

## A Rheological Approach to Differentiating Muds by Gel Structure

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This paper was prepared for presentation at the 2010 AADE Fluids Conference and Exhibition held at the Hilton Houston North, Houston, Texas, April 6-7, 2010. This conference was sponsored by the Houston Chapter of the American Association of Drilling Engineers. The information presented in this paper does not reflect any position, claim or endorsement made or implied by the American Association of Drilling Engineers, their officers or members. Questions concerning the content of this paper should be directed to the individuals listed as authors of this work.

### Abstract

Drilling muds have long been known to develop weak bonds between particles and emulsion droplets in suspension, commonly known as forming a gel structure. The usual measurement for this is obtained through a flow initiation on a standard oilfield viscometer, with the gel strength recorded as the peak in this transient test. This has been a useful test but is limited in several ways, including the sensitivity of the equipment and limitations in the test itself. Hidden behind these limitations are basic differences in the gel structures of differing mud systems, where changes in the emulsion package, base oils, organophilic clays, solids loading, and other viscosifiers affect the development of structure within the fluid.

The possibility of exploring differences in gel structure exists in the form of advanced rheometry. Presented in this paper is a comparison of traditional gel strength measurements with various rheometric techniques. The ability of each method to reveal complexities in the gel structure will be evaluated in a selection of downhole fluids.

### Introduction

The need for low viscosities for ease of flow in the annulus, the need for high viscosities to prevent fluid invasion and aid in hole cleaning, and the intermittent nature of the drilling process all compete for priority in the design of a successful drilling fluid. To meet these opposing criteria a mud must be a complex fluid, exhibiting viscoelastic properties to provide appropriate viscosities at the needed shear rates and also exhibiting viscoplastic properties through thixotropic and yielding properties to suspend solids in low-flow and stagnant conditions. For these reasons clays, which form associative networks or a microstructure in the fluid, are used as viscosifiers.

Traditionally, invert emulsion drilling fluids are prepared using various types of base oils emulsified with brine as the internal phase. Amine-treated bentonite (or other organophilic clay) is added, along with polymeric viscosifiers, to control rheological properties. Various lignitic and asphaltic materials may be added for filtration control, and barite or other weight material added to control density.<sup>1-4</sup> All of these components, including barite which is usually considered as inert in the system, interact to form the overall microstructure of the fluid. Any change in one of these components affects how

everything interacts and thus affects the nature of the microstructure.

Overly-structured fluid can provide problems as severe as an under-structured fluid. Insufficient hole cleaning and barite sag are among the common results of a drilling mud forming too little microstructure. If the fluid builds a sufficiently strong structure, however, the stress required to break the structure (by tripping pipe, initiating pump flow, etc.) and initiate flow will become excessively high, resulting in tremendous pressure surges and the likelihood of fracturing the formation.

A traditional view lists the important governing parameters for start-up circulation pressures as<sup>2</sup>:

1. Temperature
2. Aging time
3. Column rotation
4. Start-up flow rate

These parameters can be generalized to apply to any situation where the gel structure in a mud is being broken. A proposed addition to these parameters would be the nature of the gel microstructure in question. With modern options, the microstructure may be based around a traditional emulsion / organophilic clay interaction, or it may arise from emulsion only with no organophilic clays or solids added. Additionally, this microstructure may form from crosslinked polymers or self-assembly of surfactants, polymers or both together.

These fundamentally different systems cannot be expected to behave the same way, and care should be taken to understand the differences between how they behave. This paper begins to explore this concept through investigation of field muds with and without organophilic clays and other solids in the system.

### Experimental Methods

Rheological testing was performed on an Anton-Paar MCR501 stress-controlled rheometer. In general, before testing, all fluids were brought to a test temperature of 120°F and then pre-sheared for two minutes immediately before beginning the test, significantly breaking the microstructure and providing a common starting point. A standard couette cell was used to produce a flow curve for each fluid, typically in the range of 1 – 1200 s<sup>-1</sup>. Below this range, wall slip significantly distorted measured data, and further testing was performed using a six-vane stirrer. Flow curves in the range

of  $10^{-4} - 100 \text{ s}^{-1}$  were generated using the vane stirrer, and this data was compared to the data from the standard couette flow curve in order to calibrate the vane stirrer for each fluid (using the method of Choplin and Marchal<sup>5</sup>). Once calibrated, the vane stirrer was used for subsequent tests on the rheometer.

