Viscoelastic Properties of Oil-Based Drilling Fluids

Binh Bui[3], Arild Saasen[1], Jason Maxey[2], Mehmet E. Ozbayoglu[3],

Stefan Z. Miska[3], Mengjiao Yu[3], Nicholas E. Takach[3]

[1]Det norske oljeselskap ASA and University of Stavanger, Norway
[2]Halliburton Production Enhancement, 3000 N Sam Houston Parkway E Houston, TX, USA
[3]McDougall School of Petroleum Engineering, The University of Tulsa,
600 South Tucker Av., Tulsa, OK, USA

ABSTRACT

Drilling fluids are time-dependent and exhibit viscoelastic behaviour; i.e., they exhibit both viscous and elastic responses under deformation. Viscoelastic properties of drilling fluids are very important to evaluate gel structure, gel strength, barite sag, hydraulic modelling, and solid suspension. However, elastic properties are still not being properly accounted for in rheological characterization. This study seeks to investigate quantitatively the viscoelastic properties of oil-based drilling fluids and the application of viscoelastic data in drilling operations.

Standard test procedures were designed to measure viscoelastic properties of fifteen oil-based drilling fluids within the linear viscoelastic range. Experiments were performed using an Anton Paar Physica MCR 301 Rheometer. Periodic oscillatory tests were conducted to investigate material functions of fluids, evaluate gel strength, and time and temperature dependence. In oscillatory testing, amplitude sweep tests were used for determining the linear viscoelastic range, and detecting structural stability, gel strength and dynamic yield point. Frequency sweep tests were conducted to obtain storage modulus, loss modulus, complex viscosity and loss tangent. Temperature sweep tests were conducted in the temperature range from 4 to 80°C to obtain the temperature dependence on the internal gel structure of samples. Oscillatory time sweep tests were used to investigate the change and the stability of the structure of the drilling fluids as a function of time and temperature. The gelling time and gelling speed are calculated from time sweep tests. Experimental results are analyzed to evaluate the viscoelasticity of the samples. The results show that the tested fluids exhibit viscoelastic properties and the viscoelasticity of the samples strongly depends on temperature and gelling time. Further, the Cox-Merz rule, an empirical relationship between dynamic and steady-state viscosities, is very useful to estimate steady-state viscosity from complex viscosity and vice versa. It provides valuable information in the structure build-up during steady shear flow. However, data analysis confirms that the tested samples do not obey the Cox-Merz rule. The departure from the Cox-Merz rule of the tested samples is an indication of structural heterogeneities and weak structure build-up; this can be attributed to fluid structure decay. The extended Cox-Merz rule is proposed to correlate steady-state and dynamic viscosities.

The Time-Temperature Superposition Principle was validated to be inapplicable for tested drilling fluids. Frequency sweep data (storage modulus and loss modulus) at

different temperatures are not superimposed when shifted to a reference temperature.

The results of this study provide quantitative information about the dynamic properties of drilling fluids. These give useful insight into the structure, stability, and time and temperature dependence of drilling fluids. The data can be used to evaluate static barite sag, cuttings transport and suspension, and hydraulic modelling and optimization.

INTRODUCTION

Viscoelasticity is the property of materials that exhibit both viscous and elastic characteristics when undergoing deformation. Generally, the particular response of a sample in a given experiment depends on the time-scale of the experiment in relation to a natural time of the material. If the experiment is relatively slow, the sample will appear to be viscous rather than elastic; whereas, if the experiment is relatively fast, the sample will appear to be elastic instead of viscous. Therefore, at intermediate time scales, viscoelastic behavior is observed. Usually, the solid or liquid character of material is evaluated by the Deborah Number. Deborah Number is defined as the ratio of relaxation time (λ) to the experimental time (t_{exp}).

$$De = \frac{\lambda}{t_{exp}} \tag{1}$$

The relaxation time (λ) is a property of material characterizing the rate of inherent rearrangement of the material structure. Low Deborah Numbers indicate liquid like behaviour, whereas high Deborah Numbers indicate solid-like behaviour. When Deborah Number is on the order of unity, the material will behave viscoelastically.

The experimental time in shear flow and in oscillatory shear is defined as the inverse of the shear rate. Hence, in shear flow, the Deborah Number can be define as,

$$De = \frac{\lambda}{t_{exp}} = \lambda\dot{\gamma} \tag{2}$$

And in oscillatory shear, it can be defined as,

$$De = \frac{\lambda}{t_{exp}} = \lambda\gamma_o\omega \tag{3}$$

The response of drilling fluid to the deformation can be classified as viscous, elastic or viscoelastic response depending on the ratio of relaxation time to experimental time. During circulation through the well, the drilling fluid experiences a wide range of shear rates. Shear rates of order 10^3 s^{-1} are encountered in the drillpipe. In the annulus, drilling fluid experiences a shear field of order 10^2 s^{-1}. For normal drilling fluids, this operational time is usually long comparing to the relaxation time of the drilling fluids. Therefore, the steady shear of drilling fluid is strongly governed by its viscosity. However, the viscoelastic effect also may be significant under very high shear rate. Such high shear rate is commonly encountered at the bit nozzles, where the shear rate as high as 10^5 s^{-1} may develop[2]. With this very high shear rate, the operation time ($\dot{\gamma}^{-1}$) can be on the order of the material time for some drilling fluids. The elastic property of drilling fluids has a strong effect on the flow behavior and pressure drop. To characterize the elastic effect at this high shear rate, a nonlinear viscoelastic model for shear flow should be employed.

In common operations, the viscous component are dominant. However, under infinitesimal deformation, the gel structure shows viscoelastic response to the deformation. The pressure transient, pressure peak and pressure delay is a clear evidence of viscoelasticity and gel structure formation of drilling fluids. Therefore, a purely viscous model may not be sufficient to model these phenomena. In other words, a viscoelastic model should be used to characterize the response of drilling fluids in this range of strain.

To fulfill its complex functions such as cuttings transport and solid suspension, drilling fluid is desired to form a gel structure. The gel formation, when fluid is being at rest, helps to keep the solid particles from settling. The settling of the heavy components such as weighting additives, cuttings may result in severe operational problems. The formation of the gel structure of drilling fluids during shear flow is also important in dynamic condition to enhance cutting carrying capacity and reduce dynamic barite sag. Also, gel structure formation helps to prevent fluid invasion into the formation and lost circulation problem. Hence, viscoelasticity is a desired property in drilling operations.

