A device to measure flow behaviour of settling particle slurries

N. Tonmukayakul¹, J.F. Morris^{2#}, R.G. Morgan¹, and R.L. Morgan¹,

¹Halliburton, Duncan Technology Center, Oklahoma, USA 73536

²Levich Institute and Dept of Chemical Engineering, CCNY, New York, NY 10031.

ABSTRACT

Measurement of flow properties of settling suspensions is of importance to the oil service industry. This is particularly true for the hydraulic fracturing process where the rheological influence of particle settling affects the design factors of the fracturing processes. As a practical matter it is of interest to obtain reliable rheological data slurries for conditions for these representative of those found in the fracturing process, including the influence of wellbore travel. A significant problem faced in the design of a suitable rheometer for settling slurries is that the slurries are typically heterogeneous and settling in nature. Many existing rheometers using standard geometries, such as cone and plate, parallel plate, and bob and sleeve are not suitable for measuring the rheological properties of settling slurries; they cannot prevent the particles from settling during experimental measurements. Halliburton has recently developed a high-shear mixing device (HSMD) that has the capability of detecting the flow properties of gel suspensions under similar shear-rate conditions to those found in the fracture path. The HSMD was developed using highshear mixing concepts where the particles suspended throughout are kept the measurement. Due to complexity of flow fields inside the new device, a volumeaveraged shear method is used for calculating the shear-stress and shear-rate values. In this paper, the features of this unique instrument are described and the experimental technique developed for flow property measurement is outlined. Typical

measured flow properties of polymeric suspensions as well as a time-dependent yield stress liquid are presented and discussed to illustrate the potential applicability and limitations of the HSMD. One key limitation under study is related to the observation of time-dependence in the measurements for particle-laden liquids even for neutrally buoyant particles.

INTRODUCTION

The particle-fluid mixtures used for applications fracturing are specially designed to provide proppant placement and to clean up easily after completing the fracturing process. Proppant placement demands supporting the weight of solids of large excess density (with typical proppant), and relies strongly on the viscosity and/or elasticity of the base fluid, whereas cleanup of the fracture requires removing the polymer from the fracture face and proppant pack. Crosslinked polymeric solutions are commonly used as the base fluid for the fracturing mixtures, and the cleanup is easier smaller polymer loading. with The properties of rheological particle-fluid mixtures under hvdraulic fracturing conditions are important in controlling the mobility and placement of the proppant particles. Knowledge of the particle-laden gel rheology is basic to any effort to improve understanding of the mechanism associated with proppant transport efficiency, and may assist in predicting the placement of proppant.

The rheology of the base fluids has been extensively studied and comprehensive reviews have been given.^{1,2} However, this is not the case for the particle-laden liquids: understanding of the phenomena at the

[#] Corresponding author: jeff.morris[morris@ccny.ccny.edu]

particle level in a shear flow of a suspension in a viscoelastic liquid is not welldeveloped. One fact hampering understanding is the lack of a reliable device that can overcome problems of particle settling and particle migration during the rheological measurements and that could be operated under the conditions of interest.

The foregoing should not be interpreted as a lack of activity directed toward solving the problem. A survey of the available literature reveals that the instruments and techniques previously developed for settling slurries are quite diverse in design and method of operation. The rotating concentric cylinder geometry with the addition of an axial flow to lift the settling particles is probably the most widely used measuring system. This is due to its apparent simplicity in construction and operation. The system has been used by many workers³⁻⁹ for the viscosity measurements of mineral slurries. Other types of measuring geometries have been used such as a vibrating sphere viscometer¹⁰ and a modified parallel plate.¹¹ Almost all of the existing instruments are designed to operate at relatively low pressure in comparison to those found in the fracturing process, which typically ranges from 1,000 to 4,000 psi. While the pressure itself may not be a major factor, it is necessary to maintain the liquid state at the elevated temperatures found in fracturing applications at typical depths. This major limitation of the existing instruments makes them unsuitable for flow behaviour measurement of fracturing fluids under the conditions of interest in the present work.

