Rheological Measurements of Cementitious Suspensions Using a Grooved Measuring Device

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ABSTRACT

When measuring the rheology of concentrated suspensions using a concentric cylindrical configuration, having a smooth surface, the results have been found to be prone to slippage. By using a modified assembly, having the same dimensions, but with a grooved surface we have shown that the appearance of slippage during measurements has been reduced to a negligible level. However, the amount of induced shear when using various combinations of rotors and stators has also been found to vary.

INTRODUCTION

In the oil industry, the current practice for the rheological characterisation of fluids is based on rotational viscometry. The API[1] recommends the use of a coaxial cylinder viscometer with cylinders having a smooth surface. The appearance of slip when using a smooth surface for measuring the viscosity of concentrated cementitious suspensions has been identified in some earlier work[2-4]. Slip is the formation of a fluid film on the surface of the measuring device which acts as a lubricator. The result is a reduction in the measured values. Slip is found to be most profound at rather low shear rates. Using data affected by slippage can lead to erroneous conclusions when modelling in order to predict the behaviour of the suspension at low shear rates e.g., when using such data for predicting a yield stress. One way of reducing the influence of slip on the measured values is by using a roughened cylinder surface[5,6], or as in the present work a grooved cylindrical surface. Equipped with two sets of rotors and stators, one having smooth surfaces and the other having grooved surfaces, we were able to measure and compare the viscosity of our samples using four different combinations of rotors and stators.

EXPERIMENTAL CONDITIONS

Sample preparations

For our first measurements we used a Calpar 100 oil. This is a paraffinic base oil delivered by Calumet Speciality Products Partners, L.P. This oil was used as a particle free reference fluid. The Calpar 100 oil is given by the manufacturer to have a typical kinematic viscosity of 19.19 cSt at a temperature of 40°C. This kinematic viscosity is reported to be measured in accordance with ASTM[7]. According to this standard the kinematic viscosity is measured by the use of a glass capillary type viscometer which is intended for application to Newtonian liquids for which the shear stress and shear rates are proportional. The specific density of the Calpar 100 oil was measured to be 0.84026 at a temperature of 40°C. From this a typical dynamic viscosity could be calculated to be 16.77 mPas. We measured the viscosity at 40°C, using the Z3 DIN configuration where both rotor and

stator have a smooth surface, to be 18.2 mPas at a shear rate of $34s^{-1}$.

Further, we used a suspension of bentonite clay. The bentonite disperses readily in fresh water forming a stable suspension not liable to sedimentation. The suspension we used was made by mixing 25g of bentonite with 350g of distilled water. Using a density of 2.5 g/cm^3 for the bentonite gives a suspension having a solid volume fraction of ~0.15. Prior to any measurements the bentonite suspension was allowed to rest for 5 days as to allow for full hydration of the particles. The result is an almost fully dispersed suspension with individual particles of colloidal size.

For our final measurements we used a neat Class G cement[1] suspension having a solid volume fraction of 0.44 by weight. Using a density of 3.25 g/cm^3 for the cement gives a suspension having a solid volume fraction of ~0.41. The Class G cement has been measured[3] to have an almost log normal particle size distribution with a d50 of 12.6 µm, a d16 of 5.44 µm and a d84 of 29.4 µm.

Density measurements

For density measurements we used a DMA 4500 Density Meter delivered by Anton Paar.

Viscosity measurements

The rheological properties of our suspensions were measured using a Physica UDS 200 rheometer fitted with two different concentric cylindrical configurations. The configurations are shown in Fig. 1. For the configuration having a smooth surface only the rotor is shown. This is a standard concentric configuration named Z3 DIN and appears as it was delivered with the rheometer. Both rotor and stator of the other configuration is shown in Fig. 1. Here parts have been removed resulting in a ribbed or grooved surface. The result is surfaces having alternating ridges and troughs parallel to the axial direction.

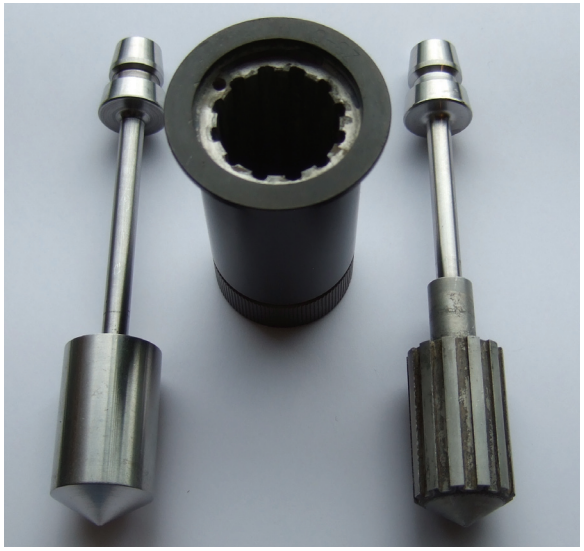

Figure 1. The concentric configurations used for rheological measurements.

