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INTRODUCTION

Success of a drilling, workover or completion fluid can be directly attributed to its viscosity at the shear rate of interest. In practice, fluids are subjected to a wide range of shear rates; therefore, a thorough understanding of fluid rheology and the impact of shear is necessary to optimize fluid design. Ideally, high viscosity is desirable only under low shear rate conditions, and should decrease as flow rate increases. Among the viscosifiers used, biopolymers exhibit unique rheological properties including increased low shear rate viscosity and an extended power law region. These differences stress the importance of understanding the rheological behavior of fluids in order to maximize performance and evaluate the relative merits of each fluid.

DEFINITION OF TERMS

Most drilling, workover and completion operations involve the movement of fluids. The study of flow and deformation of matter, including liquids and solids, is called rheology. To better understand the impact of rheological properties, Figure 1 can be used to provide a definition of some basic rheological terms.

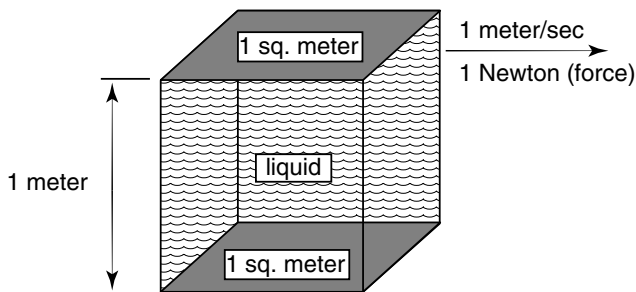


Figure 1. Diagram for Rheological Definitions

This illustration depicts the forces acting on a liquid between two 1 square meter plates, which are separated by 1 meter. The bottom plate is stationary and the top plate is moved at a rate of 1 meter per second. The amount of force required to do this is measured in Newtons. Shear Stress (τ) is defined as the force required to move a given area of the fluid. In this case one Newton is required for each square meter of area.

The units of shear stress are Newtons per square meter, also known as Pascals. Alternative units for shear stress are dynes per square centimeter and pounds force per square inch. Shear Rate ($\dot{\gamma}$) is defined as the rate of movement of the fluid between the plates. It is determined by dividing the velocity difference between the plates by the distance between them. This can also be called the velocity gradient. In this case, it is one meter per second per meter of fluid and is thus measured in reciprocal seconds (sec^{-1}). Viscosity (η) is defined as the ratio of shear stress over shear rate. Consequently, the units are Newton seconds per square meter or Pascal seconds. Another common unit of viscosity is the Poise ($\text{dyne}\cdot\text{second}/\text{centimeter}^2$). One centiPoise is equal to one milliPascal second ($1 \text{ cP} = 1 \text{ mPa}\cdot\text{s}$). Stated another way, 1000 cP is equal to 1 Pa·s. NOTE: in oilfield literature, (η) is most often substituted with (μ) to designate viscosity.

FUNCTIONS OF RHEOLOGY

The rheological characteristics of a fluid are important in evaluating the ability of a fluid to perform a specific function. Oil field fluids are often used to transport solids, suspend solids, reduce pressure drop, or control fluid loss. Examples of how flow properties affect these functions include Stokes' law, which states that the settling velocity of a particle depends directly on the viscosity of the fluid. In order to transport a solid vertically, the upward velocity of the particle must be greater than the settling velocity of the particle. To suspend a solid, the shear stress exerted by the particle must be less than the shear stress provided by the fluid. Certain high molecular weight polymers are used to reduce friction pressure losses of a base fluid and improve flow rates. Darcy's law states that fluid loss is inversely proportional to the viscosity, so higher viscosities decrease fluid loss to the formation.

Rheological characteristics of a fluid can be radically modified through the addition of a polymer. By choosing the proper polymer and polymer concentration, flow properties can be optimized for a given application to achieve maximum results.

GENERALIZED FLOW CURVES

Figure 2 illustrates the viscosity versus shear rate curve for water, a simple Newtonian fluid. For a Newtonian fluid, viscosity is independent of shear rate.

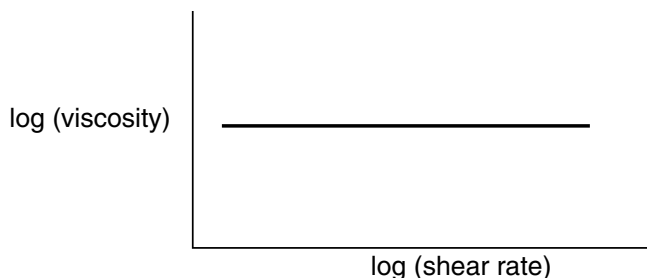


Figure 2. Viscosity of a Simple Newtonian Fluid.

When a water-soluble polymer is added to water, the curve changes dramatically as indicated in Figure 3.