This work reports development of a new device¹² designed by Halliburton for flow behaviour of settling measuring suspensions. The new device was developed using high-shear mixing concepts where the particles are kept suspended throughout the measurement. Due to complexity of flow fields inside the new device, a volumeaverage shear method is used for calculating the shear stress and shear rate values. This

technique was first mentioned by Metzner and Otto (1957)¹³ and Metzner and Taylor $(1960)^{14}$ and further developed and utilized by Steffe (1992)¹⁵ for industrial applications. The volume-average shear technique involves first measuring the viscosities of standard Newtonian and power law fluids using the HSMD, and second comparing and adjusting the viscosity data to match to the data obtained from a rheometer equipped with a standard measuring geometry. The method has been widely used in the food, plastic, and oil industries for rheological measurements of complex fluids.^{16,17} In this work, experimental results obtained for different fracturing suspensions having a solid volume fraction, ϕ , ranging from 0.2 to are presented and discussed 0.5 to demonstrate the suitability of the device as a practical instrument.

THE APPARATUS

The high-temperature and high-pressure (HTHP) viscometer constructed in this work has been designed for measurement of the rheological properties of particle-laden samples under a controlled environment at temperatures between 4°C and 150°C and at pressures up to 1,000 psi. The ranges of operating conditions are similar to those of the hydraulic fracturing processes. А schematic diagram of the HSMD is given in Figure 1. The device is designed on the Couette principle for rheological measurements; in this case, we shear a volume of sample contained in a gap between a stator and a blade located along the inside wall of the plastic cup. The stator is attached directly to the torque device of a Brookfield pressurized viscometer (PVS-Viscometer) unit and stays static during the rheological measurement. The plastic cup and the blade are attached to the PVS-Viscometer motor and rotate constantly at a given speed.

In this work, a volume-averaged shear method is used for calculating shear rate $(\bar{\gamma}_{vol})$ and shear stress $(\bar{\tau}_{vol})$. This is due to

the complexity of the flow fields inside the HSMD. The volume-averaged shear method has been developed based on the assumption that "the average shear rate for a non-Newtonian fluid is equal to the average shear rate for a Newtonian fluid when the Newtonian viscosity equals the apparent viscosity of the non-Newtonian fluid."15 Values of $\dot{\gamma}_{\rm vol}$ and $\overline{\tau}_{vol}$ require determination of the constants used for adjusting the viscosity values obtained from the device of complex geometry to match with those generated by a viscometer equipped with a standard measuring geometry. Note here that values of the constant used to calculate $\overline{\dot{\gamma}}_{vol}$ and $\overline{\tau}_{vol}$ are independent of the rotational speed of the device, and in principle of the size although we have yet to verify this. From matching and adjusting experiments, with silicon-oils having viscosity of 5×10^5 and 1×10^6 Pa.s. and two power-law fluids of known viscosity dependence upon rate, the formula used for calculating $\overline{\dot{\gamma}}_{vol}$ (in s⁻¹) and $\overline{\tau}_{vol}$ (in Pa) are given in Equations 1 and 2, respectively.

$$\overline{\dot{\gamma}}_{\rm vol} = \mathbf{k}_1 \times \mathbf{\Omega} \tag{1}$$

 $\overline{\tau}_{vol} = k_2 \times M \tag{2}$

where Ω is the rotor angular velocity in rad/s and M is the torque acting on the vane surfaces in N-m; k₁ and k₂ are the mixer viscometer constants having dimensions rad⁻¹ and m⁻³. For our high-temperature, high-pressure (HTHP) viscometer, we found k₁ = 0.36 and k₂ = 0.20.



Figure 1. Schematic diagram of the HSMD.¹²

EXPERIMENTAL PROCEDURES AND TECHNIQUES

Rheological measurements on particleladen suspensions were carried out using the following procedure. First, the base gel and sand particles were mixed together in a laboratory blender for a minimum 10-minute period prior to loading the mixture into the test volume of the device. For room temperature measurement, the experiment was performed after zeroing the torque value of the PVS-Rheometer on which the device is mounted. The rheological experiment was commenced by rotating the cup at a high speed (about 800 RPM or at $\overline{\dot{\gamma}}_{vol} = 288 \text{ s}^{-1}$) and measuring the torque acting on the device's blades. The rotational speed was then decreased in steps and the torque response at each constant speed recorded. After each test run, the fracturing sample was removed and discarded.



Figure 2. Testing results of the HSM device with linear guar CMHPG solutions at room temperature.

