.

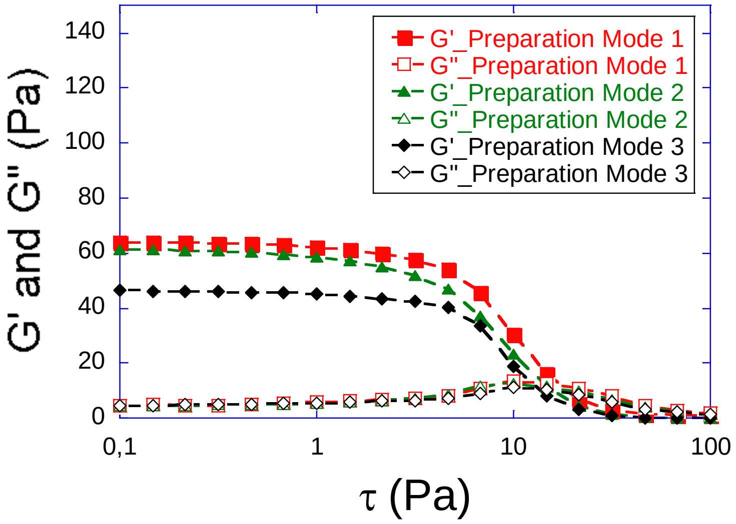

Figure 7. Stress sweep of suspensions prepared by the three different preparation modes.

After determining the best way to prepare the suspensions, tests were performed to analyze the influence of the GO concentration on the rheological behavior of the suspensions. The suspensions were prepared in aqueous dispersion with GO concentrations of 0.1 mg/ml, 1 mg/ml, and 5 mg/ml. It is worth to mention it was possible to see particles in the suspensions after they were ready, which indicates that the GrO was not completely exfoliated.

Figure 8 shows the flow curves of the suspensions with different GO concentrations. From the flow curves is possible to observe that there was no significant change for suspensions with concentration of 0.1 mg/ml GO in relation to the behavior of the base fluid. However, the increase in GO concentrations leads to a considerable decrease in viscosity and yield stress.

The microstructure present in the Carbopol dispersion at rest is formed by intermolecular bonds between the polymer chains and water. The ions formed in the polymer after neutralization establish ion-dipole bonds with water that end up binding one chain to another through a network of bonds, which are called physical crosslinking (Noro et al., 2012; Appel et al., 2012; Maitra and Shukla, 2014; Seiffert et al., 2015). The introduction of GO in the system is influencing the structuring of the fluid leading to a decrease in yield stress and viscosity.

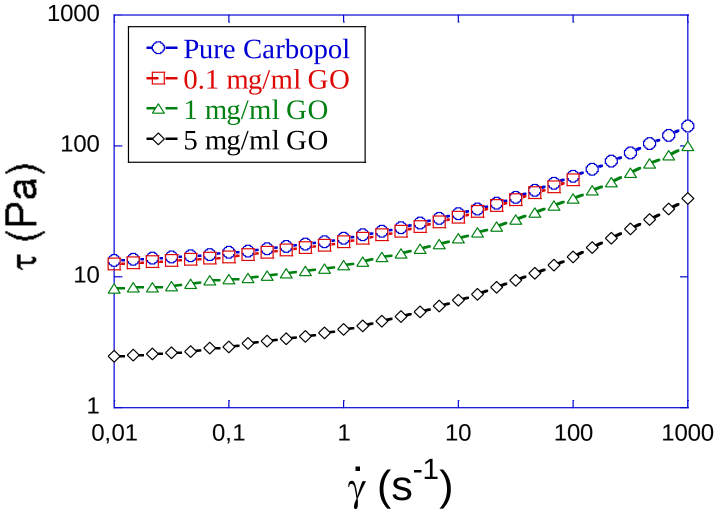

Figure 8. Flow curves of the suspensions with different GO concentrations.

The influence of GO concentration on the Herschel-Bulkley model parameters and the pH of the suspensions is shown in Table 2. The results show a significant decline in yield stress, but small variations in the index flow.

Table 2. Influence of GO Concentration on the values of Herschel-Bulkley parameters and pH of the suspensions.