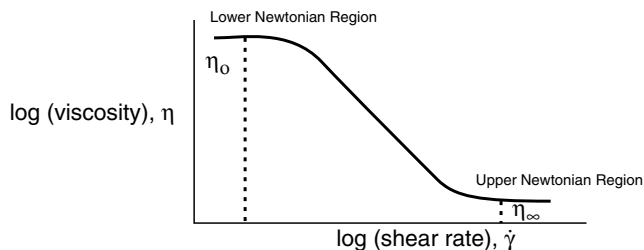


Figure 3. Viscosity of a Non-Newtonian Fluid.

Of significance, there is usually a region at both low and high shear rates where viscosity is independent or nearly independent of shear rate, and a section in between that exhibits strong shear rate dependence. For reasons that will become apparent later, this middle region is usually referred to as the power law region. In some fluids, it can be difficult to reach shear rates that are high or low enough to observe the upper and lower Newtonian regions.

Fluids are generally classified as Newtonian (shear rate independent) or non-Newtonian (shear rate dependent). Newtonian fluids follow a simple relationship between shear stress and shear rate (Equation 1). Their viscosity is a constant as described by the slope of the line on a linear plot of shear stress versus shear rate (Figure 2). Air, water and light hydrocarbon oils are examples of Newtonian fluids.

$$\tau = \mu \dot{\gamma} \dots\dots\dots (1)$$

The viscosity of Non-Newtonian Fluids at a specific shear rate can also be defined by Equation 1; however, for these fluids, viscosity will vary dependent on the shear rate.

RHEOLOGICAL MODELS

Several mathematical models have been developed to describe the shear stress/shear rate relationship of non-Newtonian fluids. These models are used to characterize flow properties in an effort to determine the ability of a fluid to perform specific functions. Misapplication of rheological data can result in an over-simplification or exaggeration of fluid features, accompanied by the failure to perform a specific task. In order to optimize fluid performance, an in-depth discussion of data acquisition, rheological models and their inherent limitations is necessary.

Rheological evaluation of oil field fluids is generally accomplished using concentric cylinder viscometers. Typically, these instruments provide a limited number of shear rates ranging from 5.1 to 1022 sec⁻¹. Data generated with these instruments are analyzed using empirical models developed to describe the flow of non-Newtonian fluids. The most frequently applied models are the Bingham Plastic and Power Law models. While the Bingham model may have sufficed during the evolution of clay based drilling fluids, it is deficient when describing the overall rheological profile of polymer based systems. Furthermore, the presence of an upper and lower Newtonian region, coupled with a region of power law behavior (Figure 3), make the interpretation and application of rheological data a challenging task. This is further complicated by the fact that the onset of Newtonian behavior varies, depending on the viscosifier or fluid system under consideration (Figure 4).

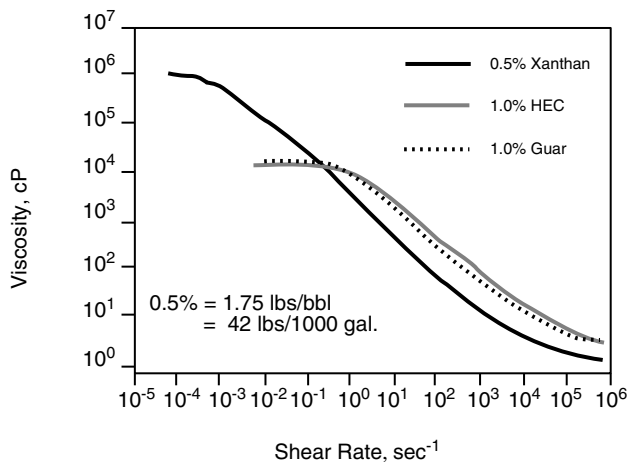


Figure 4. Viscosity Profiles of Xanthan, HEC and Guar, 75°F, 2% KCl

POWER LAW MODEL

One of the most widely used models for describing the behavior of oil field fluids is the power law model (Bird, Armstrong et al. 1977). This model (Equation 2) is valid for the linear, i.e., center section, of the curve shown in Figure 3. In the power law model, the viscosity term from the Newtonian model is replaced with a constant,

K, termed the consistency index, which serves as a viscosity index of the system. The consistency index has the unusual set of units, Force-secⁿ/Area. In addition, the shear rate term is raised to the nth power, thus the term power law. The factor, n, is called the flow behavior index which indicates the tendency of the fluid to shear thin. As the value of the flow behavior index deviates from one, the fluid becomes increasingly non-Newtonian. In oil field (especially fracturing) literature, n and K are usually expressed as n' and K'. The exponent n is equal to n' and K' is equal to K times a geometry correction factor. Because the equations discussed here are given as general examples, n and K will be used. All polymer solutions exhibit power law behavior over part of the shear rate range.

$$\tau = K\dot{\gamma}^n \quad \dots\dots\dots (2)$$

This equation can also be expressed in terms of apparent viscosity, (η_a).

$$\eta_a = K\dot{\gamma}^{n-1} \quad \dots\dots\dots (3)$$

When the log of the shear stress is plotted against the log of the shear rate (Figure 5), a straight line with a slope equal to n and an intercept (at a shear rate of one) equal to log K results. A plot of the log of the viscosity versus log of the shear rate also results in a straight line.