Many tests have been used over the years to evaluate the “strength” of the gel formed in muds and the yield stress<sup>3</sup>. The results of these tests often vary greatly, may or may not be correlated to one another for a given fluid, and ultimately may not lead to a significant understanding and differentiation of drillings fluids and their microstructure.

General rules of thumb have been found from these tests and used in both determination of the proper fluid composition and prediction of the effects on such things as flow initiation and swab and surge pressures. These are not broadly applicable, but rather are limited in scope to the “standard” muds and drilling parameters on which they were based. The focus of this paper is the use of large amplitude oscillatory shear (LAOS) to examine yield stress, which is the stress required to initiate flow, and the energy required to break the microstructure in a single test.

## LAOS

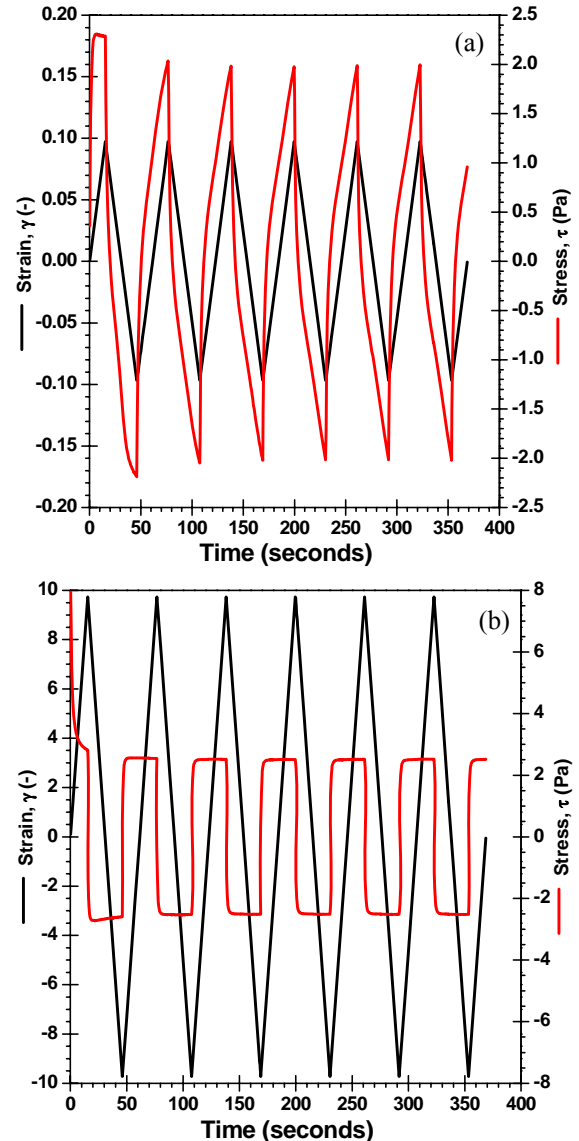
Several recent papers have examined drilling fluids through the LAOS technique.<sup>4,6</sup> A good explanation of this method and the rheological parameters which can be obtained from it can be found in the recent papers by Ewoldt and McKinley<sup>6,7</sup> among other sources. In short it systematically connects, through a limited set of experiments, steady flow viscosity  $\eta(\dot{\gamma})$ , linear viscoelastic moduli  $G'(\omega)$  and  $G''(\omega)$ , and nonlinear viscoelastic properties.<sup>8</sup> By examining the transient response of stress to an applied strain within the oscillation cycle, rather than by simply taking the absolute magnitudes of those oscillations, more information on the nature of the fluid can be obtained without disturbing the microstructure formed in the fluid.

For these tests, a triangular wave oscillation was applied at 0.1-rad/sec for strains of 10%, 50%, 100%, 500%, and 1000% (giving a maximum oscillation through  $\sim 180^\circ$ ). Triangular waves were used, rather than standard sinusoidal waves, for ease of set setup and data collection. As a result, the traditional moduli ( $G'$  and  $G''$ ) do not have the same physical meanings and thus will not be discussed here. However, this test can be used to determine at what strains the fluid becomes fully yielded (where elastic and viscous contributions are overwhelmed by the plasticity of the fluid) while remaining in a closed-loop test where the fluid is stressed but not completely disturbed. By testing under fully yielded conditions, the following parameters can be obtained:

1. Yield Stress ( $\tau_Y$ )
2. Peak, or overshoot, Stress ( $\tau_P$ )
3. Energy Dissipation ( $E_D$ ) in breaking microstructure

These will be compared to mud weight, oil-water ratio (OWR), and low gravity solids (LGS) content in a selection of muds obtained from ongoing drilling operations.