	GO Concentration (mg/ml)			
Parameters	0	0.1	1	5
τ_0 (Pa)	14.35	11.77	8.20	2.64
k	6.5	6.6	3.96	1.22
n	0.44	0.42	0.46	0.50
pH	6.6	6.3	5.2	3.9

This tendency indicates that for higher concentrations of GO, the interference of the nanoparticle in the formation of the hydrogen bonds between the chains of Carbopol will be such that the fluid will not have the capacity to form a microstructure, voiding the yield stress. However, the pseudoplastic characteristic remains, since despite an increase of *n*, the values are still far from 1.

The pH measurements show that increasing the amount of GO makes the suspensions more acidic. This behavior was expected, since the introduction of GO in the Carbopol dispersion promotes the deprotonation of the GO hydroxylates. At lower pH the weak acids - such as the hydroxyls present in Carbopol and GO - lose the ability to dissociate, so part of the oxygen groups ceases to be negatively charged, which may contribute to the decrease of the rheological properties of the suspension.

Figure 9 presents the oscillatory tests by stress sweep of the suspensions with different GO concentration. Corroborating with the results obtained by the flow curve, the crossover point occurs at lower stress amplitudes for the suspensions with higher GO concentration, and this behavior is directly connected to the yield stress. Moreover, in the LVR the values of G' decline with increasing GO concentration, and the difference between G' and G" becomes smaller, i.e. elasticity decreases. The results indicate that for suspensions from a certain GO concentration, G' and G" will be at the same order.

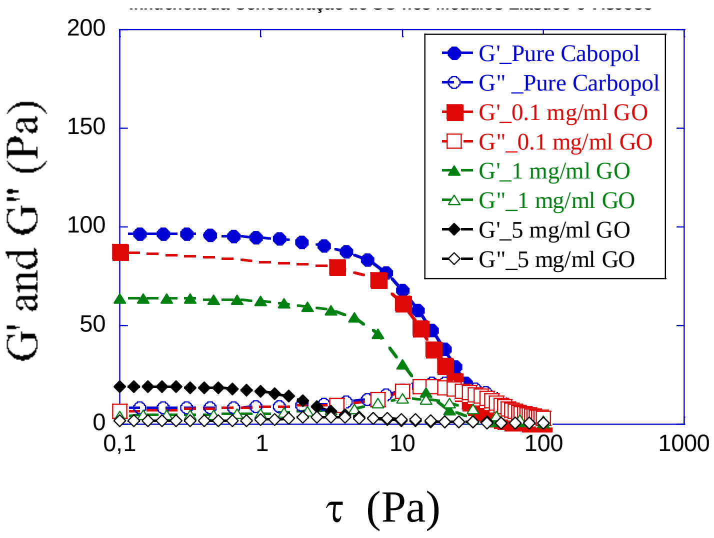

Figure 9. Stress sweep of the suspensions with different GO concentration.

4. CONCLUSIONS

The results show the suspension of graphene oxide in Carbopol aqueous dispersion decreases the rheological properties of the fluid.

The rheological characterization of the pure Carbopol dispersion indicates this fluid represents a model system of a drilling fluid. The dispersion has a yield stress and pseudoplastic behavior and is well represented by the Herschel-Bulkley model.

The suspensions prepared by the three different modes of preparation showed similar rheological results, indicating that the order of insertion of the GrO and the slight increase of basic pH, would not influence the final rheology of the suspensions. The tests results presented the same properties of the Carbopol dispersion, and the suspensions showed yield stress and pseudoplastic behavior. Thus, preparation mode 1, being simpler, was chosen as the standard method for the other tests.

The presence of GO in the suspensions prevents the formation of physical crosslinking between the polymer chains and therefore the rheological parameters decrease with the increase of the GO concentration.

The results indicate that from a certain concentration of GO in the suspensions, the elasticity and the yield stress decrease.

The pH measurements show that increasing the amount of GO makes the suspensions more acidic, which may contribute to the decrease of the rheological properties of the suspension. However, by comparing this result with the pH results for suspensions with different modes of preparation, a suspension with pH of 7.4 has similar yield stress of a suspension with pH of 5.2, which indicates that the decrease in rheological properties mainly occurs by the impediment of the physical crosslinking formation along the polymer chains caused by the presence of GO.

The physio-chemical structure of Carbopol is different from a drilling fluid, although they have similar rheology. The results obtained represent the interaction of GO with negatively charged polymer chains dispersed in water. Many of the additives used in the production of drilling fluids may have these characteristics.

5. ACKNOWLEDGEMENTS

The authors thank CAPES, CNPq, FAPERJ and Petrobras.

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