TESTING OF THE HSMD

It was necessary to test the newly developed HTHP device for its accuracy and reliability using Newtonian fluids of known viscosities. The device was also tested with known viscosity polymeric fluids (carboxymethyl hydroxypropyl guar {CMHPG} solution and cross-linked hydroxypropyl guar {HPG} gel) and tomato ketchup, which is a time-dependent yield stress fluid. Measurements from the HTHP device were compared with those obtained from Chandler-3500, HAAKE RS150 and Reologica StressTech rheometers, each equipped with a narrow gap bob-cup system.

Figure 2 shows the typical results obtained for a CMHPG solution on a logarithmic scale of volume average viscosity ($\overline{\eta}_{vol}$) versus $\overline{\dot{\gamma}}_{vol}$ plots. Over the shear-rate range tested, $\overline{\eta}_{vol}$ decreases linearly as shear rate increases, indicating shear-thinning behaviour of the material. At $\overline{\dot{\gamma}}_{vol} \approx 50 \text{s}^{-1}$, however, $\overline{\eta}_{vol}$ increases as shear rate increases. Based on the impeller Reynolds number (N_{Re,I}), we found that at $\overline{\dot{\gamma}}_{vol} \approx 50 \text{s}^{-1}$ the samples are probably in turbulent motion. Overall, it may be seen that the agreement between the viscosity

data obtained using the Halliburton HSMD and the commercial rheometers is very good within the shear rate ranging from 5 to 30 s⁻¹

In addition, cross-linked HPG solutions having pH 8.1 and 8.15, for which viscosity data are available in the literature,¹⁸ have also been prepared and tested using the HES device. Figure. 3 shows the comparison between the data obtained using the new device and these literature data. It was found that the agreement is within a maximum error of $\pm 11\%$. Fig. 3 also shows the comparison of the new device and the HAAKE rheometer equipped with a narrow gap bob-cup for a tomato ketchup sample. Again, а good agreement of the experimental data has been observed indicating the reliability of the device for measuring flow properties of power-law and time-dependent yield stress materials.



Figure 3. Testing results of the Halliburton HSM device with cross-linked HPG solutions and tomato ketchup at room temperature.

EXPERIMENTAL RESULTS

Flow characteristics of sand suspended in a 500-cp Newtonian fluid

Figure 4 shows measurement data from the 500 cp standard Newtonian silicon oil with sand having an average particle size of roughly 400 µm. In this work, the maximum volume fraction of particles (ϕ) tested was at 0.576, which is in the neighbourhood of the concentration of the fracturing sample after fluid loss occurs. The maximum shear rate value of the sample having concentration higher than 0.45 was restricted to 15 s⁻¹; at higher shear rate, the torque acting on the device blades exceeded the measuring range of the PVS. Moreover, it was found that the optimum shearing time was at 5 minutes. Figure 4 reveals that at $\phi = 0.083$, the flow behaviour of the sample is similar to the base fluid (without particles). However, for the samples having, $\phi \ge 0.18$, non-Newtonian behaviour was observed as $\overline{\eta}_{vol}$ decreases with increasing shear rate. Also, it appears that the samples may exhibit apparent yield stress behaviour.



Figure 4. Viscosity data of the mixture of sand in a 500-cp Newtonian fluid tested at room temperature at various solid volume fractions, ϕ .

Before considering the results obtained in characterizing the new device, we note some expectations. For neutrally buoyant suspensions in Newtonian fluids, non-Newtonian rheological behaviour observed include shear thinning, shear thickening and the presence of normal stress differences.¹⁹ Here we are concerned with suspensions of particles which are sufficiently large that

colloidal Brownian forces and are negligible, and in the Stokes flow limit these suspensions are expected theoretically to display a constant viscosity. This is, however, not generally observed, as weak shear thinning is often reported for such suspensions (i.e., in Newtonian liquids) for large volume fraction,²⁰ $\phi > 0.4$. This suggests surface contact plays a role in such suspensions. For settling particle suspensions, there will always be some nonuniformity of ϕ in the vertical direction, and this will lead to both time- and shear rate dependence: the magnitude of this nonuniformity is expected to scale with the Shields parameter (here this may be taken as Sh = $\overline{\tau}_{vol} / \Delta \rho \gamma d$ for a density difference $\Delta \rho$ between particles and liquid and a particle of linear size d). As seen in Figure 4, the mixtures of sand in a Newtonian fluid show a thinning at all but the smallest solids loadings. Interestingly, while the problems studied are not identical, of the reports on the effects of particle-particle interaction on rheological behaviour, those on packing fraction versus particle size distribution²¹ most satisfactorily describe the onset of solids-fraction dependence of the shear-rate dependent behaviour found for heavy particles in this work. The cited work found the minimum concentration required before particle interaction took effect is 0.127 which agrees with our observation of thinning above $\phi = 0.1$. In general, suspensions which show significant shear thinning in neutrally buoyant conditions also exhibit normal stress differences and hence migration²¹ shear-induced and this phenomenon may play some role here, particularly in moving particles away from the sensing surfaces (vanes and walls).