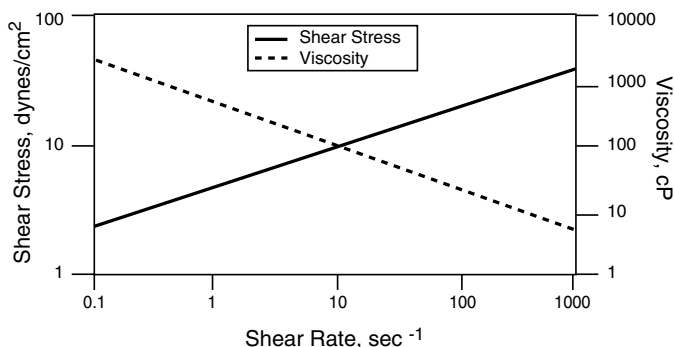


Figure 5. Idealized Power Law Fluid.

Most polymer solutions are pseudoplastic. In this case, increased shear rate causes a progressive decrease in viscosity. This is due to alignment of the polymer chains along the flow lines. For pseudoplastic fluids, the value of the flow behavior index, n, ranges from about 0.1 to <1.0. When the n value is equal to 1.0, the power law reduces to the Newtonian model. The further that n is reduced from 1.0 the more the fluid deviates from Newtonian behavior. Although this is one of the most popular models used, no known fluid exhibits power law behavior over the entire range of shear rate conditions.

Therefore, the primary drawback is the limited shear rate range over which it is valid. Care must be taken to use data within the power law region to accurately calculate power law parameters (n and K). For example, with xanthan gum, calculating n and K from 600 rpm (1022 sec⁻¹) and 300 rpm (511 sec⁻¹) on a concentric cylinder viscometer, will result in inaccurately high n and low K values as compared to values obtained at lower shear rates. As a result, at shear rates below 1 sec⁻¹, predicted viscosities using these inaccurate n and K values will be much lower than measured values (Figure 6).

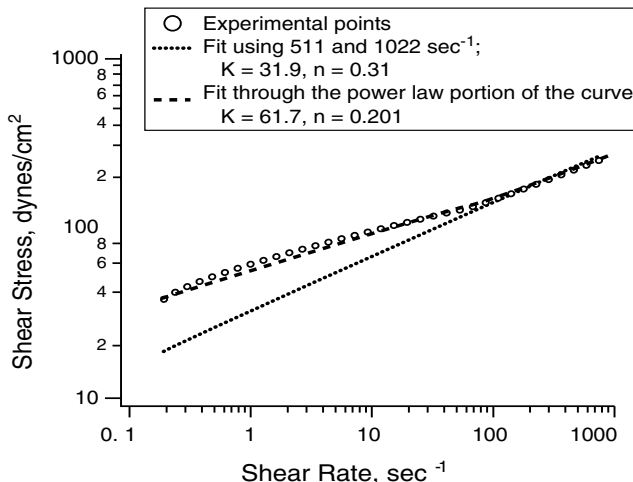


Figure 6. Application of Power Law for 2.5 lb/bbl Xanthan Gum in 3% KCl Using Data from 1022 and 511 sec⁻¹

For comparison purposes, the power law model was used to fit 2.5 lb/bbl HEC as shown in Figure 7. This illustration indicates the inaccuracy of the model to predict flow properties of HEC at low shear rates, due to the onset of the lower Newtonian region (<100 sec⁻¹).

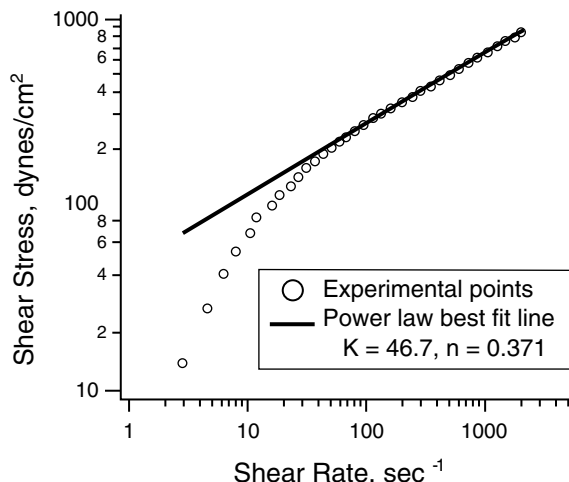


Figure 7. Power Law Fit for 2.5 lb/bbl HEC in 3% KCl

These examples illustrate the need to apply this model within the power law region of a particular fluid to assure accurate predictions of viscosity. The power law model cannot be used to predict viscosities at shear rates in the upper and lower Newtonian regions.

