

## Cellulose nanofibers facilitate heavy particle suspension in drilling fluids

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### ABSTRACT

The intermittent nature of flow during drilling and improved understanding of solids suspension allows enhanced fluid formulations. To suspend heavy particles, the yield strength of the fluid must be tailored by rheological methods. Yield strength determination with modern rheometers provides accurate results in comparison to oil industry traditional dynamic yield strength calculation using a Model 35A viscometer. In this study, we analysed the suspension capability of a water-based drilling fluid containing cellulose nanofibers, clay, polyanionic cellulose, and additives. Addition of cellulose nanofibers improve the static yield strength and elastic modulus, without largely affecting the steady shearing flow behaviour.

### INTRODUCTION

Drilling fluids in oil and gas industry are very important fluids which have several functions, such as suspend and remove cuttings, control formation pressure, lubricate, cool and transmit hydraulic energy to the drill-bit. Water-based drilling fluids exhibit viscoelasticity, apparent yielding, reversible thixotropy, and shear-thinning rheology, affording many rheological phenomena to be exploited for the purposes of optimizing the field performance [1] of a given drilling fluid formulation. Oil and gas industry has focused in describing the drilling fluid to accomplish its functions by

characterizing fluidity and viscosity in a repeatably way [2], initially adopting the Marsh funnel measurement, and then implementing the Bingham model as the fluid plastic flow common standard for shear stress measurement. Lately, the Herschel-Bulkley model has been applied for predicting drilling fluid rheological properties from the fluid viscosity flow profile using a six-points shear deformation spectrum from  $5.11 \text{ s}^{-1}$  to  $1022 \text{ s}^{-1}$ . This rheological model also helped to describe an important parameter of the drilling fluid, the yield point or yield stress, although it is obtained by a mid to high shear deformation regime.

As drilling fluid rheological characterization is an evolving knowledge, the need to go further in understanding the drilling fluids viscoelasticity and yield stress, relates to the way in which the material yields under different types of stresses [3] [4], such as extension, compression, shear, tangential, etc. The study of viscoelasticity which is the property of materials that exhibit both viscous and elastic characteristics when undergoing deformation is of utmost importance. It is necessary to perform a rheological characterization with a wider spectrum, analyzing low shear rate behavior [5] and the yield stress associated to it, to complement and update the testing methods described by industry standards.

Water-based drilling fluids exhibit viscoelasticity, *apparent* yielding, reversible

thixotropy, and shear-thinning rheology, affording a multitude of rheological phenomena to exploit for the purposes of optimizing the field performance of a given drilling fluid formulation. An exhaustive constitutive relation capturing the totality of the rheological stress ( $\tau$ ) versus strain rate ( $\dot{\gamma}$ ) response is obtainable from a generalized form of the Jeffreys model [6]:

$$\dot{\gamma} + \dot{\gamma}\theta_2 = \frac{\theta_2}{\eta_\infty} \left( \frac{\tau}{\theta_1} + \dot{\tau} \right) \quad (1)$$

In the overall constitutive relation, the characteristic relaxation and retardation timescales are denoted by the symbols  $\theta_1$  and  $\theta_2$ , respectively. Specifically,

$$\theta_1 = \frac{\theta_2}{\eta_\infty} \left( 1 - \frac{\eta_\infty}{\eta_v(\lambda)} \right) \frac{\eta_v(\lambda)}{G_s(\lambda)} \quad (2)$$

and

$$\theta_2 = \left( 1 - \frac{\eta_\infty}{\eta_v(\lambda)} \right) \frac{\eta_\infty}{G_s(\lambda)} \quad (3)$$

The viscosity  $\eta_v(\lambda)$  and shear modulus  $G_s(\lambda)$  parameters both follow complex exponential dependencies on the structural parameter  $\lambda$ ,

$$G_s(\lambda) = G_0 e^{m \left( \frac{1}{\lambda} - \frac{1}{\lambda_0} \right)} \quad (4)$$

and

$$\eta_v(\lambda) = \eta_\infty e^\lambda \quad (5)$$

In these relations,  $\eta_\infty$  denotes the infinite-shear-rate viscosity, and  $G_0$  is the maximum shear modulus, representing a fully structured fluid. The parameter  $m$  is a phenomenological coupling constant between the thixotropic response of the viscosity  $\eta_v(\lambda)$  and the thixotropic response of the shear modulus  $G_s(\lambda)$ . The structural parameter  $\lambda$  varies with time according to the relation,