Viscoelastic data obtained from experiments are the key to develop constitutive equations. The current purely viscous models recommended by API, including Yield Power Law, Power Law, Unified models, work relatively well for immediate and high shear rate to model the viscous behavior of drilling fluid. However, these models have failed to model the response of drilling at low shear rate. This may due to the effect of the viscoelastic properties. Many important phenomena related to this range of shear rate may not be modeled properly without a proper model for this viscoelastic range. Such phenomena include the transient start up flow of drilling fluid after the pump is started and pressure delay after circulation is stopped. This helps to predict the pressure peak and pressure profiles in start-up flow. The elastic and thixotropic effects should also be included in the constitutive equation to better modeling the pressure peak and obtain the pressure profiles along the wellbore. Obtaining pressure profiles and predicting pressure peaks in startup circulation are very important in offshore drilling. After a pump-off period, the gel structure in drilling fluids may develop in the wellbore. This property is important for keeping the cuttings in suspension. But it requires a higher pump pressure to initiate the flow when circulation is resumed. The highest pressure to initiate the flow is called the pressure peak. Pressure peaks may cause formation damaged if the pressure window is narrow, especially in drilling deepwater boreholes.

Viscoelastic properties are important data to evaluate the gel formation and gel structure of drilling fluids. Dynamic tests are the standard rheological techniques to investigate the physical structure of the fluid. These tests are indispensable to investigate low-shear rate properties, gel structure and determine gelling time, dynamic yield point and structural stability of drilling fluids. However, viscoelastic properties of drilling fluids have not been comprehensively presented. The drilling industry still lacks a standard test method and test procedure to evaluate experimentally the viscoelastic properties of drilling fluids. Also, the application of viscoelastic data in field operations has not been widely used.

FUNDAMENTALS

Steady-shear viscosity provides useful rheological properties of drilling fluids under large deformation or shear flow. However, many phenomena cannot be described by the viscous property alone. Many processes related to drilling fluids are governed by viscoelastic properties. Under infinitesimal strain in transient gel formation, gel breakage and at rest, drilling fluids show significant viscoelastic response to the deformation. Drilling fluids are commonly not strongly viscoelastic. Therefore, out of the linear viscoelastic range, in nonlinear viscoelastic range, the viscous property is dominant. To obtain the viscoelastic properties of a drilling fluid in the linear viscoelastic range, test methods involved in small deformation are commonly employed. These test methods are called dynamic tests, which can be divided into two major categories: transient and oscillatory. The two most common transient methods are creep-recovery and relaxation tests. The common oscillatory tests used to investigate the viscoelastic properties of materials are amplitude sweep, frequency sweep, oscillatory time sweep, and temperature sweep tests. Oscillatory testing provides a means to probe a material's structural characteristics. Oscillatory tests can detect subtle differences in materials which rotational testing cannot. They provide information on a material's response to both long time and short time processes. The most important feature of these tests is that the strain can be so small that the inherent structures in a sample can be measured without being destroyed. Hence, they are excellent for monitoring time and temperature dependent properties. Oscillatory tests are mainly used in this study.

In an oscillatory experiment a fluid is subjected to a sinusoidal deformation and the resulting fluid response, stress, is measured. The applied shear strain, γ(t), is defined as

$$\gamma(t) = \gamma_o \sin(\omega t) \quad (4)$$

The measured shear stress, τ(t), is:

$$\tau(t) = \tau_o \sin(\omega t + \delta) \quad (5)$$

For a purely viscous fluid, the phase angle (δ) is equal to 90^o. For a purely elastic material, the phase angle is equal to 0^o. And for a viscoelastic material, the phase angle has values between 0^o and 90^o.

Shear stress can be written in term of strain as

$$\tau(t) = \tau_o[\sin(\omega t)\cos\delta + \cos(\omega t)\sin\delta] \quad (6)$$

$$\tau(t) = \gamma_o\left[\left(\frac{\tau_o}{\gamma_o}\cos\delta\right)\sin(\omega t) + \left(\frac{\tau_o}{\gamma_o}\sin\delta\right)\cos(\omega t)\right] \quad (7)$$

$$\tau(t) = \gamma_o[G'\sin(\omega t) + G''\cos(\omega t)] \quad (8)$$

$$G' = \frac{\tau_o}{\gamma_o}\cos\delta \text{ and } G'' = \frac{\tau_o}{\gamma_o}\sin\delta \quad (9)$$

The shear storage modulus or elastic modulus (G'), measures the energy stored per cycle; the lost modulus or viscous modulus (G") measures the energy lost per cycle of sinusoidal deformation.

Oscillatory Amplitude Sweep Tests

In an amplitude sweep test, the amplitude of oscillation is ramped while the frequency is held constant. Under small strain, sample will be deformed viscoelastically when the internal structure is not broken. The strain is increased to a critical strain when the structure of the sample will be irreversibly deformed. Hence the response of the fluid to deformation changes from linear viscoelastic response to nonlinear viscoelastic response.

This test is the first test conducted to determine the linear viscoelastic range, the range of strain (or stress) where G' and G" are constant. Also it is also used to detect structural stability, strength and dynamic yield point of drilling fluids.

Oscillatory Frequency Sweep Tests

In a frequency test, frequency of oscillation is ramped with the amplitude held constant. We change the speed of deforming the sample and monitor the response of the sample in its linear viscoelastic range. This test is used to detect time-dependent properties, and quantifies zero shear viscosity and structural strength at rest. The shape of G' and G" curves over frequency are characteristic of material type.

Oscillatory Time Sweep Tests

Oscillatory time sweep tests directly provide the necessary information about how a material changes with time. Information on dispersion settling, structure development, gelling time and gelling speed, can be obtained. By monitoring certain viscoelastic parameters as time advances, the material's behaviour with time can be monitored directly. In oscillatory time sweep tests, the sample is pre-sheared, and the oscillatory time sweep test is started directly after the pre-shear stopped. During the sweep, the amplitude, frequency and temperature are held constant and properties monitored over time. After the pre-shear finished and the oscillatory stress sweep test is initiated, the material begins to form a structure by an increase in elastic modulus. This is similar to the gel structure of the drilling fluid forms when the circulation is stopped. This gel structure will prevent the cutting and barite from settling. Therefore, measurement of how fast the gel structure of the fluids develop is very useful to evaluate the barite sag and cuttings suspension capability of drilling fluid. A good drilling fluid should have gel structure develop fast enough and stable with time. For drilling fluid testing, this test is also important to find the gelling time of the sample before conducting other tests.

Oscillatory Temperature Sweep Tests

In the oscillatory temperature sweep test, amplitude and frequency are held constant while temperature is ramped. The temperature test is used to measure the temperature dependence of the structure of the sample. It is a means to investigate the stability of the fluid with temperature and monitor the changes in state from liquid to solid, freezing point.

