# Effect of Preconditioning and Ageing on Rheological Properties of Model Drilling Fluids

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### ABSTRACT

Many drilling fluids are thixotropic, meaning that viscosity decreases with time when subjected to shear. Other fluids may show anti-thixotropic (rheopectic) behavior, i.e. viscosity increase with time when subjected to shear. In both cases it is important to have a consistent procedure for how to treat the fluids prior to rheological measurements. This is vital in order to be able to compare measurements of different samples, with different instruments and between different laboratories.

In the oil industry, the ISO 10416/ISO 10414-1/2 standards are used for determination of viscosity and gel strength of direct-indicating of fluids by use (Fann 35 viscometers). viscometers However, these standards do not specify in detail how the fluids should be pre-treated before measurements. Further, if results from Fann 35 viscometers are to be compared to measurements done on a rheometer, it is even more important to have a consistent pre-treatment of the fluids before the measurements.

A systematic study is performed in which the effects of pre-shearing and ageing history on the rheological measurements of model drilling fluids are investigated by using a Fann 35 viscometer and an Anton Paar MCR 302 rheometer.

The results demonstrate the importance of consistent preconditioning of fluids before measurements. A test-procedure standard is proposed, enabling higher measurement precision and comparability of rheological measurements.

## INTRODUCTION

Thixotropy is defined as the decrease with time of viscosity under constant shear rate or shear stress, followed by a gradual recovery when the sehear rate or shear stress is removed<sup>1</sup>. Thixotropy is thus a reversible property, and many oil-field drilling fluids exhibit thixotropic properties as they are designed to be shear-thinning. However, rheological properties of drilling fluids may also change irreversibly over time due to degradation of the fluids over time due to pressure, temperature, shear history etc, and due to contamination with other fluids, particles and chemicals.

It is therefore important to define consistent measurement methods, which can distinguish between viscosity changes caused by these different effects.

Thixotropy originates from the microstructure of these complex fluids, and the state of this microstructure is dependent on the history of the fluid (shear stress/rate, pressure, temperature, etc). In order to be able to make reproducible experiments it is therefore necessary to recover the original microstructural state of the fluid. Different procedures have been defined in order to achieve this. A typical procedure is to preshear the fluid at some high shear rate immediately before the measurement. sometimes after a specified waiting time.

Aqueous solutions of laponite are good substances to research this problem with, as they show similar characteristics to water-based drilling fluids. They are ageing, that means their relaxation time increases with time, shear thinning, and show thixotropic behaviour<sup>2</sup>.

In a previous paper Assembayev<sup>3</sup> et al. studied the effect of preshearing and waiting time on Fann 35 and Anton Paar measurement results for an oil-based drilling fluid. It was found that with preshear at  $1020 \text{ s}^{-1}$  for 10 minutes the results were significantly less dependent on the prior history (waiting time) than without preshear for waiting times of about 8 hours or less.

In this paper we present and interpret experimental results from Fann 35 and Anton Paar measurements on different water-based model fluids.

The purpose of the paper is to investigate the impact of pre-treatment (preshear and waiting time) for different water-based model fluids, and how this is affected by salinity, temperature and addition of polymer.

## FLUIDS

Five different fluid compositions have been tested. All fluids are aqueous solutions of Laponite RD. Laponite is a synthetic clay with disc-like particles. Its chemical formula is  $Na^+_{0.7}[(Si_8Mg_{5.5}Li_{0.3})O_{20}(OH)_4]^-_{0.7}$ . A negative surface charge is generated from a substitution of magnesium atoms by lithium atoms, to counterbalance the positive charge of the sodium ions<sup>4</sup>. In aqueous media, the sodium dissociates, leading to a negative charge on the surface<sup>5</sup>. Aqueous suspensions of laponite usually show a rich variety of physical behaviour.

The fluid-mixing procedure started with hydrating the laponite in deionized water for 24 h. Afterwards xanthan gum, NaCl, NaOH and biocide were added in varying concentrations, as shown in Table 1 together with the pH and conductivity values.

## EXPERIMENTAL

Measurements were conducted using a Fann 35 viscometer and an Anton Paar MRC 302 rheometer, using a concentric cylinder configuration. (CC27) The rheometer is equipped with a peltier element heat the sample to the desired to temperature. The Anton Paar measurement procedure was as follows: The fluids were sheared for two minutes in a Waring blender at low speed and left to rest for one hour to assure an equal starting point for each fluid. For experiments with preshear the sample was placed in the measuring cup and sheared for 2 min at 1020 1/s (600 rpm in Fann 35 viscometer). Immediately after the preshear interval. the flow curve measurement was started with a decreasing shear rate from 1020 1/s to 1 1/s. Tests without preshear were conducted in the same way, but without a preshear interval. The procedure was repeated after a waiting time of 24 hours. The Fann 35 measurements follow the ISO 10414-1 standard<sup>6</sup>. Six measurements were taken at distinct rotational speeds of 600, 300, 200, 100, 6, and 3 rpm.

