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RHEOLOGICAL CHARACTERIZATION OF NON-COLLOIDAL SUSPENSIONS WITH NON-NEWTONIAN BULK: INVESTIGATING THE IMPACT OF MICROPARTICLES

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Abstract. *The rheological behavior of complex fluids such as non-colloidal suspensions is currently a required topic in the cosmetic, food, and petroleum industries. The behavior of non-colloidal suspensions is complex since these are soft materials made from the dispersion of particles with diameters greater than 10 µm in a liquid matrix. The study of the rheological behavior of the yield strength of non-colloidal suspensions and thixotropic, is underexplored. The present work aims to analyze the particle size and concentration dependence on static and dynamic yield strength in viscoelastic non-colloidal suspensions. Carbopol with a concentration of 0.2%wt was used for preparing the non-colloidal suspensions with Polyamide particles with 5, 20, and 50 µm of diameter. Rheological measurements were performed in a rotational rheometer – TA instrument with cross-hatched parallel plate geometry, with gap of 1 mm. Steady-state flow curves were carried out to understand liquid-like behavior and determine dynamic yield strength; on the other hand, creep tests were conducted to measure the static yield strength. Finally, shear stress amplitude sweeps oscillatory tests were carried out to comprehend the suspension structure in the solid-like behavior.*
The experimental results indicate that compared to the pure Carbopol solution, the suspension of 5 µm particles exhibit an increase in yield strength, whereas the suspensions of 20 µm and 50 µm particles exhibited a decreasing behavior for this parameter. The decreasing behavior for the yield stress is more noticeable for 20 µm particles suspension. This effect could be attributed to the increase in the spacing between the base fluid and the particles, since the yield strength of the fluid is caused by the intermolecular forces of the microstructure of the fluid. Creep test depicts that the static yield stress behavior during yielding process, follows the same trend of the dynamic yield strength. The stress amplitude sweeps oscillatory tests revealed that Storage modulus (G') in the Small Amplitude Oscillatory Shear (SAOS) region decreased with an increase in particle concentration, but there were no significant variations observed between the 20 µm and 50 µm particles in the pre-yielding stage. These results indicate the presence of flow heterogeneities in the 20 µm and 50 µm suspensions. The suspension of particles displays a microstructure that weakened the Carbopol polymeric chains as a result of hydrodynamic interactions between the particles spacing.

Keywords: *drilling fluid, non-colloidal suspension, Carbopol, viscoelastic, yield strength.*

1. INTRODUCTION

Suspensions of particles in a fluid matrix, whether Newtonian or Non-Newtonian, present intriguing and formidable challenges, and their rheology has been extensively studied. These suspensions have diverse applications in various industries, including the extrusion of confectionaries (Maskan and Altan, 2016), 3D printing of food (Wilms et al., 2021), non-food pastes (Buswell et al., 2018), blood pumping through arteries (Stroev et al., 2007), and the pumping of cement slurries used in oil well drilling (Quitian et al., 2022). As the global demand for oil and gas continues to increase, drilling activities, especially in high-pressure and high-temperature (HPHT) conditions (Wang et al., 2022), (Gautam et al., 2022), (Greenaway, R. et al., 2008), have escalated. The unique difficulties posed by HPHT environments necessitate the development of advanced technologies, improved drilling fluids, and enhanced knowledge to ensure safe and efficient operations. Extensive research has been conducted to investigate the impact of HPHT conditions on the rheological properties of drilling fluids and address related issues. Proper selection of drilling fluids is crucial to avoid costly delays, wellbore instability, and environmental risks.

Drilling fluids are complex suspensions comprising a liquid phase, typically water or oil, along with various solid particles, additives, and chemicals. The rheological properties and solids fraction of drilling fluids significantly influence their performance and the overall drilling process. Understanding the relationship between the solids fraction and drilling fluid behavior is crucial as it directly affects drilling efficiency, wellbore stability, and environmental risks. Numerous studies have examined the impact of solids fraction on drilling fluid properties, highlighting the challenges faced by the oil industry. One key issue associated with higher solids fractions in drilling fluids is the increase in viscosity and plastic viscosity. As the concentration of solid particles increases, the drilling fluid's viscosity rises, impacting its flow characteristics and pumpability. This heightened viscosity can result in higher frictional pressures, necessitating increased pump power and potentially causing excessive wear on drilling equipment. Furthermore, the presence of solid particles in drilling fluids can lead to wellbore instability. Particles may settle or pack at the bottom of the wellbore, leading to differential sticking and reduced drilling fluid circulation. This can result in costly delays and difficulties in extracting the drill string, posing challenges for well completion and overall productivity. Solid particles in drilling fluids can also contribute to drilling-induced formation damage. Fine particles, such as clays and drilling cuttings, have the potential to migrate into the formation, clogging pore spaces and reducing permeability. This negatively impacts well productivity and increases the risk of reservoir damage (Amani et al., 2012).