High temperature is often encountered along the well-bore. Hence, increasing the temperature during the test helps to predict the change of structure of drilling fluid along the wellbore. This is very important for barite sag and particle settling evaluation. Severe static barite sag may result from a significant change of fluid structure with the change of temperature. To eliminate the problem related to particle settling, drilling fluid should be stable with the change of temperature. Also to predict the static barite sag in the wellbore, the temperature should be selected based on the wellbore temperature.

By setting the temperature to decrease during an oscillatory temperature sweep tests, we can detect the freezing point of drilling fluid. Therefore, this test is very important test for offshore operation, since the temperature at the sea bed is very low. The melting point of drilling fluids must be higher than the lowest temperature along the wellbore.

Oscillatory temperature sweep test can also be used to detect the formation of wax, hydrate and other phenomena related to the change of temperature. Oscillatory temperature sweep test gives a better prediction of these phenomena than steady shear test method. Since, under very small deformation, structure of the sample does not change significantly so the test does not prevent or accelerate these phenomena.

EXPERIMENTAL INVESTIGATION

The measurements in this study were performed using an Anton Paar Physica MCR 301 rheometer equipped with an electrically heated temperature chamber. Experiments were conducted using three geometries, dual gap cylinder, smooth and rough parallel plate, for cross check.

Both steady-shear and dynamic experiments were conducted on each drilling fluid. For dynamic tests, the first test is the oscillatory amplitude sweep test to find the linear viscoelastic range, and the oscillatory time sweep test is used to find the proper gelling time. Trial and error were used to find the suitable gelling time and linear viscoelastic range.

Steady Shear Experiment

Steady-shear tests were conducted at constant temperature (20 ^oC). Before a test, temperature was set with an accuracy of 0.05 ^oC. Steady-shear experiments were conducted at a shear rate range of 0.001 s^{-1} to 1000 s^{-1}. The transient effect at low shear rate (shear rate smaller than 1 s^{-1}), is eliminated by setting a sufficiently long time to obtain one data point. The first normal stress difference is a characteristic of viscoelastic properties in the nonlinear viscoelastic range. The first normal stress difference is the only nonlinear viscoelastic property investigated in this study.

Dynamic Experiment

Oscillatory Frequency Sweep Tests

The first experiment in dynamic tests is the oscillatory amplitude sweep test to define the linear viscoelastic range. All other dynamic experiments were conducted in the linear viscoelastic range. Amplitude sweep tests were conducted with a constant frequency of 10 s^{-1} and a strain ramp from 0.001 to 1000 %.

Oscillatory Frequency Sweep Tests

Frequency sweep tests were conducted in the linear viscoelastic range. In this study, strains of 0.05% were used. Due to weak gel structure of drilling fluids, the suspension may move down and low density component may stay at the upper layer. This may lead to inaccurate data, especially for measurement with parallel plate. Hence, setting the frequency to sweep from high to low will give more accurate result, and also help to obtain more data points at high frequency after a short time. Hence, in this study, the frequency ramps from 100 to 0.01 in a log scale.

Oscillatory Time Sweep Tests

The oscillatory time sweep test was conducted on a sample right after it was sheared at a constant shear rate of 1000 s^{-1} for 5 minutes. The strain was set to be constant at 0.05 % and a constant frequency of 10 s^{-1}. During the oscillatory time sweep tests, we monitor the change in storage modulus with time. A test is stopped when the storage modulus reaches a stable value.

Oscillatory Temperature Sweep Tests

Temperature sweep tests were conducted with the temperature changing linearly from 4^oC to 80 ^oC, with constant strain and frequency of 0.05 % and 10 s^{-1}, respectively. The heat rate is set to be 1 F/minute. This value was experimentally validated to be sufficient for sample to reach equilibrium. Because the linear viscoelastic range decreases significantly at high temperature, the strain was selected to be very small, 0.05%. The gelling time is also obtained from oscillatory time sweep test.

RESULTS AND DISCUSSIONS

Steady Shear Test

A typical flow and viscosity curves of the drilling fluid is shown in Figure 1. The low shear rate regions for all tested drilling fluids are at very slow shear rate. Even at a very low shear rate (0.001 s^{-1}) many fluids do not reach the Newtonian shear rate region. In other words, for the tested drilling fluids, the zero-shear-rate range may be at very low shear rate. Hence, the lowest shear rate available on standard field viscometer, 5.11 s^{-1}, is not in the zero-shear-rate region of the tested fluids.

The standard rheological models give a good fit to experimental data mediate shear rates. However, these models normally fail to predict the rheological properties at very low and very high shear rates (Fig 1).

The positive first normal stress difference is commonly observed (Fig 2). This stress difference is not very high for some samples. Hence, this is an indication of weak viscoelasticity in the nonlinear viscoelastic range. The negative value of

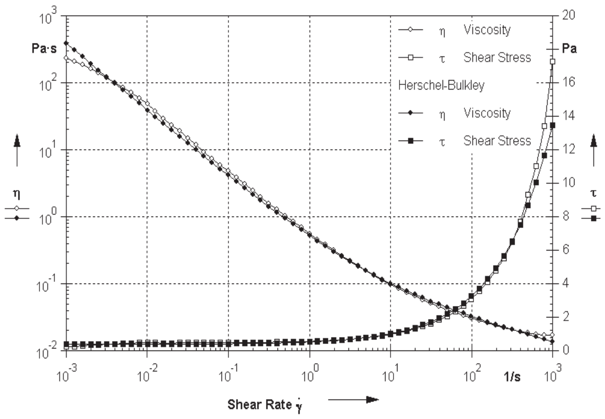

Figure 1. Viscosity and Flow Curves, Fluid 12.

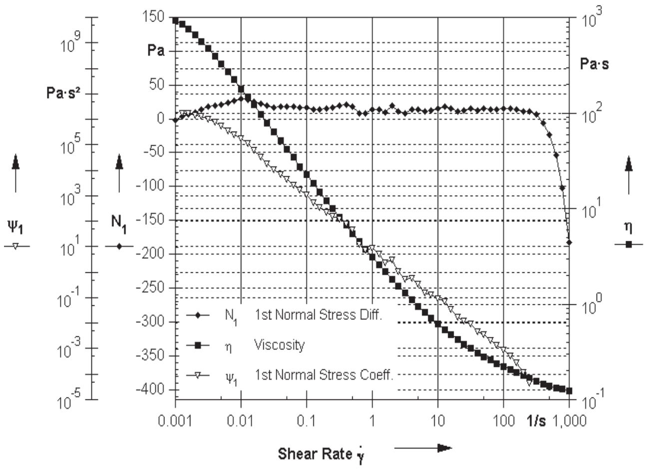

Figure 2. Steady-Shear Test, Fluid 2.

normal stress difference at high shear rate may due to inertia effect, since this test was conducted with the parallel plate geometry. There could be motion in the r-direction due to inertia at very high rotational speeds.