On both the rotor and the stator 12 troughs have been made evenly spaced circumferentially. On the rotor the troughs have a width of ~3 mm and a depth of ~1.2 mm. This leaves 12 ridges evenly spaced, of the initially smooth surface, having a width of ~3.5 mm. On the stator the width of the troughs are ~3.5 mm and the depth ~1.5 mm. The width of the ridges on the stator is ~3.6 mm.

All our measurements were carried out at a temperature of 25ºC and in a declining order regarding shear rate starting from a shear rate of 1022 s^{-1} and going down. For all shear rates the period of shear lasted for 20 seconds and all readings used were taken at the end of each period.

RESULTS AND DISCUSSION

Rheological measurements

In Fig. 2 the viscosity values are shown, measured on the Calpar 100 oil using the four combinations of rotors and stators. The measurements started at an upper shear rate of $1022s^{-1}$ and in consecutive order down to a shear rate of $34s^{-1}$. At lower shear rates we were unable to obtain stable values during the 20 second periods at constant shear. From the curves and for all our combinations of rotors and stators we find that the oil is slightly shear-thickening

within the measured interval of shear rates. Further, the curves for the various combinations are found to be almost evenly spaced with respect to viscosity. The highest values were measured using the combination of a rotor and stator both having a smooth surface. Here the viscosities were found to decrease from to 35.3 mPas at a shear rate of $1022s^{-1}$ to 33.5 mPas at a shear rate of $34s^{-1}$. The second highest values were measured for the combination of a smooth rotor and a grooved stator. For this combination the viscosities were found to decrease from 33.4 mPas at a shear rate of $1022s^{-1}$ to 31.4 mPas at a shear rate of $34s^{-1}$. The combination of a grooved rotor and a smooth stator was found to result in the second lowest readings, from 31.3 mPas at a shear rate of $1022s^{-1}$ to 29.7 mPas at a shear rate of $34s^{-1}$. Finally the combination consisting of two grooved surfaces were found to result in the lowest values measured. For this combination the viscosities were found to decrease from 28.5 mPas at a shear rate of $1022s^{-1}$ to 27.4 mPas at a shear rate of $34s^{-1}$.

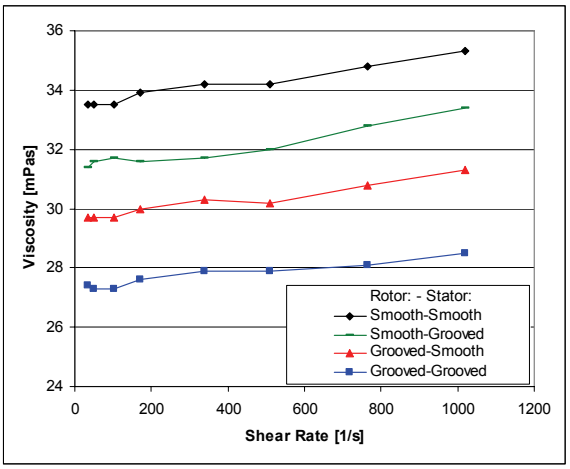

Figure 2. Viscosities of a Calpar 100 oil measured at a temperature of 25ºC using four different concentric combinations of smooth and grooved rotors and stators.

The shear thickening found and the lack of stability reported when measuring at shear rates below $34s^{-1}$ is expected to be caused by polymers added to the oil. Polymers are added in order to maintain the lubricating effect of the oil at higher temperatures[8]. The variation in the measured values for the various cylindrical combinations is expected to reflect the amount of induced shear, i.e. for this oil the combination of two smooth surfaces result in the highest induced shear.

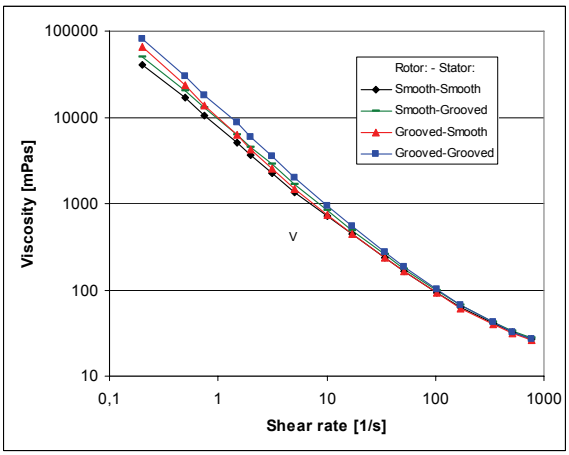

Figure 3. Viscosity of a bentonite suspension measured at 25ºC by using four different concentric combinations of smooth and grooved rotors and stators.