Understanding the rheological properties of these suspensions, specifically drilling fluids, has been a subject of study for many years. Figure 1 provides an illustration of three distinct types of fluids, depicting solutions, colloidal suspensions, and non-colloidal suspensions (Dai and Tanner, 2020). This classification is based on particle size, and the phenomena occurring within the suspension heavily depend on the particles present. Various forces, such as Brownian forces, Van der Waals forces, or hydrodynamic forces, can act between the particles, modifying the rheological behavior of the suspensions.

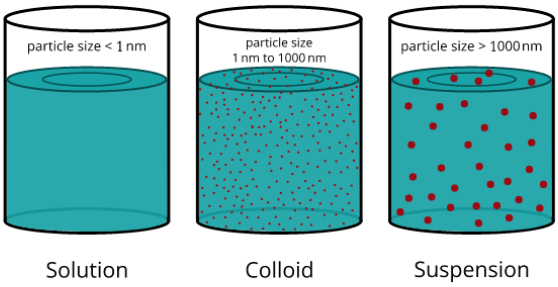

Figure 1**.** Representation of suspended systems based on the size of the dispersed or suspended particles.

The interparticle forces present in suspensions significantly influence their rheological behavior. For instance, Brownian forces, resulting from the thermal motion of particles, contribute to the random movement and collisions of suspended particles. Van der Waals forces, arising from electrostatic interactions and induced dipole attractions, can lead to particle aggregation or repulsion depending on the distance between particles. Hydrodynamic forces, originating from fluid flow, exert shear stresses on the suspended particles, influencing their arrangement and the overall flow behavior of the suspension (Tanner, 2015). Apart from size-based classification, suspensions can also be classified based on the concentration of particulate matter, ranging from diluted to semi-diluted to concentrated suspensions.

In recent times, with the advent of new technological tools facilitating the analysis of complex fluids, several authors (Tanner, 2015), (Bergenholtz et al., 2002), (Shewan et al., 2021) have focused on studying non-colloidal suspensions. Colloidal suspensions consist of dispersed phase particles with diameters typically ranging from approximately 1 *nm* to 1 μm (Qin and Zaman, 2003), (Zhou et al., 2001). On the other hand, non-colloidal suspensions are those where Brownian motion can be neglected (Shewan et al., 2021), (Tanner, 2018), (Dagois-Bohy et al., 2015), (De Rosso and Negrão, 2022). The differentiation between colloidal and non-colloidal suspensions is determined by the Brownian diffusion time scale, which is related to the Peclet number (Tanner, 2018). For example, considering a suspension with spherical particles measuring 1 micrometer in diameter and a viscosity of 1 *mPa.s* at 25 ºC, the resulting Peclet number is 4.6 times the shear rate, indicating a colloidal range. However, if the particles have a diameter of 10 μm and a viscosity of 1 *Pa.s*, the Peclet number becomes 4.6 times ten to the power of six times the shear rate. In such cases, the Peclet number for non-colloidal suspensions greatly exceeds 1, and in this study, the Peclet numbers are greater than ten to the power of seven, where Brownian forces are considered negligible.

The rheological behavior of non-Brownian viscous suspensions was initially described in terms of effective viscosity, and considerable effort has been dedicated to determining this enhanced viscosity as a function of volume fraction (Howard, 2000). Non-colloidal suspensions can be prepared using both Newtonian and non-Newtonian fluids, each fluid matrix exhibit distinct rheological behavior. Although many studies suggest that non-colloidal suspensions in a liquid matrix display Newtonian behavior, it has been observed that these suspensions often exhibit non-Newtonian rheological behavior. In the present era, issues related to the transition from solid to liquid-like behavior due to shear stress imposition are increasingly prevalent. Start-up flow poses one such challenge, as it can lead to clogging and sedimentation problems. The yield stress of a non-colloidal fluid and the yielding transition can both be studied through rheometric experiments. Therefore, it is crucial to investigate the solid-like state and the transition from solid-like to liquid-like behavior in different processes, particularly during the drilling of wells in HPHT conditions or well abandonment.