BINGHAM PLASTIC MODEL

The other two parameter model which has been widely used in drilling fluid applications, is the Bingham Plastic model (Equation 4). Fluids that exhibit Bingham Plastic behavior are characterized by a yield point (τ_0) and a plastic viscosity (μ_p) that is independent of the shear rate (Figure 8).

$$\tau = \tau_0 + \mu_p \dot{\gamma} \dots\dots\dots (4)$$

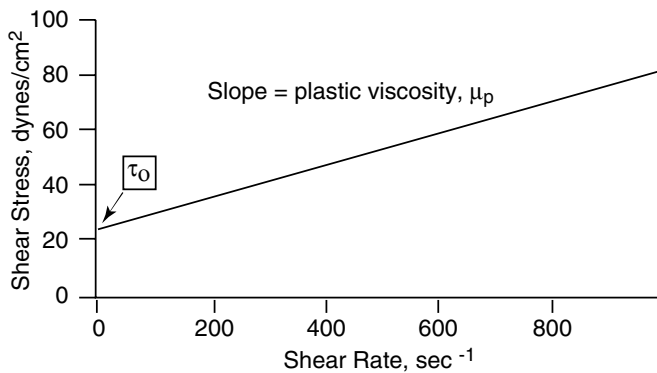


Figure 8. Idealized Bingham Plastic Fluid.

The presence of a yield stress means that a certain critical shear stress must be exceeded before flow can begin. If the fluid exhibits Newtonian flow after the yield value is exceeded, it is called a Bingham Plastic fluid. While some fluids exhibit a yield point, most oil field fluids show shear rate dependence after flow is initiated. This model is interesting but not very useful in describing the behavior of polymer based fluids. In the oil field, the Bingham Plastic model has been used to describe the flow behavior of some clay based drilling muds and a few cement slurries. An extension of the Bingham Plastic model to include shear rate dependence is the Herschel-Bulkley model, described later in the text.

There are several rheological models that involve the use of three or more adjustable parameters. It is necessary to include a third parameter to describe the flow of the fluids in the upper or lower Newtonian region as well as the power law region.

CASSON MODEL

The Casson model (Equation 5) has been used to describe the behavior of drilling fluids (Lauzon and Reid 1979). In general, it has limited application because of the difficulty in using it as a model to predict pressure losses.

$$\tau^{1/2} - \tau_0^{1/2} = \eta_{\infty}^{1/2} \dot{\gamma}^{1/2} \dots\dots\dots (5)$$

Plots of $\tau^{1/2}$ versus $\dot{\gamma}^{1/2}$ yield an intercept of $\tau_0^{1/2}$ and a slope of $\eta^{1/2}$ (Figure 9).

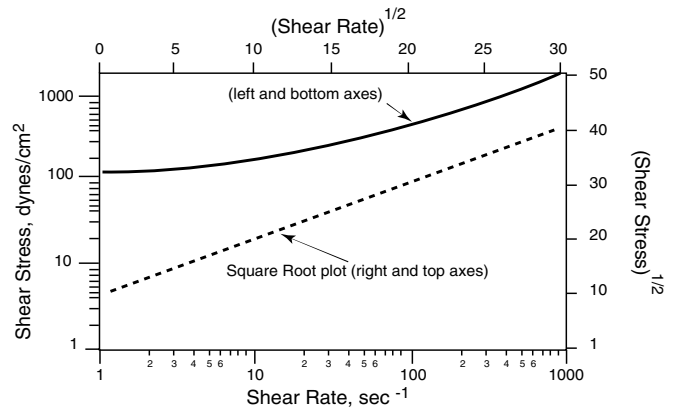


Figure 9. Idealized Casson Model Behavior.

HERSCHEL-BULKLEY MODEL

Fluids that exhibit a yield point and viscosity that is stress or strain rate dependent cannot be adequately described by the Bingham Plastic model. The Herschel-Bulkley model (Figure 10) corrects this deficiency by replacing the plastic viscosity term in the Bingham model with a power law expression (Equation 6).

$$\tau = \tau_0 + K \dot{\gamma}^n \dots\dots\dots (6)$$

This model is useful for describing a wide range of fluids used in oil field applications. The model is reduced to the power law if there is no yield point and to the Bingham model if n is equal to one. The primary limitation of this and all other models that cannot be easily linearized is curve fitting to evaluate model parameters. Computers and good non-linear curve fitting techniques have overcome this limitation. Recent publications (Clark, Sundaram et al. 1991; and Hemphill, Campos et al. 1993) have shown the usefulness of the three parameter Herschel-Bulkley model for drilling fluids.

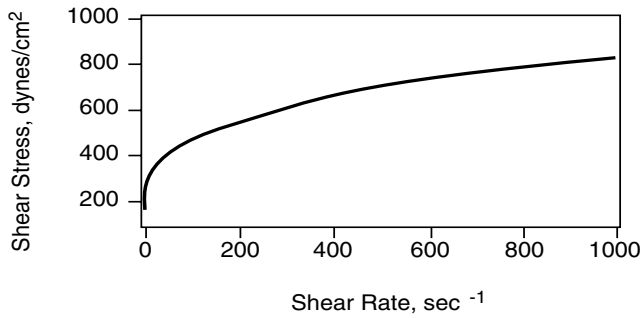


Figure 10. Idealized Herschel-Bulkley Fluid.

