

Gel Strength Measurement for Drilling Fluid: Reform of Gel Microstructure

Allan Ye, Kevin Kleinguetl, and Sandeep Kulkarni, Halliburton

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Abstract

An important functionality of drilling fluids is the ability to suspend drilling cuttings and weighting material when circulation ceases. This is quantitatively demonstrated by the gel strength. Generally, low-gel-strength drilling fluid cannot efficiently suspend cuttings and other weighting materials. This can cause several severe problems, such as accumulation of cutting beds, stuck pipe, and unbalanced pressure. A higher gel strength of the drilling mud, however, requires higher pumping energy to recirculate after stopping. The resulting high initiation pressure has the potential to exceed the formation pressure and cause damage. Thus, the gel strength of drilling fluid should be monitored closely during the process.

The accurate measurement of gel strength is a key factor to a successful drilling operation. Several methods have been developed for gel strength measurement, such as API standard testing and large amplitude oscillation shear (LAOS). However, these methods are not capable of providing information regarding the gel microstructure. Recently, nuclear magnetic resonance (NMR) has also been used to characterize the gelling mechanism. However, this is not practical because of the costs associated with testing. In this study, the small amplitude oscillation shear (SAOS) is applied to probe the microstructure during gel reforming. The results are compared to those obtained using other methods. This cost-effective technique allows studying the rebuilt gel without disrupting or influencing the gelation process. The connection between the overshoot in pressure observed during a real-time drilling fluid operation and its gelation process are discussed.

Introduction

Gel strength is a measurement of the ability of a colloidal dispersion to develop and retain a gel structure. It is widely used in the food and paint industries, as well as in pharmaceutical, medical, and cosmetic product processes. For example, it is one of the key factors used to optimize toothpaste manufacturing. Several methods, such as a puncture test, uniaxial compression test, folding test, and torsion test, have been used in these industries to characterize gel systems.¹

In the oil/gas service industry, the gel strength of drilling fluids is usually a reference to the shear stress measured at a low shear rate after the drilling mud has rest for a period of time. This is one of the crucial rheological properties that must

be considered in the design of a successful drilling fluid. According to API standard procedure, the gel strength is reported in lb/100 ft² and is predominantly evaluated using a FANN[®] Model 35 viscometer equipped with bob/cup geometry. Other rotational viscometers, such as a Brookfield viscometer, can also be used. Low-field NMR is another technique that has been used to provide fast and accurate gel strength characterization for crosslinked gels.² However, the associated costs and difficulty of use prohibit its wide application. In contrast to the gel strength measurement using simple shear in a viscometer, dynamic oscillatory shear tests are commonly used to investigate gel properties. This can be classified into SAOS and LAOS, depending on whether the test amplitude is in a linear or nonlinear regime. Recently, several papers have used the LAOS techniques to examine the rheology of drilling fluids.³⁻⁵ This technique can be used effectively to probe the material's nonlinear characteristics. However, extensive knowledge of Fourier transform is required to interpret the data. In SAOS measurement, the strain amplitude is sufficiently small to be in the linear regime. Thus, the material response is in the linear regime, so the viscoelastic properties can be fully characterized without destroying the microstructure. In other words, the material gel strength can be directly related to the microstructural growth that is performed through a SAOS test.

Drilling fluid is primarily used to provide sufficient viscosity and gel strength to support weighting materials and transport cuttings from down hole to the surface. It usually contains invert emulsions for an oil-based mud or hydrated polymers and clay for a water-based mud (WBM). The microstructure of these systems is expected to be different because of the nature of their gelation mechanisms. In this work, the gel strength is measured using SAOS to monitor the reform of these different microstructures.

Experimental Section

Test Fluids

Three drilling fluids were used in this study: a WBM with a density of 12 lbm/gal, an ester/isomerized olefin-based mud (OBM 1) with a density of 13.2 lbm/gal, and an Invermul diesel-based mud (OBM 2) with a density of 14.6 lbm/gal. These were chosen to represent different microstructures of the colloidal systems. The WBM contains a synthetic polymer as the viscosifier and was prepared in the laboratory. Tests

were performed on the sample after a 16-hr hot-roll at 150°F. The two oil-based muds (OBMs) were obtained from a mud plant and were used as received. All gel strength measurements were performed at 120°F.