For our bentonite suspensions, being rather dilute, the viscosities measured using the various combinations are shown in Fig. 3. For these suspensions the measurements were also carried out in a declining order with respect to shear rate. The highest shear rates reported here are at $765s^{-1}$ as the values obtained during the 20 second periods of shearing at the initial shear rate of $1022s^{-1}$ were found not to be stable. Stable readings were obtained at the following shear rates of $511s^{-1}$, $340s^{-1}$, and $170s^{-1}$. From a shear rate of $102s^{-1}$ and below, the viscosity started to increase during the 20 second periods at constant shear. It is expected that this build-up in viscosity is due to the phenomenon of thixotropy, as it is known that bentonite forms a thixotropic suspension when mixed with water. So for these shear rates the build-up of viscosity found for our suspensions is expected to be due to an insufficient shear with respect to hinder the forming of a structure in the suspensions. It should also be noted that the various curves showing the build-up of viscosity at low

shear rates are in the opposite order, with respect to viscosity, of that shown in Fig. 2. This we find to confirm our conclusion with respect to the various amounts of shear induced by the different combinations in that a lower amount of induced shear allows for a more rapid build-up of structure. Whether this anticipated build up of structure result in an inhomogeneous suspension could not be said. Nelson and Guillot[9] state that if the experimental data are dependent on gap size the homogeneity of the fluid is questionable. At the highest shear rates no distinct pattern was found. The measured values for the various combinations more or less coincided.

In Fig. 4 the viscosities measured on a cement suspension using the various configurations are shown. Also here the values measured at a shear rate of $1022s^{-1}$ have been omitted as the 20 seconds of shearing at constant shear rate was found to be insufficient for obtaining stable readings. Further, we were unable to obtain stable readings below a shear rate of $0.2s^{-1}$ thus these values have also been omitted.

For these measurements we find that the combination of a grooved rotor and a smooth stator results in the highest viscosities measured for all shear rates except for the lowest at $0.2s^{-1}$. At that point the combination of a grooved rotor and a grooved stator result in the highest viscosity. The lowest viscosity is measured when using the combination of a smooth rotor and a grooved stator. Although at the lowest shear rate of $0.2s^{-1}$, the measured value is the same at that measured when using the combination of both rotor and stator being smooth. When looking at the curves one could expect that at shear rates below $0.2s^{-1}$ the relative placement of the curves with respect to viscosity will be the same as that reported at the lowest shear rates for the bentonite suspension shown in Fig. 3. This could be due to a much slower build-up of structure in the cement suspensions compared to that of the bentonite suspensions.

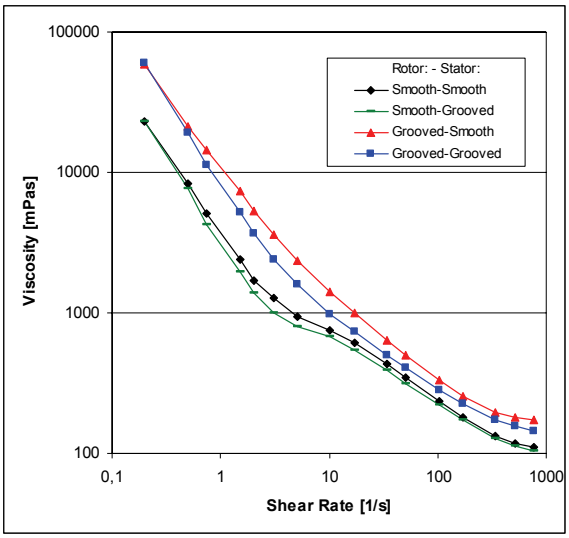

Figure 4. Viscosity of a cement suspension measured at 25ºC by using four different concentric combinations of smooth and grooved rotors and stators.

For this rather concentrated suspension there is also a distinction between the combinations having a grooved rotor and the combinations having a smooth rotor in that the latter combinations show the lowest viscosity values throughout the measured interval. Further, these two lower curves show a slight deviation from a straight line between the shear rates of $34s^{-1}$ and $2s^{-1}$. This is expected to be due to the occurrence of slippage[9]. Slippage at these shear rates has also been observed in some earlier work[3,4] when using a smooth rotor and stator and measuring on cementitious suspensions. Whether the slight deviation observed in the same region of shear rates, for the curve measured using the combination of a grooved rotor and a grooved stator could not be said for sure. Only for the combination of a grooved rotor and a smooth stator we find no sign of slippage occurring. At the highest shear rates the viscosities measured for our cement suspensions varied when using the different cylindrical configurations. This could be due to a variation in the amount of induced shear prior to any measurements, a shear being insufficient to fully disperse the cement particles.

CONCLUSION

When measuring using the various combinations of our rotors and stators we found the amount of induced shear to vary.

The rotors having a smooth surface, seemed to be most influential with respect to inducing the highest amount of shear.

Grooved surfaces allowed for a more rapid build-up of structure.

For our concentrated cement suspension the amount of induced shear was found to be insufficient to fully disperse the particles.

Slippage was found to occur only for our cement suspensions when using a smooth rotor.

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