Oscillatory Test

Oscillatory Amplitude Sweep Test

Experimental results show that the liner viscoelastic range of drilling fluids is commonly less than 1% at the frequency of 10 s^{-1} and at 20°C. For some week gel structure samples, the linear viscoelastic range is very small (Figure 3).

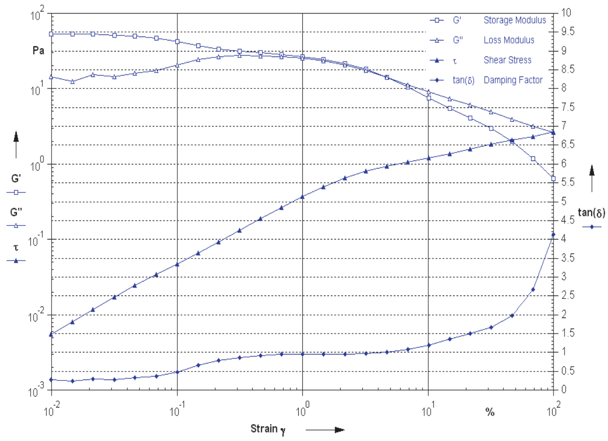

Figure 3. Oscillatory Amplitude Sweep Test, Fluid 9.

The maximum strain of the linear viscoelastic range shows the maximum strain that we can deform a sample without breaking its internal gel structure. Hence, under the deformation, these samples will transform quickly from viscoelastic response to viscous response. In other words, the transient viscoelastic response is very short.

In fact, the cross point (between G' and G") and the linear viscoelastic range may change with frequency. The curves obtained from these tests may have similar form, but cross point and linear viscoelastic range may be different. This can be observed from frequency sweep test data. The linear viscoelastic range and the strain or stress at the cross point between G' and G" are dependent on frequency. These values also strongly depend on gelling time and temperature. In other words, the dynamic yield point varies with frequency, temperature and gelling time. At higher frequency, the linear viscoelastic range and dynamic yield point are typically higher. This means that the gel strength of the fluid is higher when it is under fast deformation. Hence, in practice a slow increase of pump pressure in gel breaking may help reduce the pressure peak.

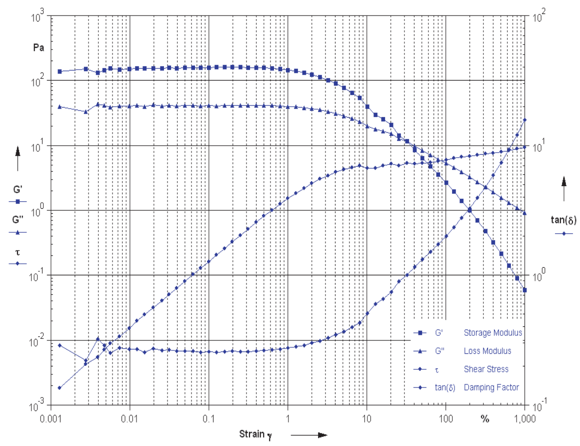

Figure 4. Oscillatory Amplitude Sweep Test, Fluid 4.

In steady shear, the viscoelastic properties of drilling fluid are not significant and the response of the fluid to the deformation is dominated by the viscous property. Hence, the settling of particles in dynamic condition is governed by the viscosity. Viscoelastic properties may not be closely related to the settling of particles in dynamic conditions. However, highly viscoelastic fluids normally show strong gel structure and structure build-up under shearing conditions. Hence, it is somehow related to the dynamic settling of particles. Under static condition, the viscoelastic properties dominate. In the linear viscoelastic range, response of the sample is governed by the viscoelastic properties. A larger viscoelastic range and higher dynamic yield point is sometimes desired to prevent the settling of particles.

Oscillatory Frequency Sweep Test

The viscoelastic property of a drilling fluid in its linear viscoelastic range is shown in the frequency sweep data. This test method shows us information about both viscous and elastic properties. Since the inverse of frequency is time, frequency sweep tests are used to investigate time-dependent deformation behaviour. High frequency corresponds to fast deformation and low frequency corresponds to slow deformation. Hence, one can look at frequency sweep data to evaluate if the response of the sample to deformation at a certain speed of deformation is a viscous response or elastic response. At a given frequency, if storage modulus (G') is higher than loss modulus (G"), the response of the sample to deformation is dominated by elasticity and the sample behaves more elastically. If the storage modulus is smaller than the loss modulus, the sample behaves more viscously.

Experimental data (Figure 5) show that for most of the tested fluids, the elastic modulus (G') is nearly independent of frequency. In the investigated range of frequency storage frequency (G') is greater than loss modulus (G"). This is an indication of a stable gel structure or a solid-like property. This is a characterization of viscoelastic solid. In other words, the response of the sample to the deformation is dominated by elastic behaviour. For drilling fluids, a stable structure is important to keep small particles in suspension.

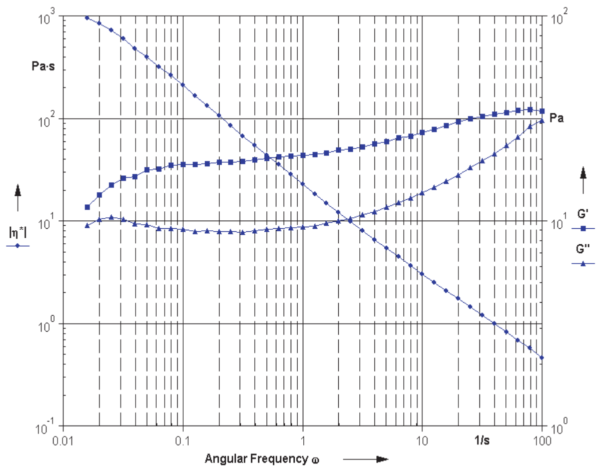

Figure 5. Oscillatory Frequency Sweep Test, Fluid 1.

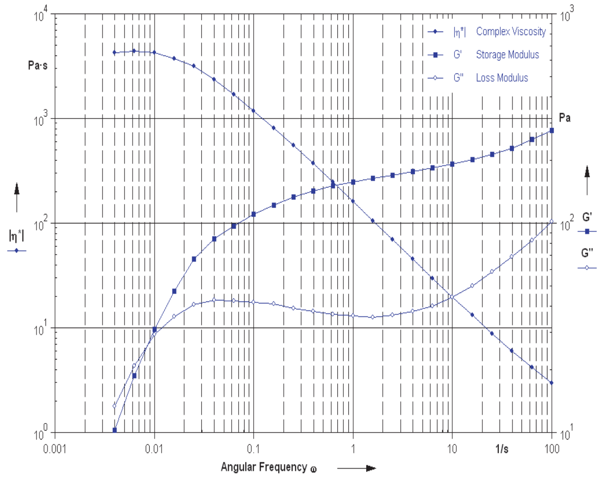

Figure 6. Oscillatory Frequency Sweep Test, Fluid 7.