Gel Strength Measurement

In this work, a Brookfield viscometer equipped with a modified V-73 vane was used for the gel strength measurements. The V-73 vane was modified to be half the length of the original to prevent over-torquing at the desired shear rates. API procedure was followed (i.e., the sample was subjected to low shear after rest at various periods, the stress profile was obtained, and the peak value was recorded as the gel strength). The Brookfield viscometer was used because it is capable of automatic data acquisition, which helps minimize human error compared to dial reading on a FANN viscometer. Moreover, the Brookfield viscometer allows one run to be performed at multiple low shear rates, which can impact the stress peak reading. This will be presented and discussed in a separate paper. In this work, however, 0.5 rev/min (corresponding to a shear rate of 0.11 s⁻¹) was used for the gel strength measurements, which was found to be in good agreement with those obtained using a FANN viscometer at 3 rev/min.

SAOS

In this measurement, both stress and strain vary cyclically with time, where a sinusoidal wave is typically followed. By monitoring the material response as a function of this sinusoidal deformation, their viscous and elastic contributions can be obtained in molecular and microscopic levels. In this work, the sinusoidal strain is applied to the sample:⁶

$$\gamma(t) = \gamma_0 \sin \omega t, \quad (1)$$

where ω is the frequency and γ_0 is the strain amplitude. When the chosen γ_0 is sufficiently small to be in the linear regime, the response of material to this deformation can be written as

$$\sigma(t) = \sigma_0 \sin(\omega t + \delta), \quad (2)$$

where σ_0 is the stress and δ is the phase angle. This stress response can be decomposed into two components: in-phase and out-of-phase with the imposed strain, i.e.,

$$\sigma(t) = \sigma_0 \cos \delta \cdot \sin \omega t + \sigma_0 \sin \delta \cdot \cos \omega t. \quad (3)$$

Consequently, the storage (G') and loss (G'') moduli can be defined as ratios of the in-phase stress and out-of-phase stress to the strain amplitude, respectively. That is,

$$G' = \sigma_0 \cos \delta; \quad G'' = \sigma_0 \sin \delta. \quad (4)$$

The storage module G' is the material capability to maintain energy after deformation, which will fully recover upon removal of the strain. Its magnitude is related to reform

of the microstructure and thus should directly characterize the gel strength of the material.

Rheological tests were conducted on a stress-controlled rheometer (MCR 501, Anton Paar) equipped with a 27 mm bob/cup geometry. A solvent trap was used to help minimize the solvent evaporation under the testing temperature of 120°F. Typically, the sample is pre-sheared at a high shear rate, which is then followed by a SAOS time sweep. The storage module profile is recorded as a function of time, which provides insight of the gel structure reforming. In the next section, this will be compared to the gel strength obtained from the Brookfield viscometer.

Results and Discussion

The gel strength measurement was conducted with a Brookfield viscometer. Before the test, the sample was pre-sheared to obtain the same initial condition. Then, the sample was rest for various time periods (i.e., 10 seconds and 5, 10, 15, and 30 minutes) before resuming the shear at a rate of 0.11 s⁻¹. The shear stress was monitored over time, as can be observed in **Figure 1** for OBM 1 at 120°F. Except for the case of resting for 10 seconds, the shear stress showed a peak, which was recorded as the gel strength for the mud. As expected, the longer the mud was in rest before resuming the shear, the higher the gel strength achieved. The peak occurred at approximately the same time (i.e., 8 seconds) after resuming the shear. Thus, the gel strength for the 10-second rest time (where no peak stress is observed) was taken as the shear stress at 8 seconds. All of the gel strength data were obtained at 120°F and are listed in **Table 1** for comparison. For the two OBMs, the gel strength was a monotonic function of the resting time. This is because both OBMs were primarily formed by the emulsion gel microstructures. The bridging of the emulsion droplets and organophilic clays is the gelling mechanism for traditional invert emulsion drilling fluids.^{4, 7, 8} An increase in the gel strength indicates the reforming of the emulsion microstructure over time after pre-shearing. In contrast, the gelation mechanism was quite different for the WBM, where the gel microstructure was controlled by the polymeric viscosifier. This relatively weak interaction force was not sufficient to reform the gel microstructure upon removal of the pre-shear deformation. As a result, the gel strength for the WBM was much lower, almost independent of the resting time.