# RESULTS AND DISCUSSION

Fann 35 measurements

Generally, the fluids show shear thinning trends, and experiments with preshear and without waiting time led to the lowest The viscosities. biggest difference in viscosity can be seen between the measurements without waiting time and a waiting time of 24 h, especially for fluids #1a and #2a. Small amounts of salt increase the viscosity, this applies both with and without xanthan gum present in the composition, see Figure 1 - Figure 4.

A high salt concentration of 12 g/l NaCl reduced the viscosity of sample #2c, compared to #2b (Figure 5).

Figure 1 - Figure 4 show that the preshearing procedure is not sufficient to obtain results independent of waiting time for fluids #1 and #2. However, preshearing

is much more efficient when 0.6 g/l NaCl is added, as seen in Figure 2 and Figure 4. The measurements with fluid #1a and #1b were also conducted at 50 °C and show the same qualitative trends as at 24 °C.



Figure 1. Fann 35 measurements for fluid #1a (water-laponite without salt) at 24 °C.







Figure 3. Fann 35 measurements for fluid #2a (water-laponite-xanthan gum without salt) at 24 °C.



Figure 4. Fann 35 measurements for fluid #2b (water-laponite-xanthan gum with 0.6 g/l NaCl) at 24 °C.



Figure 5. Fann 35 measurements for fluid #2c (water-laponite-xanthan gum with 12 g/l NaCl) at 24 °C.

Interestingly, the experiments with fluid #2, which include xanthan gum, show similar trends as fluids #1. For fluids without salt and measurements without preshear the viscosity is much lower.

As expected, preshear does not have much effect on the result with 0 hr waiting time.

### Anton Paar measurements

The observations made from the Fann measurements are confirmed by the flow curves generated from Anton Paar measurements.

The flow curves measured with the Anton Paar rheometer show apparent shear thickening trends in the samples without preshear for shear rates >800 1/s (Figure 7). This trend is more pronounced for samples

with a waiting time of 24 h and higher NaCl concentrations and especially predominant for fluid #2c, which contains xanthan gum and a high concentration of salt. This effect might be a result of the thixotropic behaviour, because the measurements start at high shear rate and are affected by a shear history.

In Figure 6 the curves without waiting time show a kink at a shear rate of about 450 1/s. The kink was observed regardless of measuring direction and at 50°C. We do not have a consistent explanation, because we see it only in the Anton Paar measurements and not in the Fann 35 measurements. The Taylor number for this case was well below the critical Taylor number, see Equation 1, where  $\omega$  is the rotational speed, b the radius of the bob, a the radius of the cup and v the kinematic viscosity.

$$Ta = \frac{\omega^2 b (a-b)^3}{v^2} \tag{1}$$

Figure 8 shows a comparison of Fann 35 and Anton Paar measurements of fluid #1a at 24 °C. The results show a difference for the measurements without waiting time. The shear stress of the Fann 35 measurement is much below the shear stress value of the Anton Paar measurement at high shear rate. It is possible that this can be a geometry effect, although we then would expect to see this in other measurements as well.



Figure 6. Anton Paar flow curve measurements for fluid #1a (water-laponite without salt) at 24 °C.



Figure 7. Anton Paar flow curves of fluid #2c (water-laponite with salt and xanthan gum) at 24 °C.



Figure 8. Comparison of Fann 35 and Anton Paar measurements for fluid #1a (waterlaponite without salt) at 24 °C.

### CONCLUSION

Preshearing a thixotropic sample before the measurement flow of curves is recommended to achieve more reproducible results. Preshearing is not recommended when measuring structural properties, as preshearing will reduce or destroy the microstructure in the fluid. The findings work as a methodical guideline for rheological characterization of water-based model fluids. The effects of waiting time and/or preshear are significant and will influence the quality of the results. Salt affects the influence of preshear, both with and without xanthan gum. Preshear is much more effective with a small quantity of NaCl than without NaCl. The effects of concentration of salt on viscosity and on the effect of preshear is strongly nonlinear, however we have only investigated two different salt concentrations. Xanthan gum alone does not have this effect. We see the same qualitative trends for 50 °C as for 24 °C. An anomaly was found in the Anton Paar measurements with a laponite fluid without salt. This was consistent but not seen in the Fann 35 measurements. Also the Taylor number is well below the critical Taylor number for this fluid.

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|                       | -               | 1     | 2     |       |      |
|-----------------------|-----------------|-------|-------|-------|------|
| Component             | а               | b     | а     | b     | с    |
| Water                 | Deionized water |       |       |       |      |
| Laponite RD<br>[wt %] | 1.5             | 1.5   | 1.5   | 1.5   | 1.5  |
| Xanthan Gum<br>[wt %] | 0               | 0     | 0.1   | 0.1   | 0.1  |
| NaCl [g/l]            | 0               | 0.6   | 0     | 0.6   | 12   |
| NaOH [mmol/l]         | 0.1             | 0.1   | 0.1   | 0.1   | 0.1  |
| Biocide               | 0               | 0     | 0.1   | 0.1   | 0.1  |
| pH value              | 10.07           | 10.31 | 10.51 | 10.21 | 9.31 |
| Conductivity<br>[mS]  | 0.576           | 1.525 | 0.546 | 1.382 | 17.7 |

Table 1 Fluid composition.