$$\frac{d\lambda}{dt} = \frac{1}{\tau_{eq}} \left[ \left( \frac{1}{\lambda} - \frac{1}{\lambda_0} \right)^a - \left( \frac{\lambda}{\lambda_{eq}(\tau)} \right)^b \left( \frac{1}{\lambda_{eq}(\tau)} - \frac{1}{\lambda_0} \right)^a \right] \quad (6)$$

where  $\lambda_{eq}(\tau)$  is the equilibrium structural parameter corresponding to any given applied stress level. The maximum value of the structural parameter,  $\lambda_0$ , representing a fully structured material, also inherently reflects the sharpness of the yielding transition as well as the validity of the yield stress approximation, and  $a$  and  $b$  are empirical parameters. Large values of  $\lambda_0$  correspond to an apparent yielding transition, while a rigorous (theoretical) invariant yield stress threshold corresponds to an infinite value of  $\lambda_0$ .

The hypothesis of the influence of retardation timescale in cuttings removal performance is corroborated by experimental evidence that the elastic modulus  $G_s$  strongly correlates to the performance of a drilling fluid in suspending both rock cuttings and weighting material [7] [8].

Cellulose nanofibrils (CNF) addition to drilling fluids have shown to have effects on the drilling fluid rheological properties [9] [10] [11], although more scientific and complex characterization methods are performed in this research to fully understand the drilling fluid functionality and tailoring capabilities when adding CNF into it.

## EXPERIMENTAL PROCEDURES

### Materials

The materials used in this study were a standard water-based drilling fluid with density of 1.68 g/cm<sup>3</sup> containing KCl, soda ash, polyanionic cellulose, starch, xanthan gum, barite, and a commercial Cellulose Nanofibrils (CNF) which was obtained from Borregaard. CNF was added to the drilling fluid in different w/w percentages.

### Rheological experimentation

To understand the functionalities modified by the addition of CNF into the

drilling fluid, the following three types of rheological characterization were performed, focusing primarily on heavy particle suspension capabilities: flow curve, oscillation strain and 3 interval thixotropy test.

An Anton Paar Physica MCR301 rheometer, has been used for the oscillation and rotational testing, and a grooved Couette geometry (bob and sleeve) was used to avoid slippage. The viscosity profile was obtained from a flow curve, by initially pre-shearing the samples at  $1000\text{ s}^{-1}$ , for 120 seconds to reach steady-state shear viscosity. After this, a rheological measurement protocol was started with a ramp down from  $1200\text{ s}^{-1}$  to  $60\text{ s}^{-1}$  in 100 linear steps, then 5 linear steps from  $60\text{ s}^{-1}$  to  $10\text{ s}^{-1}$ , and finally with 100 linear steps from  $10\text{ s}^{-1}$  to  $0,1\text{ s}^{-1}$ . The measuring time per point was set to 2 s.

To describe the static yield strength and viscoelastic behavior, oscillatory tests were performed. The frequency was set to 1 Hz, increasing amplitude logarithmically from 0.001 to 100% strain with 60 measuring points. The storage modulus ( $G'$ ) and loss modulus ( $G''$ ) curves characterize the materials elastic and viscous behaviors, respectively. When  $G' > G''$ , the elastic behavior dominates over the viscous behavior, thus the drilling fluid shows a solid-like character. The  $G''$  to  $G'$  ratio gives a measure of the stiffness of the material and defines the gel property of the fluid sample. In the case where  $G'' > G'$ , the viscous behavior dominates over the elastic behavior and the sample acts as liquid-like. The cross-over point ( $G' = G''$ ), is called the flow point. The length of the linear viscoelastic range (LVER) indicates the minimum strain to initiate breakage of the inner structure, identifies the static yield strength, as it is calculated as the stress at which deviation from linearity reaches 10%, and also it helps to determine the strain value for the 3-interval-thixotropy tests.