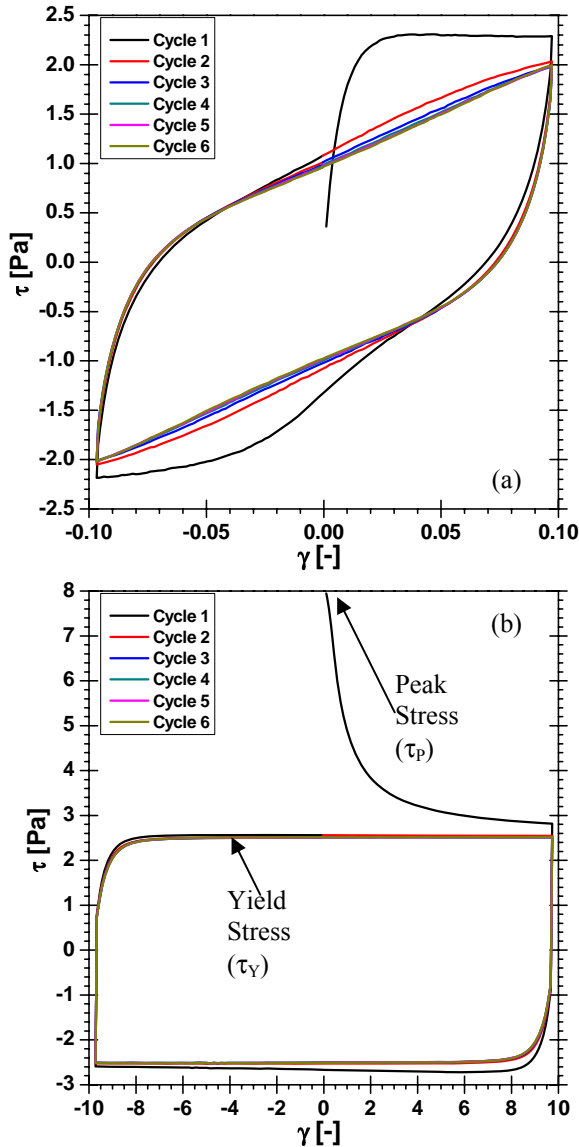
The typical response of a fluid to a triangular-wave LAOS at small and large strains is presented in Figure 1. At small applied strains, the stress response is roughly in the same form as the input – a triangular wave with some bending due to elasticity in the sample. For very large deformations the viscoelastic contributions are dwarfed by the yielding behavior and the stress response looks like a square wave.



**Figure 1** Triangular-wave LAOS raw data for a drilling fluid sample at (a) 10% and (b) 1000% strain amplitude.

A more convenient way to look at this data is using a Lissajous curve, plotting the stress as a function of strain. The data in Figure 1 is replotted in this way and presented in Figure 2. This allows one to view the test as an enclosed curve, which as an oscillatory test it is, and observe the transient response of stress as a function of strain. In this figure, each oscillatory cycle is presented as a different color curve. What is most obvious is that the first cycle is appreciably different from subsequent cycles; this is the result

of the start-up response of the mud to flow initiation. For the small strain case (Figure 2a) stress rises quickly in the first cycle and then plateaus before the flow direction reverses. This is due to partial yielding of the fluid at this strain, which becomes quickly dominated by viscoelastic response as the gel structure is partially broken and finds a steady state. Subsequent cycles do not display this yielding behavior, only the viscoelastic response of the fluid.



**Figure 2** Raw data from Figure 1, for a drilling fluid sample at (a) 10% and (b) 1000% strain amplitude, replotted as Lissajous curves. A total of six oscillatory cycles are presented for each test.

The Lissajous curve for the 1000% strain test (Figure 2b) is quite different from the test at 10% strain. Rather than a slow rise to a plateau in the first cycle, a large spike is observed as a large stress (large amount of energy) is required to yield the fluid and initiate flow. Subsequent cycles are square, owing to the dominance of yielding, plastic behavior over viscoelastic

responses at this strain, and approach a steady state with the microstructure broken. The magnitude of the peak stress,  $\tau_p$ , varies from fluid to fluid, and is tracked in this paper as a parameter of interest. The stress plateau observed here does not change appreciably (at constant oscillatory frequency) once the fluid is fully yielded and is thus a reliable measure of the yield stress of the fluid. All data compared here is performed at 1000% strain amplitude, which was found to be fully yielded for all mud samples tested.