The objective of this work is to analyze the dependence of particle size and concentration in non-colloidal suspensions (drilling fluids) during yielding and pre-yielding flow. This technological challenge motivates an experimental study of non-Newtonian fluids in the liquid-like and solid-like regimes, with a specific focus on yield stress and yielding in non-colloidal suspensions. Studying phenomena related to the transition from solid-like to liquid-like regimes is vital for start-up flow processes. Additionally, developing a methodology to identify changes in properties due to wall slip in the liquid regime and during flow transition is essential. These conditions contribute to the understanding of complex fluids (drilling fluids). Furthermore, examining the behavior of non-colloidal particles and the fluid within simple tubes and contractions will enhance our understanding of these fluids.

2. MATERIALS AND METHODS

The aim of this study was to create formulated samples of non-colloidal suspensions that mimic the behavior of drilling fluids in the annular region of the drilling bit. These samples were prepared to serve as a base fluid, enabling the identification of crucial phenomena and improving operational conditions during drilling.

2.1 Materials

The non-colloidal suspension in this study was prepared by utilizing Carbopol dispersion Ultrex [Figure 2(a)] at a concentration of 0.2%wt. Carbopol dispersion Ultrex is a commercially available product that contains water-dispersed particles of cross-linked polyacrylic acid polymer. To achieve the desired pH value, the dispersion was neutralized using aminopropane, which served as a pH adjuster. For the experiment, Polyamide particles (PSP) [Figure 2(b)] were employed, with a relative density of 1.15 and sizes of 5μm, 20μm, and 50μm.

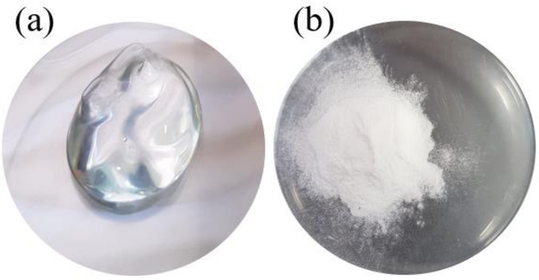

Figure 2. Materials used in the formulation of non-colloidal suspensions (a) Carbopol dispersion Ultrex and (b) PSP microparticles.

These PSP particles were obtained from a reputable source (Dantec Dynamics co., Denmark). Prior to their usage, the PSP particles were handled with utmost care to prevent any contamination and were stored in a dry and controlled environment. The suspensions were prepared using the Hamilton Beach HMD200 model, a low-capacity industrial mixer. The particles were added gradually to the Carbopol and homogenized at a speed of 1000 *rpm* for 20 minutes. After the homogenization process, the prepared samples were left undisturbed at room temperature for 24 hours. To visualize the suspensions an Olympus BX51 microscope was utilized and to measure the size distribution of particles was employed a Microtrac S3500. Figure 3 displays the images obtained from the microscope, providing a zoomed image of particle distribution within the non-colloidal suspensions. Upon visual examination using the microscope, it was observed that some suspensions exhibited particle agglomeration. These agglomerates consisted of clusters of particles that could potentially disrupt precise rheological measurements. These agglomerations were subsequently dispersed using an ultrasonic bath.

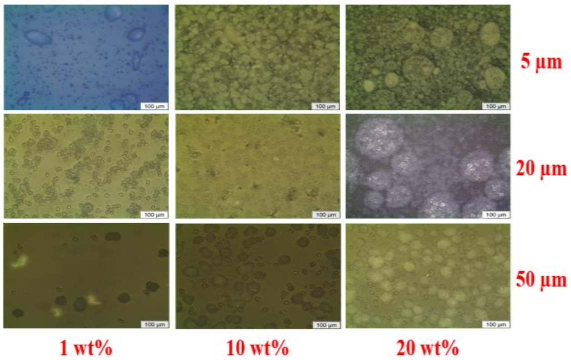

Figure 3. Visualization of the different non-colloidal suspensions.