Thixotropy was analyzed by the 3-interval thixotropy test (3iTT) and the following

protocols were applied: first a rest interval at a strain of 0.1% with a frequency of 10 Hz, taking 10 measuring points, and a measuring point time of 20 s, so that the sample can reach the steady state. This is followed by a load interval with a constant shear rate of  $10\text{ s}^{-1}$  with 10 measuring points and measuring point time of 0.1 s, to generate a structural deformation on the sample. Finally, a recovery interval at a strain of 0.1% of 1000 measuring points, with a measuring point time of 10 s and a frequency of 10 Hz, to observe the sample regenerating capability.

All tests were performed at  $25^{\circ}\text{C}$ ,  $30^{\circ}\text{C}$  and  $50^{\circ}\text{C}$ , to help describe better the CNF effect and the relationship of these effects with temperature.

## RESULTS AND DISCUSSION

The effect of CNF addition on the drilling fluids viscosity profile was analyzed using flow curves and the results for different CNF concentrations and temperatures are presented in Fig. 1(a), (b) and (c). As it was expected, with increasing temperature [12] from  $25^{\circ}\text{C}$  to  $50^{\circ}\text{C}$ , the shear deformation spectrum decreased for all the samples. Despite the temperature effect on the shear spectrum, it is possible to observe that the addition of CNF into the drilling fluids generated an increment in the variable shear profile for all temperatures.

Even though the drilling fluids without CNF and with 1% CNF addition showed similar flow curve, the formulation with 1% CNF addition exhibited higher shear stress response particularly for temperatures of  $30^{\circ}\text{C}$  and  $50^{\circ}\text{C}$ . In the case for the drilling fluid with 2,3% CNF concentration, the shear stress spectrum increased significantly, which could be explained by stronger network interaction provided by the CNF [13]. It is important to recognize that the high increment of viscosity due to the addition of 2,3% CNF would cause a significant increase in the Equivalent Circulating Density during field operations, thus could result in well control issues.

For this study the dynamic yield strength calculation derived from non-linear models such as Bingham Plastic or Hershel-Bulkley were not used as it is an extrapolated value based on data points obtained in the mid-to-high shearing rate regime.

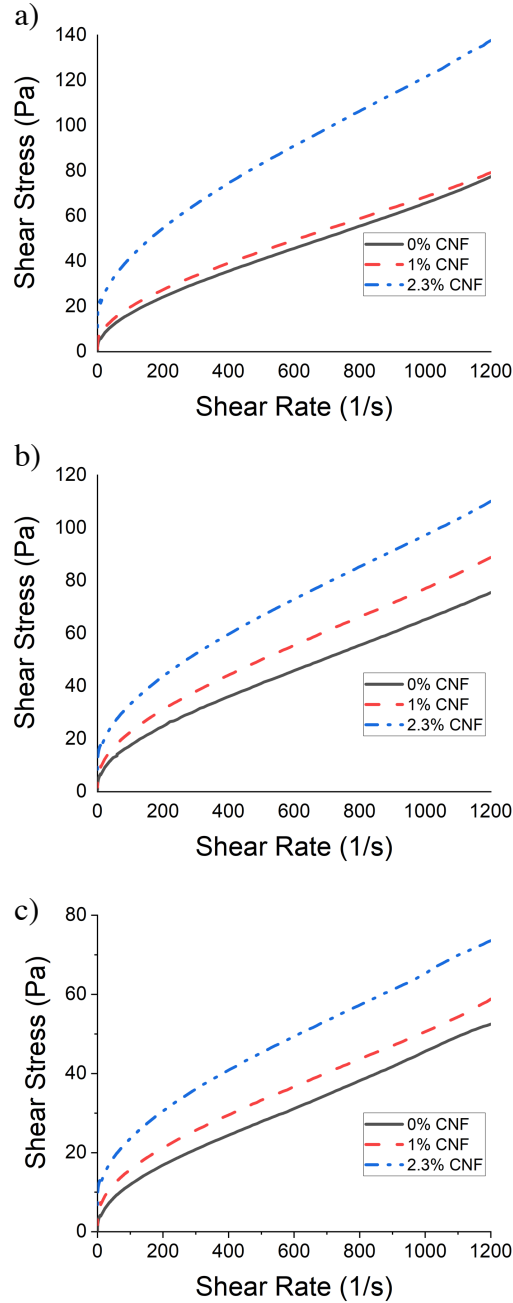


Figure 1. Drilling fluids shear flow profile with 0, 1, and 2.3 wt% of CNF at different temperatures (a) 25°C, (b) 30°C and (c) 50°C.