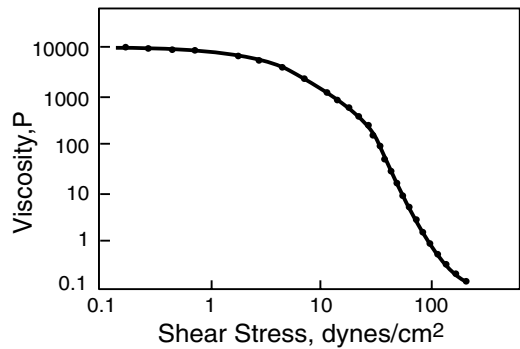


Figure 12. Viscosity as a Function of Shear Stress for Xanthan, 1.75 lb/bbl, 2% KCl

ELLIS MODEL

In all previously discussed models, viscosity or shear stress has been expressed as a function of shear rate. The Ellis model (Equation 7) expresses viscosity as a function of shear stress (Figure 11).

$$\frac{\eta_o}{\tau} = 1 + \left(\frac{\tau}{\tau_{1/2}} \right)^{\alpha-1} \dots\dots\dots(7)$$

In this equation, α is equal to $1/n$ and $\tau_{1/2}$ is the shear stress at η equal to $\eta_o/2$. Plotting $\log \eta_o/(\eta - 1)$ versus $\log \tau/\tau_{1/2}$ yields a straight line with a slope of $(\alpha - 1)$.

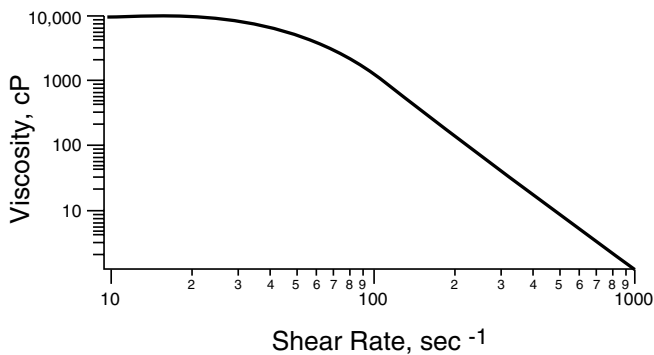


Figure 11. Idealized Ellis Model Fluid.

There are many examples in literature where the three parameter Ellis model is used to describe the behavior of fluids that exhibit a lower Newtonian region followed by a power law region. While viscosity can be expressed as a function of shear stress, it is not commonly done in oil field applications, since most data is obtained from instruments that use shear stress as the dependent variable. Controlled stress instruments are finding wider use, but these instruments normally require a compressed air flow and do not work well outside a laboratory. Plots of $\log(\text{viscosity})$ versus $\log(\text{shear stress})$, Figure 12, indicate the sensitivity of viscosity to changes in shear stress.

SISKO MODEL

The Sisko model (Equation 8) is another three parameter model, which is useful in describing flow in the power law and upper Newtonian regions.

$$\eta = \eta_\infty + K \dot{\gamma}^{n-1} \dots\dots\dots(8)$$

The Sisko model (Figure 13) is best suited to describe the flow behavior of fluids in the high shear rate regions ($1000-10,000 \text{ sec}^{-1}$). This model may be useful in evaluating fluid behavior at high flow rates to determine the impact of viscosity on pressure drops.

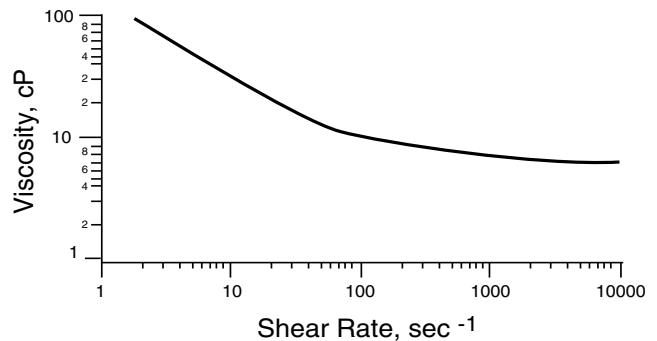


Figure 13. Idealized Sisko Model Fluid.

CARREAU MODEL

This model (Equation 9) is an example of a four parameter model that should describe the flow behavior over the entire range of shear rates.

$$\frac{\eta - \eta_\infty}{\eta_o - \eta_\infty} = [1 + (\lambda \dot{\gamma})^2]^{(n-1)/2} \dots\dots\dots(9)$$

The parameter, λ , is a time constant, calculated from the point on the viscosity versus shear rate curve where

the flow changes from the lower Newtonian region to the Power Law region. The other parameters have been previously defined. This model has received a lot of attention in rheological literature; however, it has not been extensively used in describing oil field fluids due to difficulties in obtaining data to define upper and lower Newtonian regions. Figure 14 is an idealized fluid with a lower and upper Newtonian region. This type of fluid can be modeled using the Carreau, Cross or Meter models.