2.2 Equipment

The rheological tests were conducted using a Rotational Rheometer, model DHR-3 (TA Instruments, USA). The rheological measurements were performed using a cross-hatched parallel plate geometry. The plates had a diameter of 40 *mm*, providing a suitable surface area for the sample under investigation. The gap between the plates was set to 1 *mm*, which allows for controlled shearing and deformation of the material during testing.

2.3 Rheometer tests

Steady-state flow curves were generated to investigate the liquid-like behavior of non-colloidal suspensions. Different shear rates were applied to the samples for a duration of 120 seconds. The sample was placed in the parallel plate geometry, and initially, a temperature of 22°C was achieved through a Peltier system in the rheometer. Subsequently, the fluid was pre-sheared at 100 s^{-1} for 300 seconds to ensure the same shear history in all measurements. Then, the sample was allowed to equilibrate at zero shear for 30 seconds, after which shear rates were ramped up in different steps.
The steady-state flow curves provided insights into the viscosity and flow characteristics of the suspensions under varying shear conditions.
A creep test was conducted to examine the material's behavior during yielding. The fluid was pre-sheared at a shear rate of 30 s^{-1} for 1 minute to ensure uniform conditions. Subsequently, the sample was subjected to a zero-shear stress for a duration of 300 seconds. This step allowed the material to relax and reach a steady-state condition before further testing. Finally, the shear stress was applied for 1000 seconds.
Shear stress amplitude sweeps were performed as oscillatory tests to gain insights into the microstructure of the non-colloidal suspensions prior to yielding. This analysis provided information on the stability and structural properties of the suspensions before undergoing yielding behavior. The test involved imposing a frequency of 1 Hz and a strain of 0.001, and a logarithmic ramp of shear stress amplitudes ranging from 0.01 to 300 Pa was applied. This allowed us to observe the behavior of dynamic moduli (G' and G").

3. RESULTS AND DISCUSSION

In this study, we aim to analyze and investigate the distinct behavior exhibited by materials across three fundamental regimes: the solid-like regime, the liquid-like regime, and the transition from solid-like to liquid-like behavior. Understanding the characteristics and transitions between these regimes is crucial for gaining insights into the mechanical properties and rheological behavior of drilling fluids.

Initially, a sample of pure Carbopol, representing the viscoelastic behavior of drilling fluids, was characterized to establish its baseline behavior. This characterization aimed to understand the modifications induced by the addition of particles to the Carbopol matrix. The pure Carbopol sample exhibited shear thinning behavior, where the viscosity decreased as the shear rate increased. This behavior is commonly observed in non-Newtonian fluids and indicates that the Carbopol undergoes structural changes under shear. To quantitatively describe the flow behavior of the pure Carbopol, the experimental data were fitted to the Herschel Bulkley model. The fitting analysis demonstrated a good agreement between the measured data and the model. The Herschel Bulkley model fitting revealed that the pure Carbopol sample exhibited a yield stress of 60 Pa, as shown in the Figure 4. This indicates that the fluid requires a certain minimum shear stress to initiate flow and confirms the viscoelastic nature of the Carbopol.

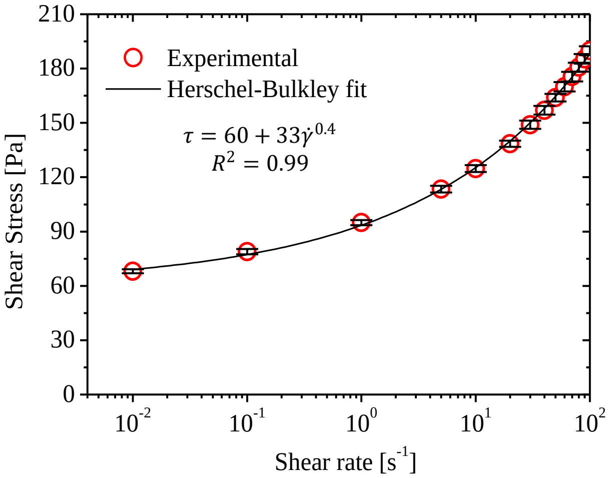

Figure 4. Steady-state flow curve pure Carbopol at 22 ºC and atmosphere pressure. Experimental data were fitted along with the best fit of the Herschel Bulkley equation (black line). The error bars denote the standard deviation of experiments performed in triplicate.