The stress required to initiate flow represents an energy burden for breaking microstructure in the fluid. The LAOS test is also ideal for examination of this energy. The energy for a single cycle can be found by simply integrating the stress as a function of strain

$$E_D = \int \tau d\gamma \quad (1)$$

where  $E_D$  is the energy dissipation per unit volume. By evaluating  $E_D$  for each cycle individually, the energy required to initiate flow (cycle 1) can be compared to the energy required for flow at steady state (usually by cycle 6). This energy is that which is required to break microstructure in the fluid, is greatest in the first cycle (where most bonds are broken) and less in subsequent cycles as all microstructural bonds are broken over time. By comparing  $E_D$  in each cycle verses that at steady state, and excess energy dissipation,  $E_D^E$ , is found

$$E_D^E = \sum_i (E_{D,i} - E_{D,6}) \quad (2)$$

which shows the total energy per unit volume required to break microstructure and initiate flow at this strain.

### Test Fluids

A total of 12 invert emulsion muds were sampled from ongoing field operations and tested for this paper. No laboratory-mixed muds were used in this study, as they are known to vary in performance from similarly formulated fluids that have been used in drilling. The mud weight, OWR, and percent by volume low gravity solids for the 12 muds are presented in Table 1.

The peak stress, yield stress, and  $E_D^E$  are reported as observed from tests at 1000% strain amplitude. Prior to testing, each mud was blended at low shear for 30 minutes to re-suspend any solids which may have settled out of the fluid and reincorporate any oil which may have undergone syneresis. All fluids were tested as received from field operations, without any chemical treatments. Fluids #4, #5, and #6 are all organophilic clay-based invert emulsion fluids, while the remaining nine fluids contained no organophilic clays or other organophilic solids.

**Table 1** Properties of the invert emulsion field muds tested. Fluids #4, #5, and #6 contain organophilic clays while the remaining fluids are free of organophilic clay.

#	Mud Weight (lb/gal)	OWR	LGS (% <sub>v</sub> )	$\tau_P$ (Pa)	$\tau_Y$ (Pa)	$E_D^E$ (J/m <sup>3</sup> )
1	12.4	69/31	7.7	7.95	2.53	13.67
2	11.9	69/31	4	4.39	2.04	10.77
3	11.6	68/32	5.5	4.72	2.27	-0.95
4	13.35	82/18	14	8.99	3.47	28.52
5	11.9	70/30	12	14.55	4.39	25.85
6	12.7	70/30	5.8	6.49	3.068	24.43
7	14.1	80/20	4.94	6.16	2.95	15.32
8	14.1	80/20	4.94	6.36	2.46	13.89
9	12.5	75/25	3.1	1.67	0.86	1.57
10	14.3	--	--	0.80	0.29	1.56
11	11.6	77/23	4.4	5.31	3.97	12.16
12	14.9	74/26	1.5	7.98	2.99	17.45