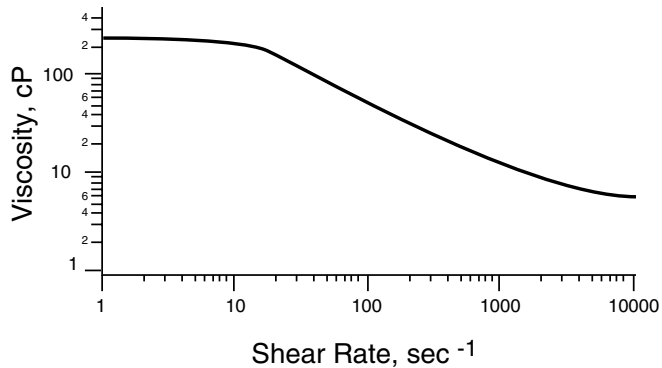


Figure 14. Idealized Non-Newtonian Fluid.

CROSS MODEL

The Cross model (Equation 10) is similar to the Carreau model in that it is a four parameter model that covers the whole shear rate range (Figure 14).

$$\frac{\eta - \eta_{\infty}}{\eta_0 - \eta_{\infty}} = \frac{1}{[1 + (\lambda \dot{\gamma})^m]} \dots\dots(10)$$

In this equation, λ is a time constant and m is a dimensionless exponent.

METER MODEL

The Meter equation (11) is a four parameter equation that expresses viscosity as a function of shear stress. Because it contains terms for the upper and lower Newtonian viscosities and terms that describe the power law region, this model can be used over a wide range of shear rates.

$$\frac{\eta - \eta_{\infty}}{\eta_0 - \eta_{\infty}} = \frac{1}{1 + \left(\frac{\tau}{\tau_{1/2}} \right)^{(\alpha-1)}} \dots\dots(11)$$

In general, the four parameter models are difficult to apply, because there is seldom enough data to allow good model fitting.

TIME DEPENDENT FLUIDS

Due to physical and/or chemical associations between particles in a fluid, time dependent behavior can be exhibited in response to an applied stress or strain. Viscosity of the fluid will increase or decrease with time while shearing at a constant rate. There are two possible time-dependent effects as shown in Figure 15. Time dependence can be manifested in viscosity measurements as hysteresis in flow curves (Figure 16).

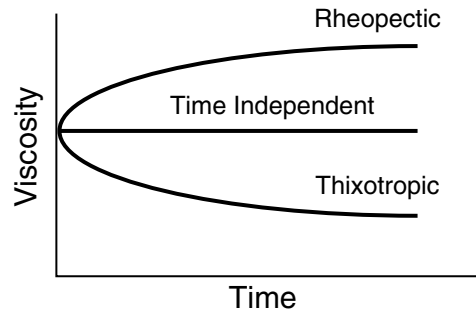


Figure 15. Time Dependent Effects.

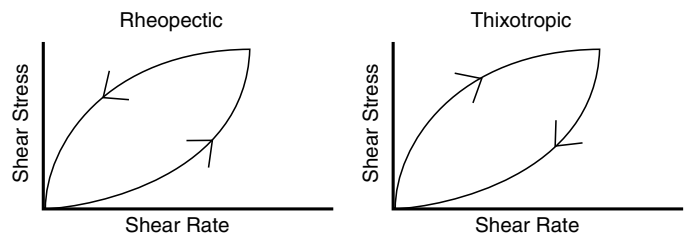


Figure 16. Manifestation of Time Dependency in Rheological Measurements.

MEASUREMENTS

The viscosity of a thixotropic fluid decreases with time. Many polysaccharides such as guar gum, carboxymethyl cellulose and starch are thixotropic to a certain degree. At concentrations used in most field applications, biogum solutions do not show significant thixotropic behavior. In some cases, fluids can display shear thickening or rheopectic behavior. This occurs in solids laden fluids containing high concentrations of bentonite (>20 lbs/bbl) or drilled solids. This behavior is also known as anti-thixotropic or negative-thixotropic behavior. Thixotropic or rheopectic behavior is often a manifestation of a structure build-up or breakdown, as a result of shear.

MEASUREMENT METHODS

Measurement of fluid rheology presents a number of interesting challenges. Flow must be steady and uniform (laminar flow) in a well defined geometry. Numerous viscometers are available for measuring the rheological properties of a fluid. The most basic viscometers provide shear stress versus shear rate data.

In the oil industry, rotational and tube type viscometers are the most popular. Tube or capillary viscometers are easy to set up and run, but the fluid has a relatively short residence time in the measurement section and large volumes of fluid are required. Rotational viscometers, on the other hand, use small volumes of fluid and offer the option of long residence times. Measurements at elevated temperatures (above 200°F) are difficult because the viscometer must be pressurized. Historically, the Marsh Funnel has been used to provide a quick and easy measurement in the field. However, it is not a viscometer, rather it only gives relative measurements, which cannot be converted to shear rate/shear stress data.