In contrast, steady-state flow curves were conducted to examine the liquid-like characteristics of non-colloidal suspensions. The experimental findings of these suspensions were compared to Carbopol, as well as the fitted values of the model. When comparing the suspension containing 50µm particles, it exhibited shear thinning behavior. However, as the concentration increased, both the yield stress and viscosity decreased. A similar trend was observed for the suspension with 20µm particles, where the yield stress decreased with increasing in concentration. It is notice that the most significant point of the presentation emerged when analyzing the suspension with 5µm particles, which exhibited an opposite behavior. In this case, both the yield stress and viscosity increased with the concentration. Additionally, when considering the effect of the three particle sizes, it was found that the suspension with 20µm particles had a more pronounced impact than the suspension with 50µm particles, as shown in the Figure 5. Conversely, the suspension containing 5µm particles exhibited an opposite effect. It is evident that the non-colloidal suspensions display a decrease in the gel strength with the increase of the particles during the flow of the fluid and maintaining their shear-thinning behavior.

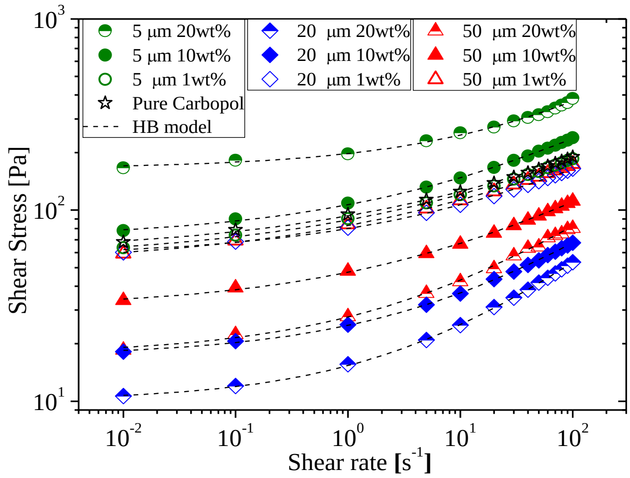

Figure 5. Steady-state flow curves of non-colloidal suspensions with 5, 20, and 50 µm particles in concentrations of 1, 10, and 20 %wt for each particle size

The data clearly demonstrates that the dynamic gel strength of the 5 µm suspension significantly increased nearly threefold as the particle concentration reached 20%wt, compared to the 1%wt suspension. In contrast, the suspensions containing 20µm and 50µm particles exhibited a gradual decrease in gel strength. Additionally, an important observation is the consistent behavior of the fluid indicated by the flow behavior index fit constant "n," which remained unchanged in most instances.

The transition from a solid-like regime to a liquid-like regime refers to a significant change in the behavior exhibited by a material as it undergoes deformation or experiences an increase in stress. This transition is often accompanied by alterations in the material's rheological properties, such as viscosity, flow behavior, and gel strength. In addition to studying this transition, another important test commonly employed to investigate the behavior of materials during yielding is the creep test. The primary objective of the creep test is to gain insights into how a material responds under sustained loading conditions. In the conducted experiments, the creep test involved subjecting the material to pre-shearing at a constant rate of 30 s^{-1} for a duration of 1 minute. Following this pre-shearing phase, the sample was left under a zero-shear stress condition for 300 seconds, allowing it to relax and stabilize. Subsequently, shear stresses were applied for a period of 1000 seconds, enabling the measurement of the material's response to prolonged deformation.

In the context of determining the static yield stress (yield point), the creep test proved be valuable. By monitoring the shear rate values over time, it was possible to identify the point at which the shear rate approached zero, indicating that the sample ceased shearing. This critical point corresponded to the static yield stress of the material. In the present example, the static yield stress for non-colloidal suspension of 20 microns and 20%wt in particle concentration varied between 10 and 5 Pa, as seen in Figure 6.