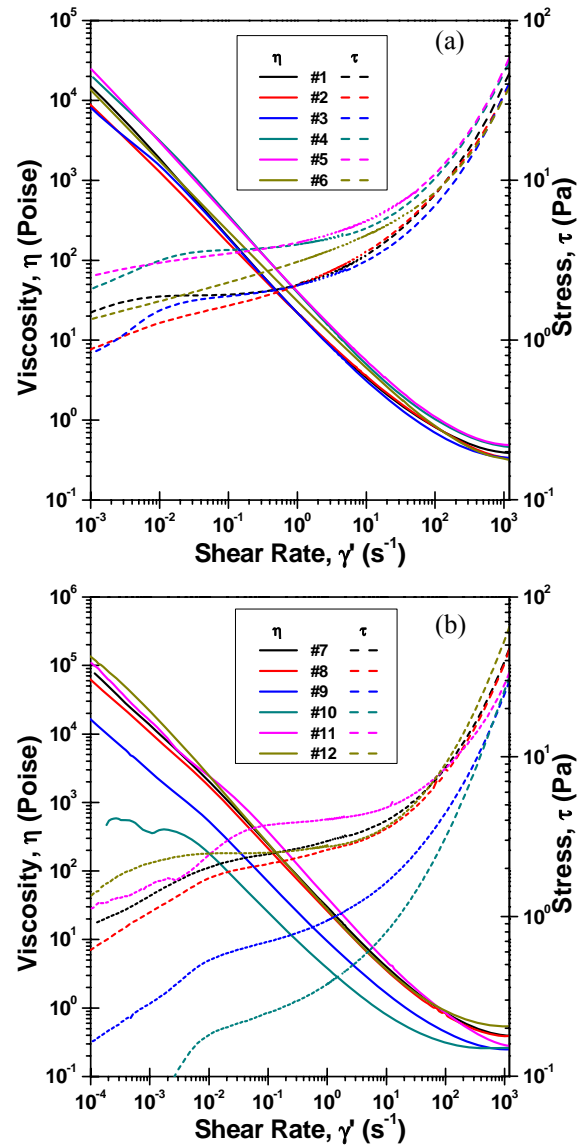
### Flow Curves

Figure 3 compares the flow curves of the 12 test fluids. A comparison of the Bingham plastic and Hershel Bulkley parameters is presented in Table 2, again demonstrating the relative similarity of most of the fluids. With the exceptions of fluids #9 and #10, which are very thin muds, all of the test fluids exhibited relatively similar flow profiles, with modest variation in the location and appearance of the yield stress plateau.

For some fluids, such as #11 in Figure 3b, there is a very clear transition from shear-thinning at high shear rates to a relatively flat yielding plateau at mid-range shear rates, followed by what appears to be a shear-thinning region at very low rates. This low-shear behavior is opposed to the expected Newtonian behavior noted in literature at low shear rates in yielding fluids.<sup>9</sup> Other fluids show a gradual transition from the upper shear-thinning region to a lower shear-thinning region and do not clearly exhibit a yield plateau, for example fluid # 6 in Figure 3a.

**Table 2** Herschel Bulkley and Bingham plastic model parameters for the test fluids.

#	Bingham plastic		Herschel Bulkley		
	PV (Poise)	YP (Pa)	n	K (Pa·s <sup>n</sup> )	$\tau_0$ (Pa)
1	0.38	2.60	0.821	0.127	2.14
2	0.33	2.54	0.706	0.240	1.79
3	0.33	2.28	0.841	0.096	1.95
4	0.44	4.09	0.832	0.137	3.61
5	0.46	4.42	0.817	0.158	3.82
6	0.31	3.37	0.692	0.244	2.56
7	0.39	2.61	0.794	0.154	2.22
8	0.38	2.36	0.821	0.126	1.95
9	0.25	0.92	0.835	0.074	0.684
10	0.26	0.30	0.991	0.027	0.292
11	0.27	3.56	0.639	0.301	2.84
12	0.52	2.69	0.929	0.083	2.51



**Figure 3** Flow curves for the twelve test fluids, at 120°F.

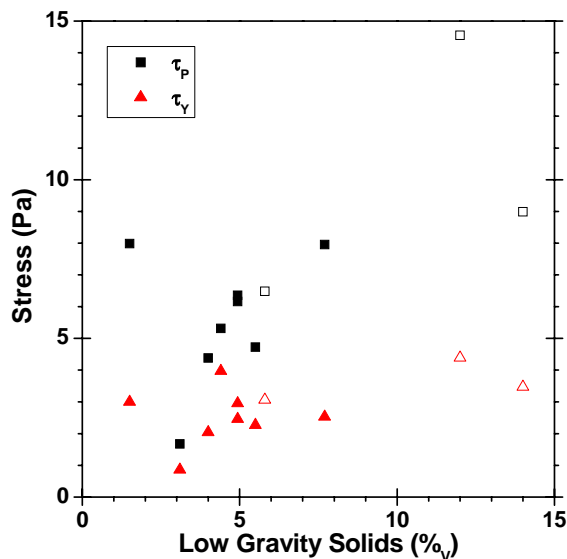
### Triangular-wave LAOS Results

LAOS experiments were performed on all mud samples using triangular-waves at a frequency of 0.1-rad/sec and multiple strains. Since all fluids exhibited fully developed yielding behavior at strain amplitudes of 1000%, data from these tests will be analyzed here. Some debate may be considered as to the proper strain at which to compare these fluids. One argument would suggest that using a uniform yielding strain for all comparative tests provides a simple basis to examine the energy dissipated in microstructure breakage with a minimal number of tests. This is the approach taken in this paper. Another argument would suggest the best comparison would be at the lowest strain at which each fluid fully yields, the yield strain. This has the advantage of

potentially better portraying the actual energetic requirements for microstructure breakage when tripping pipe or during pump startup in drilling operations; however, this method requires many more tests and greater care to identify the yield strain. A third argument would promote the use of the gel point, the strain at which  $G'$  and  $G''$  are equal, as a comparative point. This would not require an extensive test matrix, but would not reveal information on the actual yield stress or the overshoot experienced during flow initiation.