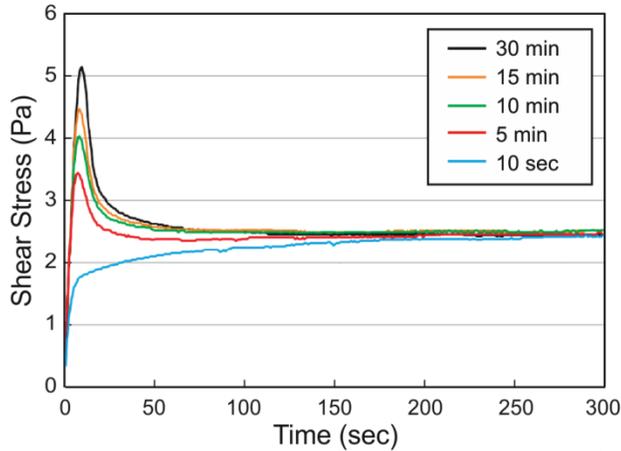


Figure 1: Shear stress profile at a shear rate of 0.11 s^{-1} for OBM 2 at 120°F after rest for various time periods.

Before the oscillatory test, the flow curve was obtained for all samples using an MCR 501 rheometer. This simple shear is a nonlinear deformation to the material. As can be observed in **Figure 2**, the steady shear viscosity trend was similar for all muds, following a power law relationship at low to intermediate shear rates and approaching a constant at high shear rates. OBM 1 had the highest viscosity over the entire shear rate measurement window, which was almost double that of OBM 2. The WBM showed a very similar shear viscosity to OBM 2. In other words, these muds behaved similarly when the gels were broken by shear. However, how the gel structures reform after shear was unknown based on the observation of these nonlinear shear data.

Table 1: Gel strength at 120°F for all samples obtained from Brookfield viscometer.

Rest Time (min)	Gel Strength (Pa)		
	OBM 1	OBM 2	WBM
0.167	1.0	1.74	1.47
5	5.19	3.46	1.52
10	6.10	4.04	1.54
15	6.58	4.48	1.56
30	7.56	5.1	1.56

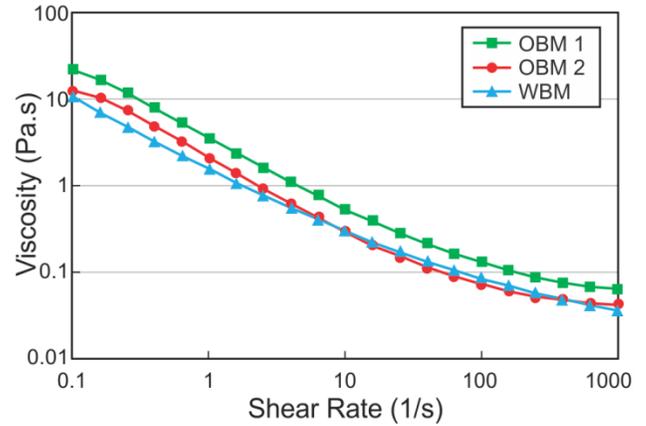


Figure 2: Flow curves for the three muds at 120°F .

To determine the linear and nonlinear regimes of the sample, a strain sweep test was conducted at a fixed frequency. **Figure 3** shows the storage and loss moduli as a function of strain for all samples at a frequency of 10 rad/s . The linear viscoelastic regime is defined as the strain range, where the storage modulus is independent of strain. In other words, a deformation of a strain within the linear viscoelastic regime results in a full recovery of structure upon removal of the deformation. With increasing strain, the storage and loss moduli decrease significantly, and the gel structure is not fully recoverable. This nonlinear response is out of the scope of the current work. Both OBMs demonstrated approximately 10 times less strain range (0.4%) for the linear viscoelastic regime compared to the WBM (strain of 4%). Moreover, their magnitude of storage modulus was approximately 10 times greater than that of the WBM, indicating that these OBMs had higher gel strength than the WBM. In the following tests, a strain of 0.25% was used for both OBMs and 2.5% was used for the WBM.

A frequency sweep test was also performed for all samples to investigate the gel response to different time scales. The overall material response is a result of the contributions from several mechanisms at the molecular and microscopic levels. **Figure 4** shows the storage and loss moduli as a function of angular frequency. A similar dependence of storage and loss moduli on the angular frequency is observed for all muds. Compared to the rotational shear data shown in **Figure 2**, the SAOS data show more remarkable differences between these muds.