The viscoelastic behavior was analyzed through oscillation measurements, showing a strain thinning behavior caused by chain orientation or alignment of microstructures along the flow direction [14].

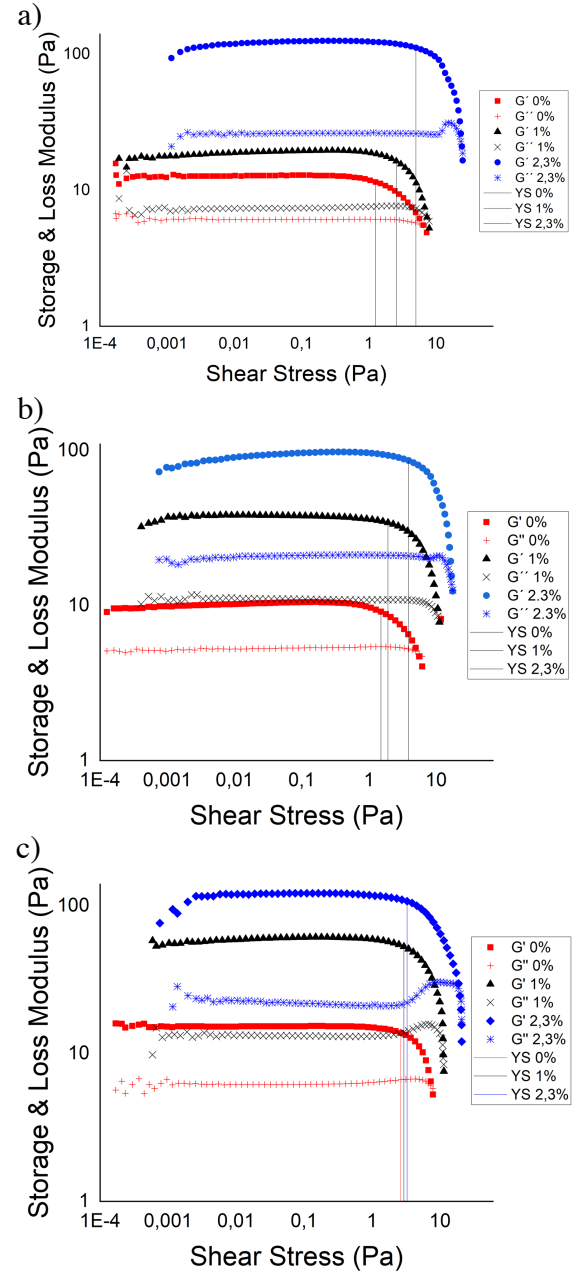


Figure 2. Drilling fluids storage modulus ( $G'$ ) and loss ( $G''$ ) modulus, and yield strength (YS) with 0, 1, and 2.3 wt% of CNF at different temperatures (a) 25°C, (b) 30°C and (c) 50°C.

It is possible to observe increments in the elastic value, the LVER and the static yield strength value (YS) as CNF concentration increases, as presented in Fig. 2. This indicates that due to CNF addition, the drilling fluid acts more solid-like, and higher energy is needed to swift it from solid-like to liquid-like behavior, which could imply enhanced heavy particle suspensions capability in quiescent state.

With the results obtained from oscillation, it is possible to observe that temperature has no significant effect on the drilling fluid elastic modulus. In contrast, the addition of CNF enhances the elastic modulus and the drilling fluid static yield strength for all temperatures. This leads to hypothesize that the long persistence length of CNF nanofibrils reduces the effective retardation timescale  $\theta_2$  and as such will be able to improve material suspension at static conditions.

Another observation is that the yield zone (between the LVER and the flow point) shows a slight peak in  $G''$  curve for the fluid samples with 2.3 wt% CNT. This can be interpreted that the structural network of the fluid sample does not collapse suddenly in the whole shear gap if the LVER has been exceeded. It begins with the forming of micro-cracks which grow into macro-cracks until the  $G''$ -peak is exceeded, and finally a large crack divided the entire shear gap [15].