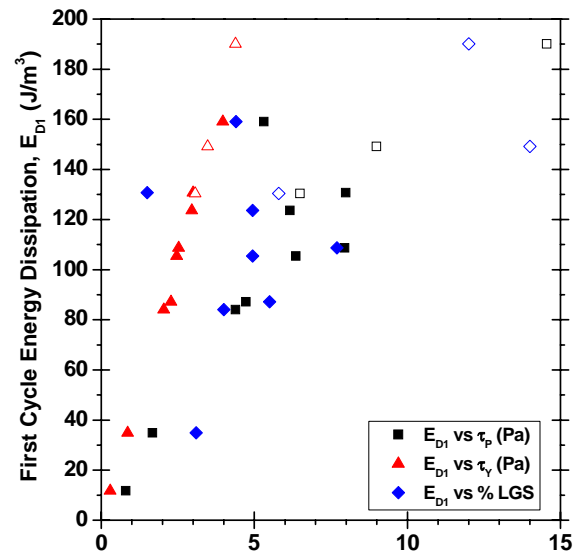
The peak stress, yield stress, and energy dissipation as calculated from LAOS tests were compared to the physical properties of the muds presented in Table 1 (mud weight, OWR, and % LGS). As expected, some good correlations were made and are presented in Figures 4-8. In general, it was observed that there was no correlation between the rheological properties observed in triangular-wave LAOS and either mud weight or OWR. Rather than suggesting that these do not affect rheological properties, it would seem to indicate that the design process of these fluids takes that into account and mitigates their effects to produce rheologically similar fluids at different densities and oil content.

In Figure 4,  $\tau_p$  and  $\tau_Y$  are plotted as functions of LGS content. Traditional wisdom has told us that increasing LGS content in the mud makes the yield stress increase and the mud overall more difficult to treat. That is borne out here, as both the  $\tau_p$  and  $\tau_Y$  are observed to be roughly linearly dependent on % LGS. The correlation holds better for the  $\tau_p$  than for the  $\tau_Y$ , and better below 10% LGS. This strongly indicates that running a mud at higher LGS levels will impact the energy required to initiate flows during pump startup and tripping pipe, leading to larger pressure surges downhole.



**Figure 4** Plot of  $\tau_p$  and  $\tau_Y$  from triangular-wave LAOS as functions of low gravity solids content for the 12 drilling fluid samples. Hollow data points represent the three muds containing organophilic clays.

An examination of  $E_{D,1}$  (the energy dissipation in the first cycle) is presented in Figure 5. Here, very good linear fits can be made, most especially for  $E_{D,1}$  as a function of  $\tau_Y$ . This is expected, as the  $E_D$  in this case is essentially the area of a square (see Figure 2b) with one dimension as  $\tau_Y$ . Good correlation of  $E_D$  to  $\tau_p$  is not as intuitive but follows when it is observed that  $\tau_p$  is well-correlated to  $\tau_Y$ . This would indicate that the impact of fluids with higher yield stresses on flow initiation is somewhat predictable, but should be based not on  $\tau_Y$  but on  $\tau_p$ . In Figure 5 we also observe that  $E_{D,1}$  is also a rough function of % LGS. This reinforces the observations from Figure 4 that higher LGS content leads to increased energy required to break microstructure and initiate flow.



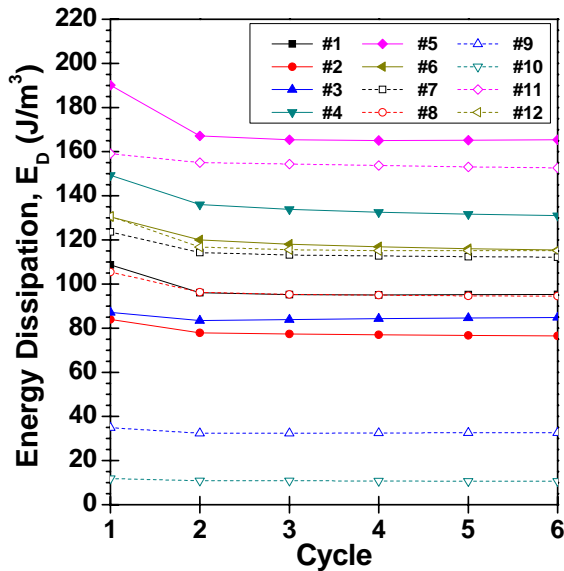
**Figure 5**  $E_{D,1}$  plotted as a function of  $\tau_p$ ,  $\tau_Y$ , and % LGS. Hollow data points represent the three muds containing organophilic clays.