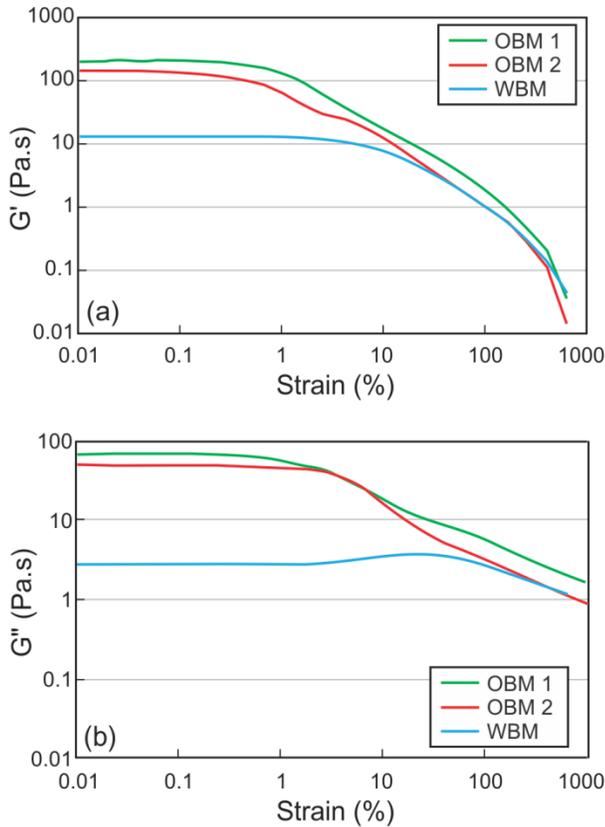


Figure 3: Strain sweep for all samples at 120°F: (a) storage (G') and (b) loss (G'') moduli as a function of strain.

Finally, an SAOS time sweep test was conducted to monitor the gel structure reform after shearing. Before the test, the sample was sheared at 1000 s^{-1} for 100 seconds to destroy the gel structure. **Figure 5** shows the storage modulus as a function of time after pre-shearing. As expected, the storage modulus of both OBMs increased with time and eventually leveled off after 15 minutes. This is related to the emulsion reform over time. Both OBMs had strong flocculated emulsion that could recover upon removal of deformation (pre-shearing). In contrast, the storage of the WBM was almost constant during the gel reforming period, with a magnitude much smaller than that of the OBMs. Again, this suggests that the gel microstructure reform was unlikely to occur for the WBM, where weak gelation was observed through interaction of the hydrated polymeric viscosifier and colloidal particles.

As mentioned previously, the gel was measured through a rotational test, where the sample was constantly sheared in a single direction. This was in the nonlinear viscoelastic regime. The data obtained through the SAOS time sweep test were a direct indication of the gel microstructure reform process. Because this was in the linear viscoelastic regime, however, adjustment was necessary to make a comparison between the storage modulus profile and those gel strengths obtained using the Brookfield viscometer.

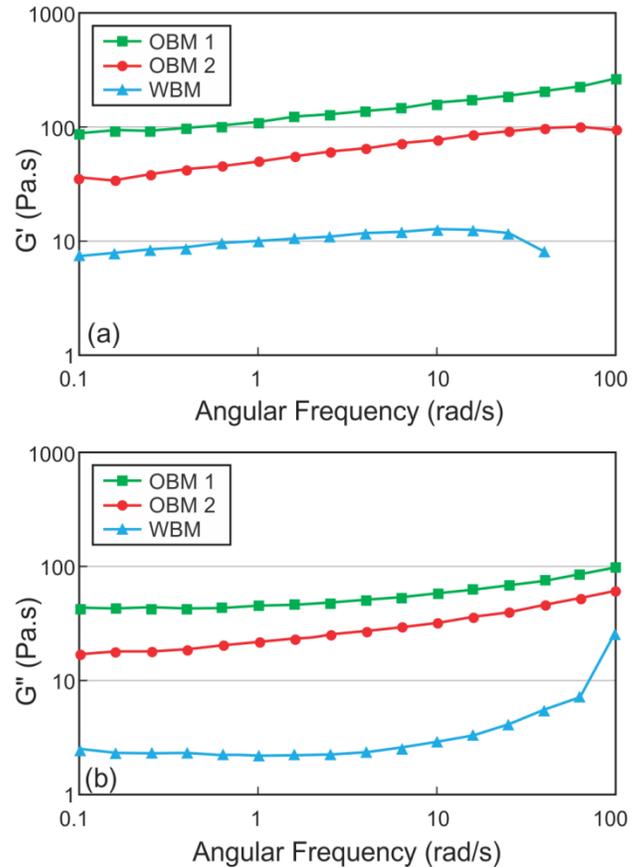


Figure 4: Frequency sweep for all samples at 120°F: (a) storage (G') and (b) loss (G'') moduli as a function of angular frequency.