For some of the samples, elastic modulus (G’) shows a strong dependence on the frequency. The storage increases as frequency increases (Figure 6). At low frequency, the storage modulus (G’) is even smaller than the loss modulus (G”). This is an indication of liquid-like property. The frequency at which the storage modulus is smaller than the loss modulus is the transition from more solid-like to more liquid-like behaviour. This means that in linear viscoelastic range, under relatively slow deformation, the sample is more viscous and under relatively fast deformation, i.e. the sample is more elastic.

Oscillatory Time Sweep Test

The rapid increase of dynamic viscosity with the gelling time, as shown in Figure 7, is the reason why dynamic barite sag is more severe than static barite sag. In other words, the gel structure of drilling fluids and the rapid formation of gel structure reduces the settling of particles when the fluid is at rest. Hence, settling of particles such as barite and cuttings under the static conditions is governed by viscoelastic properties. It should be noted that this holds true if the particles small enough. Thus the shear stress that particles create on the fluid is in the linear viscoelastic range. In this range of shear stress or strain, the response of drilling fluids to deformation generated by the particle is a viscoelastic response. Hence, the settling of these small particles is governed by the viscoelastic properties of drilling fluids. The settling of particles that create a shear stress on higher than the limit of the viscoelastic range of the fluid is governed by the viscous property of the fluid.

Due to the change of the gel structure of the sample with time, to obtain reproducible test results, the oscillatory time sweep test should be conducted first. This test will provide the final gelling time. In fact, the structure of the sample changes continuously with gelling time and with different speeds due to the settling of small particles and the relaxation of the gel structure. This is commonly observed in weak gel structure samples (Figure 8). Hence, it is very difficult to define an absolutely gelling time for some samples. For practical purpose, we use the gelling time as the time when the sample reaches a nearly stable gel structure. In other words, it is the time that the storage modulus reaches a stable value.

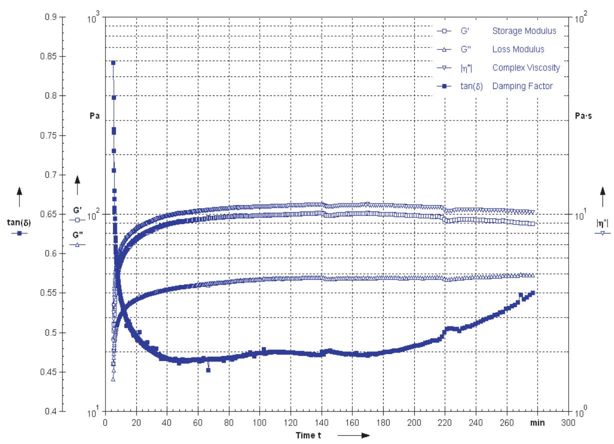

Figure 7. Oscillatory Time Sweep Test, Fluid 4.

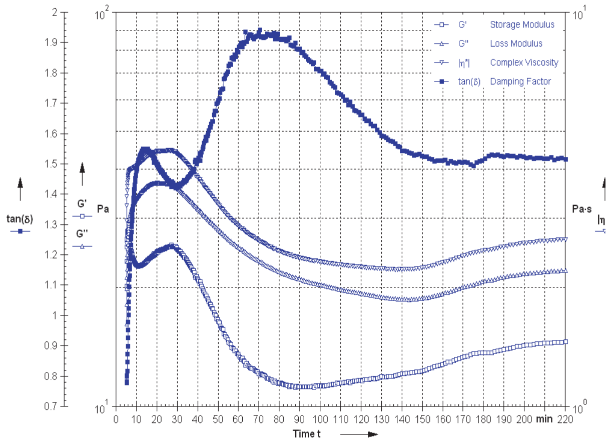

Figure 8. Oscillatory Time Sweep Test, Fluid 8.

For strong gel structure samples, storage will reach its highest value and remain stable (Figure 9). However, for some samples, the storage modulus increases and then gradually decreases. Thus, the gelling time is not always the same as the time needed by a sample to reach its highest gel strength.

This gelling time is the time that we set for the samples at rest before all dynamic tests. By doing this, we set samples at the same initial condition and also avoid errors in measurement due to change of gel structure with time, as discussed above.

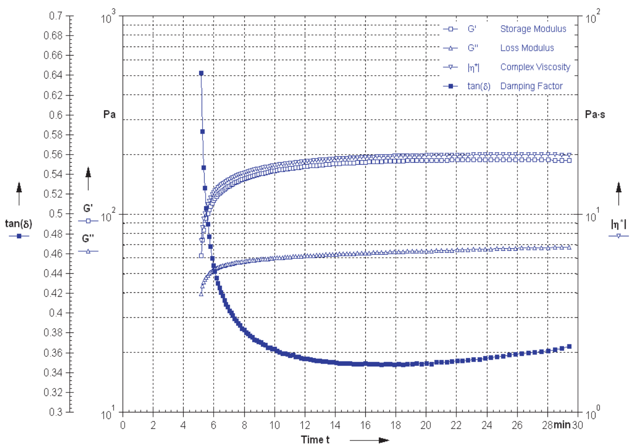

Figure 9. Oscillatory Time Sweep Test, Fluid 16.

A fast recovery and stable structure after being at rest is commonly observed in the test results. However, some drilling fluids show unstable gel structure after being at rest. The storage modulus has a tendency to decrease after it reaches to a peak value and it reaches a nearly stable value after a long time. This could be due to the settling of particles and the relaxation of structure when the sample is at rest. The decrease of storage is the indication of an unstable and weak gel structure. Hence, it is a good indication of static barite sag. Also experimental results show that for some sample the gelling time of 30 minutes is not sufficient to reach the final gel structure. Hence, the gelling time of 30 minutes, as recommended by API, should be applied with care especially for weak gel sample.

Oscillatory Temperature Sweep Test

An unusual change of the viscoelastic properties of the sample with the increase of temperature is observed for some samples (Figure 10).

The complex change of the visocelastic data with the change in temperature is an indication that the rheological properties of the fluids do not change monotonically with the change in temperature. This could be due to the complex components in the fluids and the rheological properties of each component can vary differently with temperature. Hence, rheological characterization at room temperature may not be accurate for characterizing rheological properties at downhole conditions.