As is obvious from the Lissajous curves in Figure 2, the enclosed area in each cycle progressively decreases from the first cycle until a steady state is reached. The change in  $E_D$  by oscillation cycle is presented in Figure 6.

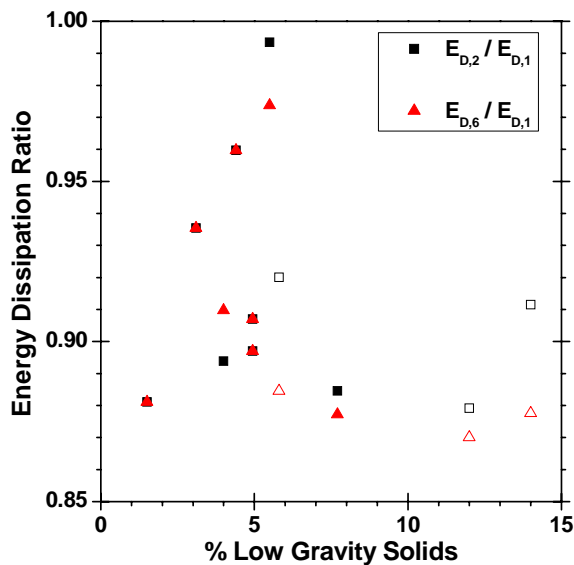
For all fluids, the energy dissipation in the first cycle was greater than that of the second cycle, while only minimal changes are observed in subsequent cycles. The magnitude of change from  $E_{D,1}$  to  $E_{D,2}$  varies between the muds and is explored in greater detail in Figure 7.

One noticeable difference between Fluids #4, #5, and #6 – the fluids containing organophilic clays – and the other organophilic clay-free fluids is that steady state is not reached as quickly, as observed by the slight downward slope in the  $E_D$  curve after the second cycle. For the organophilic clay-free fluids, with only the exception of Fluids #3, steady state is reached in the second cycle – no appreciable difference is observed in the energy dissipated per cycle between the second oscillation cycle and the sixth cycle. This indicates that, even though  $E_{D,1}$  may have a large value, all the microstructure is broken quickly. By contrast, for the

organophilic clay-based fluids, the microstructure breaks slowly, sometimes taking more than six cycles to reach a



**Figure 6**  $E_D$  from triangular-wave LAOS as a function of oscillation cycle for the 12 drilling fluid samples.



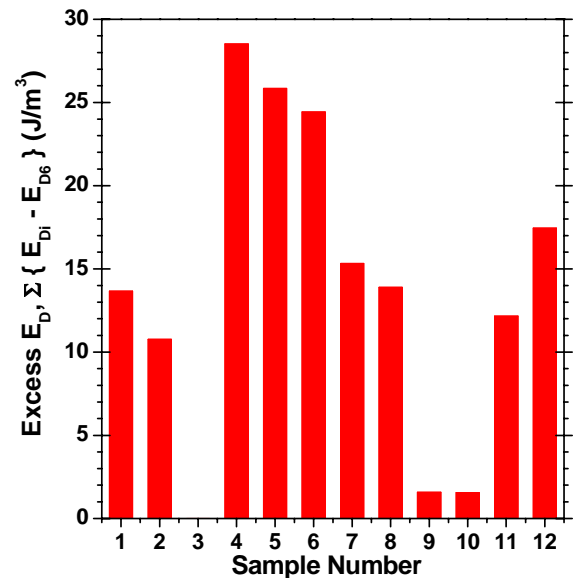
**Figure 7** Effect of low gravity solids content on the decrease in energy dissipated from the first oscillation cycle to cycles two and six. Hollow data points represent the three muds containing organophilic clays.

steady state. These differences will be explored more in discussion of Figure 8.

As noted, Fluid #3 is an exception to this observed ready breakage of microstructure in the organophilic clay-free fluids. In this case, the decrease in  $E_D$  observed after the first oscillation cycle followed by a very modest increase in  $E_D$  through the sixth cycle. The overall change is not large, but indicates that the fluid is actively building microstructure during the test.