In the work, a simple empirical method is proposed to describe the connection between simple shear (nonlinear) and SAOS (linear). This is based on the so-called Cox-Merz rule.⁹ This empirical relationship states that the dependence of the steady shear viscosity on the shear rate can be estimated from the dynamic viscosity as a function of frequency, as the two curves are approximately identical. That is,

$$\eta(\dot{\gamma}) = |\eta^*(\omega)|_{\omega=\dot{\gamma}} = \sqrt{[(G'/\omega)^2 + (G''/\omega)^2]}_{\omega=\dot{\gamma}} \quad (5)$$

where $\eta^*(\omega)$ is the dynamic viscosity. Although this empirical rule has been proved for several polymer melts and solutions, it is not valid, in general, for suspensions with large solid content. It is not surprising that this rule does not apply to drilling fluids. In this work, a factor (α) is defined as the ratio of the dynamic viscosity to the shear viscosity and is used to convert the storage modulus to the gel strength in such way that

$$GS_{SAOS} = \frac{G'}{\alpha} \quad (6)$$

where G_{SAOS} is the gel strength obtained from the SAOS time sweep test. **Figure 6** shows the gel strength obtained from **Equation 6** as a function of time. The gel strength data obtained from the Brookfield viscometer (listed in **Table 1**) are also included as symbols for comparison. Clearly, the gel strength obtained from both techniques is in good agreement for all three muds studied in this work. Moreover, the advantage of using SAOS compared to the traditional method of rotational shear is the time savings and accuracy, where a single run of the SAOS time sweep provides details of the gel strength during the reform process. The benefit is more remarkable when the gel strength is required at multiple resting time periods.

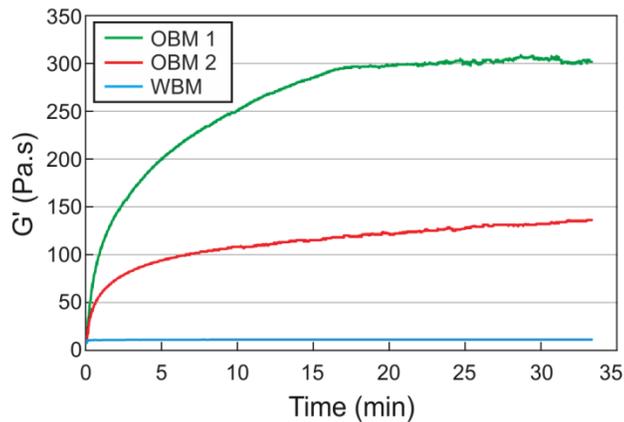


Figure 5: Storage (G') as a function of time after pre-shearing all samples at 120°F.

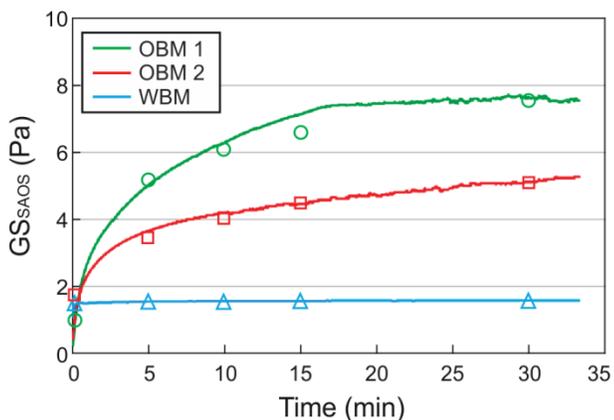


Figure 6: Comparison of the gel strength obtained from SAOS (lines) and those obtained from a Brookfield viscometer (symbols) for all three muds at 120°F.

Conclusions

Nonlinear steady shear (flow curve) cannot provide sufficient evidence of the different gel structures for drilling muds. Instead, SAOS can be used to probe the microstructure of the drilling fluid without disturbing its gelation and thus provides an alternate option to characterize gel strength. This work demonstrates that gel strength can be better measured through a SAOS time sweep test. The benefit is more obvious

using this method compared to the API standard test of gel strength, especially when the gel strength is required at multiple rest periods.

Acknowledgments

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Nomenclature

<i>WBM</i>	=	Water-based mud
<i>OBM</i>	=	Oil-based mud
<i>SAOS</i>	=	Small amplitude oscillatory shear
<i>LAOS</i>	=	Large amplitude oscillatory shear

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