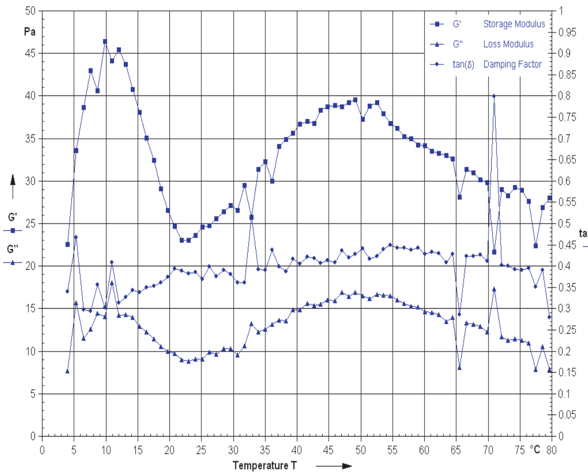

Figure 10. Oscillatory Temperature Sweep Test, Fluid 14.

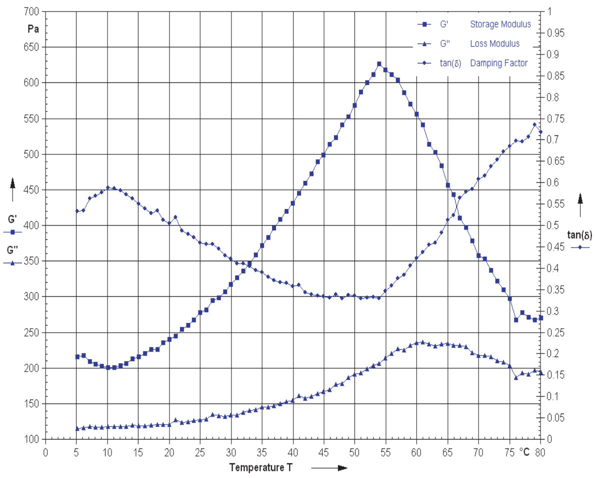

Figure 11. Oscillatory Temperature Sweep Test, Fluid 6.

When temperature is decreasing the storage modulus (G') normally increases simultaneously as the loss tangent (tan δ) decreases (Figure 12). This means that the drilling fluid changes from liquid towards more elastic. The very high value of storage modulus at low temperature is an indication of melting point. This freezing temperature is very important in deepwater drilling. To prevent the change of rheological properties along the wellbore, the stability of the drilling fluid with temperature is usually desired.

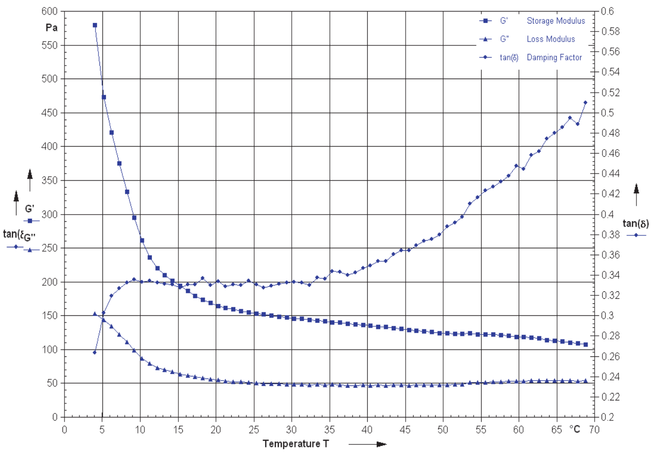

Figure 12. Oscillatory Temperature Sweep Test, Fluid 1.

The Extended Cox-Merz Rule

Cox and Merz[20] (1958) observed that the complex viscosity is nearly equal to the steady shear viscosity when shear rate and frequency are equal.

$$\eta(\omega) = \sqrt{\left[\left(\frac{G'}{\omega}\right)^2 + \left(\frac{G''}{\omega}\right)^2\right]}\Bigg|_{\omega=\dot{\gamma}} = \eta(\dot{\gamma})\Big|_{\omega=\dot{\gamma}} \qquad (10)$$

This empirical relationship is referred to as the "Cox-Merz rule". It is very useful for materials that are more easily tested under oscillatory test than steady-shear conditions. It is also an indication of structure decay of a fluid. The Cox-Merz rule works very well for many structured fluids, such as polymeric fluids. However, many viscoelastic systems do not obey this empirical rule. Therefore, many attempts have been made to extend it. Following are some common forms of an extended Cox-Merz rule[21]:

The modified Cox-Merz Rule

$$\eta(\omega) = \eta(c\dot{\gamma})\Big|_{\omega=\dot{\gamma}} \qquad (11)$$

and the generalized Cox-Merz Rule:

$$\eta(\dot{\gamma}) = k\eta^*(\alpha\omega)\Big|_{\omega=\dot{\gamma}} \tag{12}$$

To verify the application of the Cox-Merz Rule for drilling fluids, we plot dynamic and steady shear viscosities on the same graph with frequency equal to shear rate. It is observed dynamic viscosity is normally higher than steady shear viscosity and two curves do not overlap. Hence, this sample does not follow the Cox-Merz Rule. The deviations from the Cox-Merz Rule are attributed to structure decay due to the effect of strain deformation applied to a fluid that is low in oscillatory shear but sufficiently high in steady shear to break down intermolecular associations.

The available Extended Cox-Merz Rules in literature do not work well for the tested drilling fluids. Therefore, we propose the Extended Cox-Merz Rule in the following form to correlate the dynamic and steady shear viscosities.

$$\eta(\dot{\gamma}) = \left[\eta^*(\alpha\omega)\right]^k\Big|_{\omega=\dot{\gamma}} \tag{13}$$

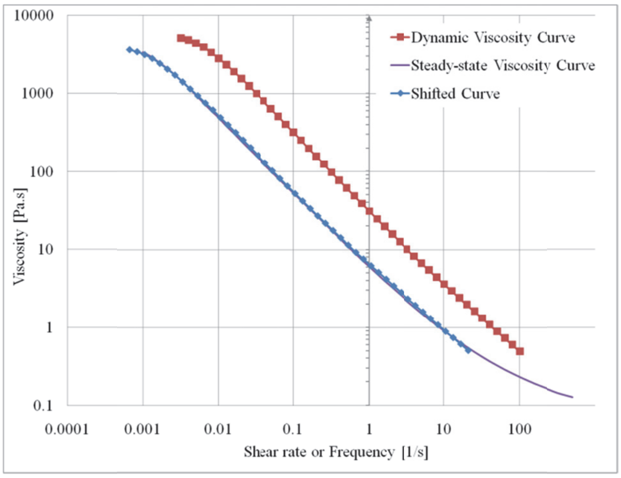

Figure 13. Correlation of dynamic and steady shear viscosities for Fluid 15. (α = 0.21 and k = 0.96).