Flow characteristics of sand suspended in a linear CMHPG solution

Figure 5 shows the viscosity data of sand suspended in a linear CMHPG solution. The CHMPG solution had the polymer concentration at 80 lb/Mgal (Mgal = 1000 gallons), and sand concentrations of $\phi = 0.08$ to 0.52. As shown in Figure 5, the apparent viscosity of the sand-CMHPG mixtures is not constant but decreases dramatically with increasing shear rate. At any given shear rate, the mixture viscosity increases with increasing solid concentration. А comparison of the results at a given solid concentration and shear rate between the 500 cp oil and CMHPG solution shows that the mixture of 500 cp oil is more viscous and has a higher apparent yield stress than the CMHPG mixture. This is caused by the difference in the viscosity of the base fluid and has direct effects on the sand transport ability. Further study is required in this area.



Figure 5. Viscosity data of the mixture of sand in a CMHPG solution tested at room temperature.

The steady rheological data are fairly well described by the Carreau model especially at $\overline{\dot{\gamma}}_{vol} > 10s^{-1}$. The Carreau model

is
$$\eta = \left(\frac{\eta_0 - \eta_\infty}{\left(1 + (\lambda \dot{\gamma})^2\right)^{\alpha}}\right) + \eta_\infty^{22}$$
. For $\overline{\dot{\gamma}}_{vol} < 10s^{-1}$,

however, the model fails to describe the rheological data, possibly due to the effects of particle settling and/or particle-particle interaction on rheological properties and measurements. It may also be that the effects of particle settling and particle-particle interaction may cause the appearance of the yield stress behaviour of the mixture in this test, and yield stress is not captured in the Carreau model.

PRACTICAL CAPABILITIES AND LIMITATIONS

This work has analyzed a new device designed for measuring flow behaviour of particle-laden fluids. The relative merits of the device were judged based on its ability to measure the rheology, in the sense of a volume average shear response, of Newtonian, non-Newtonian and time dependent yield stress materials. The work has shown that the rheological behaviour of a typical complex industrial material can be characterized and understood by means of the instrument and technique developed. The experimental data obtained can be interpreted within the framework of general suspension rheology.

It is important to note here that interpretation of the rheological data obtained from the HES device must be made with care. This is due to the fact that this device has been designed based on the high shear mixing concepts, and the mathematics needed to describe flow fields inside the device have not been fully developed. As a consequence, the experimental data obtained are only qualitative. Nevertheless, we believe that our newly developed device could be used as a practical tool for screening, developing, and monitoring flow behaviour of particle-laden fracturing fluids under conditions similar to actual fracturing processes.

However, there is significant timedependence observed in the data for particleladen liquids, a point not discussed at length here but still under study. For highly elastic liquids, whether bearing solids or not, the device is presently being studied further, because we observe that the "constants" in Equations 1 and 2 depend on the level of elasticity (meaning the cross-linking density, pH, polymer loading and other variables).

CONCLUSIONS

The device developed in this work is unique in that it is designed based on highshear mixing concepts for measuring rheological behaviour of particle-laden fracturing fluids under conditions which, as previously noted, are similar to those found in field fracturing processes. Extensive testing of the device using standard Newtonian and fluids of known rheological properties has established the suitability and reliability of the device for measuring apparent rheological properties. Results obtained from a 500-cp Newtonian fluid and linear CMHPG solution indicated that the "non-Newtonian" samples exhibit behaviour, in the sense that the volumeaveraged shear stress divided by the volume averaged shear rate is rate dependent, when solid fraction exceeds $\phi = 0.18$. For neutrally buoyant suspensions, this is surprising and implies the need to consider known such phenomena as shear-induced migration,²¹ although this has not been proven as the source of the behaviour. The effects of particle-particle interaction on rheological behaviour found in work on packing fraction versus particle size distribution is found to be the most suitable for describing the non-Newtonian behaviour found in this work. Highly elastic crosslinked suspending fluids have not been characterized satisfactorily in the device; this is a topic still under study.