Further investigation revealed intriguing observations when studying suspensions of different particle sizes. For the 50µm suspensions, an inverse relationship between concentration and yield stress was observed in the Figure 6. Specifically, as the concentration of particles increased, the yield stress exhibited a decreasing trend. Similarly, for the 20µm suspensions, an analogous decrease in yield stress with concentration was noted. However, the most interesting finding emerged from the analysis of the 5µm suspension. Surprisingly, the yield stress showed an opposite behavior compared to the other suspensions. It was observed that the yield stress increased as the concentration of particles in the suspension increased. This unexpected trend demands further investigation and highlights the intricate relationship between particle size and material behavior during yielding.

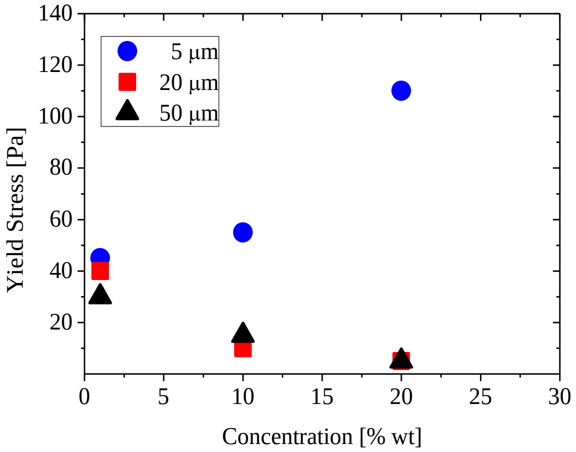

Figure 6. Yield stress as a function of concentrations of suspension with 5, 20 and 50µm particles for concentrations of 1, 10 and 20%wt

The solid-like regime of a fluid is characterized by its gel-like properties or viscoelastoplastic behavior when at rest or under low shear conditions. This regime holds significant importance in various aspects of drilling fluid behavior and performance. One of the key benefits of the solid-like regime is its impact on wellbore stability. The gel-like structure formed by the drilling fluid helps create a filter cake on the wellbore wall. This filter cake acts as a barrier, preventing fluid invasion into the surrounding formation and maintaining the stability of the wellbore itself. By minimizing fluid loss and maintaining pressure control, the solid-like properties of the fluid contribute to efficient and safe drilling operations.

In addition to wellbore stability, the solid-like regime also plays a crucial role in the suspension of drill cuttings. As the fluid exhibits solid-like behavior, it can effectively suspend and carry the drill cuttings to the surface. This prevents the cuttings from settling at the bottom of the well, which could lead to blockages, hinder drilling progress, or even cause damage to the drilling equipment. By facilitating efficient removal of cuttings, the solid-like behavior of the fluid ensures smooth drilling operations. Moreover, the solid-like properties of the drilling fluid are instrumental in erosion control. The fluid's gel-like characteristics provide a protective layer along the wellbore walls, minimizing erosion and maintaining the integrity of the drilled hole. By preventing the degradation of the wellbore structure, the solid-like regime enhances the overall performance and lifespan of the well.

Turning to the experimental techniques employed, shear stress amplitude sweeps oscillatory tests were conducted to gain insights into the microstructure of the fluid prior to yielding as shown in Figure 7. These tests involved subjecting the fluid to pre-shearing for 1 minute at a rate of 30 s^{-1} (reciprocal second), followed by a resting period of 1 minute. Subsequently, a ramp of shear stress with an amplitude of 0.01 at 300 Pa was imposed to examine the fluid's response. Analyzing the dynamic moduli in comparison to a 50µm, it was observed in the Figure 7 that the storage modulus values decreased by approximately fourfold. The post-test appearance of the fluid also exhibited a flatter profile, suggesting possible modifications in the Carbopol structure, which could have led to a reduction in its solid-like behavior. Similarly, for the 20µm suspension, the storage modulus showed a decrease of approximately twofold [Can see Fig. 8]. This finding was reflected in the altered appearance of the fluid after the test, indicating changes in its overall characteristics.