The 3 interval thixotropy test (see Fig. 3 (a), (b) and (c)), shows the material deformation after the load interval, at which the samples were subjected to a higher shear. At the recovery interval it was observed that the samples without any CNF addition showed structural deformation, remaining stable with little, to no regeneration during the recovery interval. This can be explained by breakage of intramolecular forces due to forces generated during higher shearing [16].

On the other hand, the samples with 1% CNF showed some thixotropic recovery at 30°C and a higher structural build-up at 50°C. The samples with 2,3% CNF presented

structure breakage at 25°C, but at 30°C and 50°C it enhanced its storage modulus. This could be explained by the presence of a large number of hydroxyl groups on the CNF, thus enhancing their interaction via hydrogen bonding [17], other than hydrophobic bonds which are known as weak bonds that can be broken easily due to shearing [18].

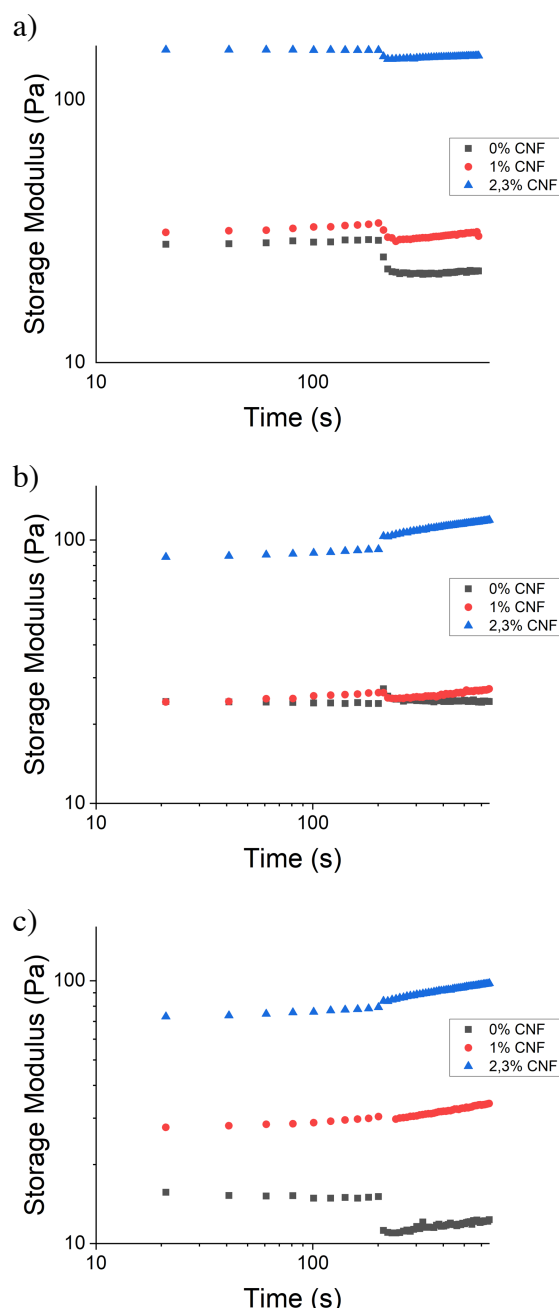


Figure 3. Drilling fluids thixotropy test with 0, 1, and 2.3 wt% of CNF at the different temperatures (a) 25°C, (b) 30°C and (c) 50°C.

These results also indicate that the rest time for drilling fluids with higher CNF concentrations is not enough for the internal structure to fully build, thus requiring more time to reach steady state.

Storage modulus during the first step reached steady state within the step time for the samples with 0% and 1% CNF addition for all temperatures, but 2,3% CNF addition at temperatures of 30°C and 50°C could not reach steady state during the rest interval and continues to build up the internal structure after the load is applied. This demonstrates that it is not only CNF addition, but also the combination with temperature plays an important role in the deformation characteristics [19].

## CONCLUSION

The highly entangled structure of CNF causes a 3D network that increases the resistance to flow, which could explain the shear stress increment during variable shearing flow, and also the necessity of extended periods of time for the 2.3% CNF concentration in drilling fluids to reach steady state during the 3 interval thixotropy test and was not affected by the higher shear applied during the same experiment.

CNF addition to drilling fluids improves certain fluid properties, such as the elastic modulus during quiescent state, becoming a great possibility to enhance heavy particle suspensions to help with the drilling cuttings removal and to avoid barite sagging.

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