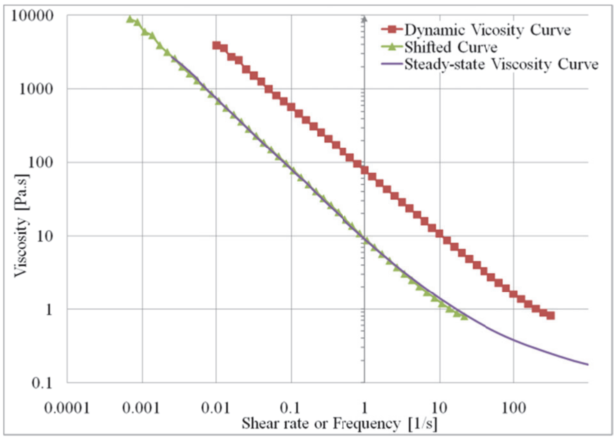

Figure 14. Correlation of dynamic and steady shear viscosities for Fluid 5, α = 0.068 and k=1.1.

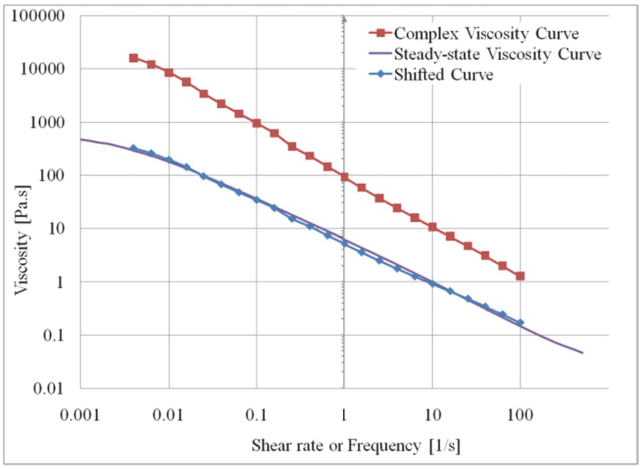

Figure 15. Correlation of dynamic and steady shear viscosities for Fluid 16 (k = 0.21 and α = 0.96).

The coefficients k and α can be called vertical and horizontal shift factors, respectively. The shift factors are obtained by trial and error procedure. Experimental data show that this correlation gives a close fit for 15 samples in this study (Figure 13).

It can be observed that the proposed Extended Cox-Merz Rule is not very applicable at high frequency (Figure 14).

Only Fluid 16 (Figure 15), does not show a good fit to this model. They fit better to the Generalized Cox-Merz Rule (Eq. 12)

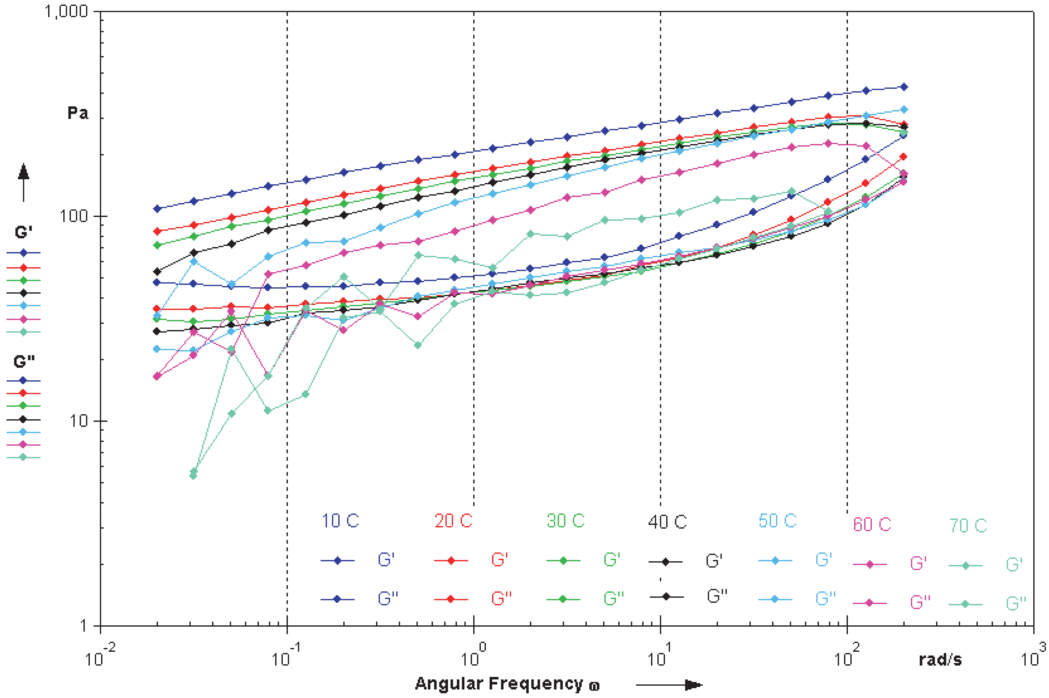

Figure 16. Frequency Sweep Test at Different Temperatures, Fluid 1.

<u>Time - Temperature Superposition Principle</u>

Frequency sweep experiments were conducted at constant temperature. They provide us with the-time dependent properties of the sample at constant temperature. However, we also want to know the response of the sample at different temperatures or different ranges of frequency. Very low frequency data provide the long term stability of the sample. However, the time required to obtain one data point in low frequency range is very long. Hence, it is not practical in measurement. High frequency data characterizes the response of a sample in very fast deformation. Sometimes this range of frequency may be out of the measurement window of the rheometer. Very high and very low temperature can also be out of the measurement window. To overcome these experimental difficulties, the Time and Temperature Superposition Principle was investigated. If the sample is thermorheologically simple, by shifting the modulus curve, one may obtain the viscoelastic properties of the sample at other temperatures and frequency ranges.

To validate if the sample is thermorheologically simple or not, in this study the frequency sweep test was conducted at different temperatures in the linear viscoelastic range. If the storage modulus and loss modulus curves at different temperatures can be shifted to a reference curve, then the sample is thermorheologically simple. However, when all curves were shifted to a reference curve, they do not superimpose (Figure 16). Therefore drilling fluids in this study are thermorheologically complex. For some samples, at temperatures lower than 40 ^{o}C, the shifted curves do superimpose. In other words, the Time and Temperature Superposition Principle is applicable for some samples at low temperatures. However, in the range of low temperatures, this principle does not work well for both storage modulus and loss modulus.