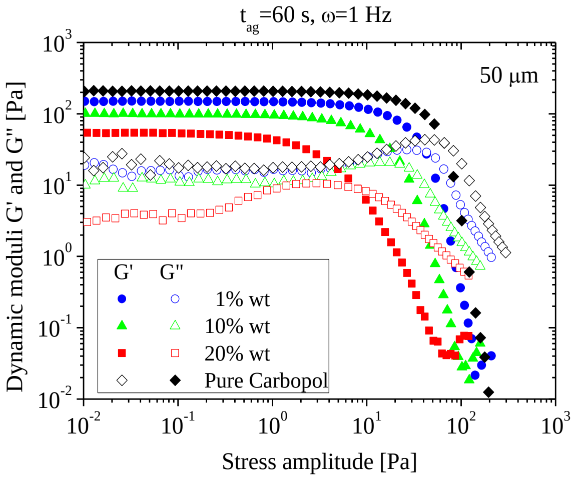

Figure 7. Storage and loss moduli (G´ and G´´) as a function of the shear stress amplitude of the suspension with 50µm particles.

In contrast, the 5µm suspension displayed a different behavior. In the stress amplitude oscillatory sweep (SAOS) region, the dynamic moduli demonstrated an increase with concentration, as shown in Figure 8. The storage modulus, in particular, exhibited a remarkable tenfold increase compared to the reference Carbopol fluid. This observation suggests significant alterations in the fluid's rheological properties, potentially attributed to the presence of smaller particles and their interactions within the suspension.

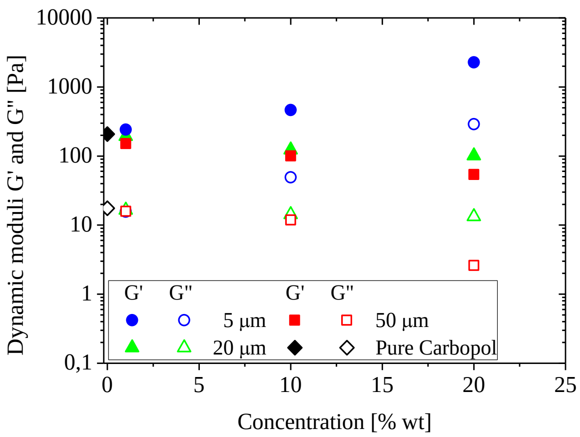

Figure 8. Storage and loss moduli (G´ and G´´) as a function of the concentration of the suspension with 5, 20, and 50µm particles.

Overall, these experimental findings shed light on the intricate relationship between particle size and concentration, and its consequences on the behavior of the drilling fluid within the solid-like regime.

4. CONCLUSIONS

In conclusion, the presence of particles suspended in Carbopol significantly affects the rheological behavior of drilling fluids. The concentration of particles becomes a critical factor in determining the extent of this influence, playing a crucial role in the performance of these fluids during drilling operations. For drilling fluids containing particles with sizes of 20 and 50µm, it was observed that higher concentrations of particles resulted in lower yield strength and decreased viscosity of the fluid. This finding implies that as the concentration of these particles increases, the fluid becomes less resistant to deformation and exhibits reduced flow characteristics. These observations have important implications for wellbore stability, cuttings suspension, and erosion control, as the fluid's ability to maintain a stable filter cake, suspend cuttings, and prevent wellbore erosion may be compromised with higher particle concentrations at these sizes. Understanding the effects of particle concentration on these key properties is crucial for optimizing drilling fluid formulations and ensuring the efficiency and safety of drilling operations. Interestingly, a contrasting behavior was observed for particles with a size of 5µm. In this case, an increase in particle concentration led to an opposite effect, with the fluid exhibiting higher yield strength and increased viscosity. This finding suggests that the presence of particles at this size range introduces unique interactions and structural modifications within the fluid, resulting in enhanced resistance to deformation. Further investigation is required to unravel the underlying mechanisms and the implications of this behavior in drilling fluid performance. The non-monotonic effect of particle concentration on the rheological behavior of drilling fluids is a complex phenomenon that demands further research. The interaction between particle size, concentration, and their effects on the fluid's flow properties must be comprehensively explored to gain a deeper understanding of the underlying mechanisms. Future studies should focus on elucidating the particle-fluid interactions, examining the microstructure of these suspensions, and developing predictive models to optimize drilling fluid formulations for enhanced wellbore stability, cuttings management, and erosion control. By advancing our understanding in this field, we can strive to improve drilling practices, mitigate operational challenges, and enhance the overall efficiency and sustainability of drilling operations.

5. ACKNOWLEDGEMENTS

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