As noted, the degree to which  $E_D$  changes as oscillations progress varies between the different fluids. Some explanation for these variations was found in comparison to the LGS content in the fluid (Figure 7). Here, rather than looking just at the total magnitude of energy dissipation, the ratios of first cycle energy dissipation to that of the second and sixth cycles ( $E_{D,2} / E_{D,1}$  and  $E_{D,6} / E_{D,1}$ ) are examined. This allows evaluation of the energy requirement to break the microstructure in the fluid and bring it to steady state. At lower solids concentrations, below ~6% LGS, the energy dissipation ratios are widely scattered, with either high or low dissipation ratios. Above 6% LGS, however, the energy dissipation ratio is always low, indicating a large energy burden in breaking microstructure to initiate flow. Most of these points are from fluids containing organophilic clays (the hollow points in Figure 7), but includes samples that are free of organophilic clays. This would indicate that, at high LGS loading, the microstructure requires a significant amount of energy to break as compared to steady state flow conditions and is an argument for solids management as part of the drilling process.

One final parameter, the excess energy dissipation, was examined to differentiate the fluids samples. This gives the total energy required to completely break microstructure within the fluid, and can be related to pressure spikes observed during pump startup and tripping pipe. A comparison of  $E_D^E$  for the 12 fluids is presented in Figure 8.



**Figure 8** Excess dissipated energy,  $E_D^E$ , for the 12 sample drilling fluids.

As noted previously, Fluids #9 and #10 have minimal rheology, and they are set apart here with minimal  $E_D^E$ . Also, Fluid #3, which actually exhibited a moderate increase in  $E_D$  after the second oscillation cycle, actually had a negative excess energy.

Further distinction can be found between Fluids #4, #5, and #6 (containing organophilic clays) and the remaining

organophilic clay-free fluids. For the organophilic clay-based fluids,  $E_D^E$  was  $\sim 25\text{-J/m}^3$  compared to  $\sim 15\text{-J/m}^3$  for the organophilic clay-free fluids. This would indicate that the interaction of the treated clay particles with the emulsion droplets adds complexity to the microstructure that proves difficult to disassociate. The excess energy dissipation does not appear to be influenced by either  $\tau_Y$  or  $\tau_P$ , demonstrated in a comparison of Fluids #1 and #6. Both fluids have similar OWR and LGS content (see Table 1) and while  $\tau_Y$  is slightly higher in Fluid #6, its  $\tau_P$  is lower than that of Fluid #1.

Additionally, the energy dissipation ratio from the first to second oscillation cycle ( $E_{D,2} / E_{D,1}$ ) for fluid #1 is 0.88 compared to 0.92 for Fluid #6, indicating that relatively more energy is required to break the microstructure in Fluid #1 in order to complete the first cycle. However,  $E_D^E$  for Fluid #1 is  $13.67\text{-J/m}^3$  opposed to  $24.43\text{-J/m}^3$  for Fluid #6. This result stems from the comparative ease of breaking the microstructure in Fluid #1, which is accomplished significantly accomplished in the first cycle. Fluid #6 breaks microstructure more slowly, over many cycles, before steady state is reached. Thus, the cumulative energy required to completely disassociate microstructural bonds in Fluid #6 is greater

## Conclusions

- Though great rheological differences exist, little differentiation can be made with simple viscometry between drilling fluids which contain significantly dissimilar gel microstructures. These differences can impact drilling operations, especially through pressure spikes observed during pump startup and tripping pipe.
- The presence and concentration of low gravity solids in drilling fluids affects the nature of microstructure formed in the fluid. Above a threshold concentration, the difference between stress required to initiate flow and that required to maintain flow at steady state is always large.
- Significant differences are observed in the disassociation of microstructure between fluids which contain or lack organophilic clays. In fluids free of organophilic clay, microstructure breaks quickly once flow is initiated and the total energy required to break that microstructure is lower than in fluids with organophilic clays. Total energy required for breaking microstructure is apparently independent of the yield stress of the fluids.

## Nomenclature

LAOS= *Large Amplitude Oscillatory Shear*

$G'$  = *Storage modulus (Pa)*

$G''$  = *Loss modulus (Pa)*

$\tau$  = *Stress (Pa)*

$\tau_Y$  = *Yield Stress (Pa)*

$\tau_P$  = *Peak Stress (Pa)*

$\eta$  = *Viscosity (Poise)*

$E_D$  = *Energy dissipation per cycle per unit volume ( $\text{J/m}^3$ )*

$E_D^E$  = *Excess  $E_D$  ( $\text{J/m}^3$ )*

LGS = *Low Gravity Solids*

OWR = *Oil to water ratio*

## Acknowledgments

The author would like to thank Halliburton for granting permission to publish the paper.

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