Deviation from the Time and Temperature Superposition Principle of the tested drilling fluids shows that the rheological properties of the fluids do not change monotonically with the change in temperature. This could be due to the complex components in the fluids and the rheological properties of each component can vary differently with temperature. Also, the very small range of the linear viscoelastic region makes it difficult to apply this principle.

SUMMARY AND REMARKS

This research focuses on the linear viscoelastic range of drilling fluids. The standard rheological tests and test procedures have been designed to evaluate experimentally the viscoelastic properties of drilling fluids. The viscoelastic properties in linear viscoelastic range were investigated using periodic oscillatory tests.

The linear viscoelastic ranges sixteen different drilling fluids were obtained from amplitude sweep test. For tested drilling fluids, the linear viscoelastic range is relatively small, less than 1% at 20 ^{o}C and frequency equal to 10 s^{-1}. Linear viscoelastic range, gel strength and dynamic yield stress decreases as temperature increases and frequency decreases.

The frequency sweep data, in the linear viscoelastic range, show that elastic modulus (G') is commonly higher than viscous modulus (G") in the tested frequency range. This is evidence that elastic property is dominated. Viscous property is commonly dominated at low frequency in linear viscoelastic range.

The gel structure formation of each sample was obtained by the oscillatory time sweep test. The structure of the sample develops very fast right after at rest and a different tendencies after this fast changing were observed. The time required for the gel structure of the sample to reach the stable structure is sometimes higher than the time recommended by API for determining gel strength (30 minutes).

The change of the structure of the sample with temperature was obtained from oscillatory temperature sweep test. Temperature has a significant effect on the viscoelastic parameter of the fluids. Storage modulus, loss modulus and complex viscosity and the linear viscoelastic range normally decrease as temperature increases. However both decrease and increase tendency were observed as temperature increases. Hence, the structure of the sample does not change monotonically with the change of temperature.

None of the tested drilling fluids follows the Cox-Merz rule. The complex viscosity is usually higher than the steady shear viscosity. That is the indication of structure heterogeneities of drilling fluids. The extended Cox-Merz rule with the new form: $\eta(\dot{\gamma}) = [\eta^*(\alpha\omega)]^k|_{\omega=\dot{\gamma}}$ was proposed to correlate dynamic and steady shear viscosities.

Time-Temperature Superposition Principle is not applicable for the tested drilling fluid in wide range of temperatures. Therefore, tested drilling fluids are not thermorheologically simple.

ACKNOWLEDGMENTS

Authors would like to thank all company members of The University of Tulsa Drilling Research Project (TUDRP) for allowing the publication of this research.

REFERENCES

1. Reiner, M. (1964) "The Deborah Number" *Physics Today,* **17**.
2. Tehrani, A. (2007), "Behavior of Suspensions and Emulsions in Drilling Fluids" *Annual Transactions Of The Nordic Rheology Society*, **15**.
3. Saasen, A., Liu. D., C.D. Marken and Isambourg, P. (1996), "Prediction of Barite Sag Potential of Drilling Fluids from Rheological Measurement" Paper SPE/IADC 29410.
4. Tehrani, A., Zamora, M. and Power, D. (2004), "Role of Rheology in Barite Sag in SBM and OBM", paper AADE-04-DF-HO-22, Texas.
5. Maxey, J. (2006),"Rheological Analysis of Oilfield Drilling Fluids" Paper AADE-06-DF-HO-01, Texas.
6. Dye, W., Hemphill, T., Gusler, W., Mullen, G. (2001.), "Correlation of Ultralow-Shear-Rate Viscosity and

Dynamic Barite Sag", *Journal of Petroleum Engineering,* SPE-70128-PA.
7. Martins, A.L., Waldmann, A.T.A., Ribeiro, D.C. and Massarani, G. (2005) "The Conceptual Design of a Non-Invasive Solids Free Drill-in Fluid", Paper SPE 94287, Spain.
8. Blanco, J., Ocando, D., Lanza, A., Rendon, R., Rodríguez, F., Caligiore, A., Acosta, J., and Carrero, N. (2007), "Viscoelastic System as an Alternative to UBD for Drilling for a Severely Fractured Limestone Reservoir" SPE 109203, *SPE Annual Conference and Exhibition.*
9. Khan, R., Kuru, E., Tremblay, B., and Saasen, A. (2007), "Extensional Viscosity of Polymer Based Fluids as a Possible Cause of Internal Cake Formation" *Energy Source*, Part A, **29**,1521-1528.
10. Kakadjian, S., Rauseo, O. and Mejias, F. (1999), "Dynamic Rheology as a Method for Quantify Gel Strength of Water Shutoff Systems" paper SPE 50751.
11. Gandelman, R.A. and Leal, R.F. (2002), "Study on Gelation and Freezing Phenomena of Synthetic Drilling Fluids in Ultradeepwater Environments" paper SPE 105881.
12. Herzhaft, B. and Ragouillaux, A. (2006), "How To Unify Low-Shear-Rate Rheology and Gel Properties of Drilling Muds: A Transient Rheological and Structural Model for Complex Wells Applications", paper SPE 99080-MS.
13. Riande, E., Díaz-Calleja, R., Prologo, M.G., Masegosa, R.M., Salom, C., Dekker, M. (2000), "Polymer Viscoelasticity Stress and Strain in Practice", CRC Press.
14. Steffe, J.F. (1996) "Rheological Methods in Food Process Engineering", Freeman Press, 2nd Edition.
15. Barnes, H.A., Hutton, J.F. and Walters, K. (1993), "An Introduction to Rheology", Elsevier Science Publisher, Third Impression, Netherland.
16. Macosko, C.W. (1994), "Rheology Principles, Measurements, and Applications", Wiley-VCH, Inc.
17. Morrison, F.A. (2001), "Understanding Rheology", Oxford University Press.
18. Gebrueder, G.S. (2000), Karlsruhe 2000 "A Practical Approach to Rheology and Rheometry", HAAKE GmbH, Federal Republic of Germany.
19. Mezger, T.G. (2011), "The Rheology Handbook", 3rd Revised Edition, European Coating Tech Files.
20. Cox, W.P. and Merz, E.H. (1958), "Correlation of Dynamic and Steady-Flow Viscosities", *Journal of Polymer Science*, **28**.
21. Gunasekaran. S. and Ak, M.M. (2000), "Dynamic oscillatory shear testing of foods – selected applications", *Trends in Food Science and Technology,* Volume 11 Issue 3.
22. Bui. B. (2012), "Determination of Viscoelastic Properties of Drilling Fluids", M.Sc. Thesis, The University of Tulsa, Tulsa, USA.