TUBE OR CAPILLARY VISCOMETERS

Design of the tube or capillary viscometer is based on the measurement of flow rate and pressure drop through a small diameter tube. Although the principles of the capillary viscometer were employed as early as 3000 B.C., it was not until 1839 that Hagen reported his experimental data. This later resulted in the Hagen-Poiseuille equation (12) for laminar flow that is the basic equation for a capillary viscometer:

$$\eta = \frac{\pi \Delta p D_c^4}{128 Q L_c} \dots\dots\dots (12)$$

where Δp is the pressure drop over a length L_c of the capillary of diameter D_c and Q is the flow rate through the capillary (Figure 17).

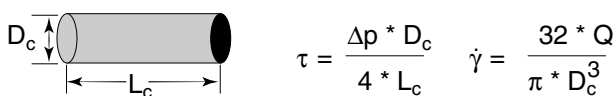


Figure 17. Diagram of Capillary Viscometer With Calculations.

The capillary viscometer usually consists of a reservoir, a capillary, and methods to control and measure flow rate, pressure drop, and temperature. Although glass capillary viscometers are sometimes used in labs, metal tubes are typically selected for field conditions. The Combs capillary viscometer (Figure 18) is a commercially available oil field version.

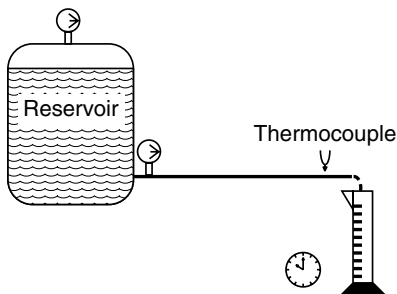


Figure 18. Capillary Viscometer Schematic.

A pipe viscometer is a variation of the capillary viscometer in which larger diameter tubes or pipes are utilized. Although larger diameter pipes are used, the same basic equation is applicable. Rheological data is usually taken in laminar flow; however, data can be obtained under turbulent flow conditions to obtain friction pressure information for design purposes.

ROTATIONAL VISCOMETERS

In rotational viscometry, torque is applied to a fluid by the rotating element of the viscometer. This torque, which is proportional to the shear stress of the fluid, is transmitted to a dial. Torque measurements can be converted to shear stress units and the rotation rate converted to shear rate. The two can then be used to calculate viscosity.

Concentric Cylinder Viscometers. In 1890 Couette described what was the first practical rotational viscometer. It was a concentric cylinder design that had a rotating outer cup and an inner cylinder on which the torque was measured. The Fann viscometers are based on this design (Figure 19). This type of viscometer is probably the most popular in the oil industry.

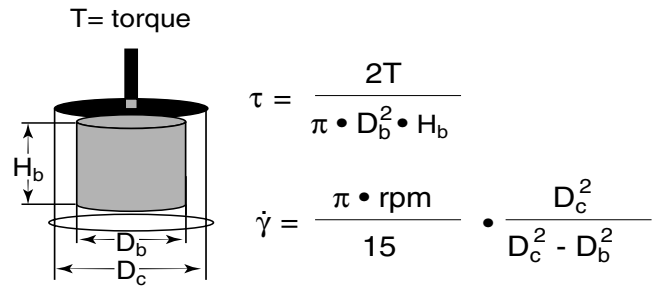


Figure 19. Diagram of a Couette Viscometer With Calculations.

The Fann or similar viscometers as used in the oil industry offer either six or twelve rotational rates. Values of the rotational rate chosen for these instruments tend to emphasize shear rates above 100 sec⁻¹. Because of the mechanical configuration of the viscometer, the low shear rate measurements have limited accuracy. Recent studies have shown that low shear rate data is useful for understanding particle transport (Bloodworth, Keely et al., 1992). Applications of this technology include the use of a Brookfield viscometer, which is capable of measurements at shear rates as low as 0.06 sec⁻¹.

Cone and Plate and Parallel Plate Viscometers. Laboratory measurements on water-soluble polymers are often made with cone and plate or parallel plate viscometers. There are advantages to each of these geometries, but both are difficult to use at elevated

temperatures with water-base fluids. They are used to provide high quality data at or near ambient temperatures.

Cone and plate viscometers (Figure 20) are equipped with small angle cones (<4°). This geometry is not suitable for fluids containing particles, but is capable of providing accurate rheological data on water-based fluids.

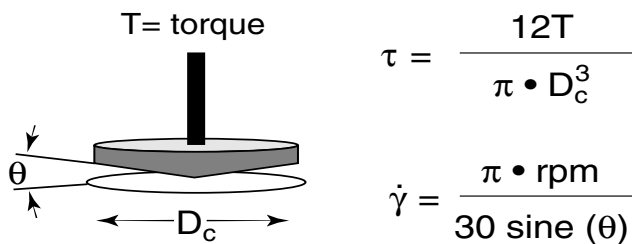


Figure 20. Diagram of a Cone and Plate With Calculations.

Parallel Plate Viscometers. The parallel plate geometry is useful for measuring the rheology of slurries of small particles at or near ambient conditions. This geometry has a complex shear rate pattern since the velocity varies from the center to the edge of the plate; therefore, data analysis is not as accurate as in the cone and plate geometry.