ACKNOWLEDGEMENTS

The authors thank the management of Halliburton for support of this project and the opportunity to present this paper. We acknowledge the individual contributions of Johnny Johnson, Ian Robb, Malcolm Talbot and Jason Bryant.

REFERENCES

1. Gidley, J.L., Holditch, S.A., Nierode, D.E. and Veatch, R.W., (2001), 'Recent advances in hydraulic fracturing', SPE, Texas, pp. 24-25.

- Economides, M.J., Watters, L.T. and Dunn-Norman, S., (1998), 'Petroleum well construction'. John Wiley & Sons, New York, pp. 471-479.
- Bhattacharya, S.N., Chryss, A., Connell, H.J. and Shepard, J.J., (1990), 'Rotational rheometer for settling multiphase mixtures', 5th Nat. Conf. Rheol., Melbourne, pp. 15-18.
- 4. Blaszczykl, J. and Petela, R., (1986), 'Application of a modified rotary rheometer to the investigation of slurries', Rheol.Acta, 25, pp. 521-526.
- Ferrini, F., Ercolani, D., de Cindio, B., Nicodemo, L., Nicolais, L. and Ranaudo, S., (1979), 'Shear viscosity of settling slurries' Rheol.Acta, 18, pp. 289-296.
- Reeves, T.J., (1985) 'On-line viscometer for mineral slurries' Trans.I.M.M., 94, pp. 201-208.
- 7. Shi, F.N. and Napier-Munn, T.J., (1996), 'Measuring the rheology of slurries using an on-line viscometer', Int.J.Miner.Process., 47, pp. 153-176.
- Nguyen, Q.D., Devasagayam, C. and Bown, D.J., (2000), 'Development of an on-line flow rheometer', Mineral Processing and Extractive Metallurgy Review, 20, pp. 75-91.
- 9. Akroyd, T.J., (2004), 'Continuous flow rheometry for settling slurries', Ph.D. Thesis. The University of Adelaide, Australian.
- Kawatra, S.K. and Bakshi, A.K., (1996), 'On-line measurement of viscosity and determination of flow types for mineral suspensions', Int.J.Miner.Process., 47, pp. 275-283.
- Vlachou, V. and Piau, J., (2000), 'A new tool for the rheometric study of oil well cement slurries and other settling suspensions', Cement and Concrete res., 20, pp. 1551-1557.
- 12. HES docket number-2005-IP-018807u1.
- 13. Metzner, A.B. and Otto, R.E., (1957), 'Agitation of non-Newtonian fluids', AIChE J., 3, pp. 3-10.

- 14. Metzner, A.B. and Taylor, J.S., (1960), 'Flow patterns in agitated vessels', AIChE J., 6, pp. 109-114.
- Steffe, J.F., (1992), 'Rheological methods in food process engineering', Freeman Press, East Lansing, Michigan, pp. 128-138.
- 16. Fan, Y. and Holditch, S.A., (1994), 'Use of Volumetric-Average Shear Rate to test Crosslinked Fluids with the Fann 50 viscometer', SPE, Pittsburgh, pp. 191-196.
- 17. Harris, P.C., Morgan, R.G. and Heath, S.J., (2005), 'Measurement of proppant transport of frac fluids', SPE95287, Dallas, pp. 1-13.
- Tehrani, M.A., (1996), 'An experimental study of particle migration in pipe flow of viscoelastic fluids', J.Rheol., 40(6), pp. 1057-1077.
- Stickel, J.J. and Powell, R.L. (2005) 'Fluid mechanics and rheology of dense suspensions,' Ann. Rev. Fluid Mech. 37, pp. 129-149.
- Zarraga, I.E., Hill D.A. and Leighton D.T. (2000) 'The characterization of the total stress of concentrated suspensions of noncolloidal spheres in Newtonian fluids' J. Rheol., 44, pp. 185-220.
- Morris, J.F. and Boulay, F. (1999) 'Curvilinear flows of noncolloidal suspensions: The role of normal stresses, J. Rheol., 43, pp. 1213-1237.
- 22. Barnes, H.A., Hutton, J.F. and Walters, K., (1989), 'An introduction to rheology' Elsevier, Amsterdam, pp. 18.