Index Viscometers. Simple devices have been developed that do not provide well defined shear stresses and/or shear rates. These instruments are termed index methods since their one point data is only an index (number) relative to other such numbers. Obviously they do not adequately quantify non-Newtonian fluids.

Marsh Funnel. For over fifty years the oil industry has used an orifice device called a Marsh funnel. This simple tool provides the time (in seconds) for one quart of test fluid to flow through the funnel. It is designed to yield a funnel viscosity for water of 26 (±0.5) seconds at 70°F. This instrument provides only one data point, usually at elevated shear rates for water-soluble polymer solutions. In addition, the flow rate, and thus the shear rate, decreases as the liquid level drops in the funnel. While their use has been associated with clay based fluids, the Marsh funnel provides misleading data especially for biopolymer based fluids. Biopolymer fluids exhibit low funnel values, yet develop high viscosity at low shear rates for optimum hole cleaning and suspension. In contrast, cellulosic materials provide high funnel values but limited viscosities at low shear rates and negligible suspension capabilities. **Marsh funnels cannot measure viscosity and should not be used to predict the performance of polymer based fluids.**

Falling Ball Viscometers. Another type of instrument which applies index methods is the falling ball (sphere) viscometer. This device is based on Stokes' law (Equation 13). It correlates the relationship between the viscosity (η), the diameter of the ball (d), the density difference between the ball (ρ_s) and the liquid (ρ), and the settling velocity of the ball (v):

$$\eta = \frac{(\rho_s - \rho)gd^2}{18v} \dots\dots\dots (13)$$

where g is the gravitational constant.

The device is generally used for relative measurements or for Newtonian fluids; however, viscosity cannot be accurately calculated. Since it is easy to pressurize, the falling ball viscometer has found some use in measuring the properties of drilling fluids and cements at high temperatures. This concept has been extended to a falling cylinder or driven cylinder device where one cylinder moves coaxially within another, which is often referred to as a telescopic-shear viscometer. The test fluid is placed in the annular gap. The Fann Consistometer was built on this design to allow high temperature/high pressure measurements. However, the time of travel for its inner cylinder is only a relative measure of the viscosity.

VISCOELASTICITY

As indicated earlier, materials that follow Newton's Law have a shear stress, τ , that is proportional to the shear rate, $\dot{\gamma}$. These materials are liquids and the proportionality constant is called the viscosity, η . Materials that follow Hooke's Law are solids. They have a shear stress, τ , that is proportional to the shear strain, γ . The constant that relates stress and strain in solids is the elastic or rigidity modulus, G .

$$\tau = G\gamma$$

The distinction between solids and liquids is not as clear as the earlier section might indicate. Most materials have some fluid-like (viscous) properties as well as some solid-like (elastic) characteristics. They are viscoelastic in nature. For example, biopolymer solutions such as xanthan gum are viscoelastic. They pour with ease (and so appear to be viscous liquids) yet are able to suspend solid particles (and so have the characteristics of an elastic solid). In fact, most fluids used in oil field applications are viscoelastic to a certain degree.

One manifestation of viscoelasticity is the presence of normal forces in a viscoelastic fluid undergoing shear. When a fluid is sheared between two plates, a stress exerts a normal force on the plates which is perpendicular to the shearing direction. This force tends to push the plates apart. Normal forces result in the "Weissenberg effect" which allows a viscoelastic liquid to climb up (rather than vortex down) a rotating stirring rod.

The most common way of quantifying the viscoelastic properties of fluids is by measurement of their elastic modulus (G') and viscous modulus (G''). G' is also called the storage modulus (since elastic energy is stored), while G'' is often referred to as the loss modulus (since viscous energy is lost).

It is convenient to think of G' and G'' as vector quantities located 90° apart. Vector quantities have a magnitude (which is indicated by the length of the vector) as well as a direction. Figure 21 illustrates these values for two fluids.

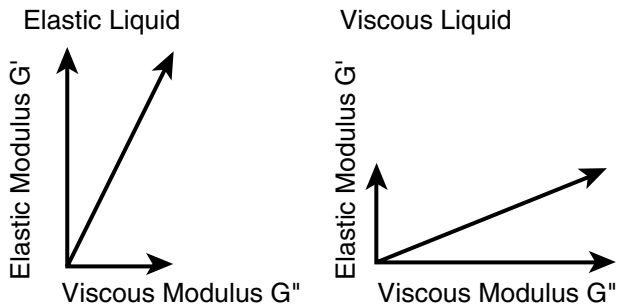
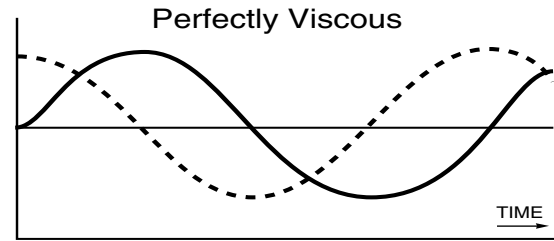


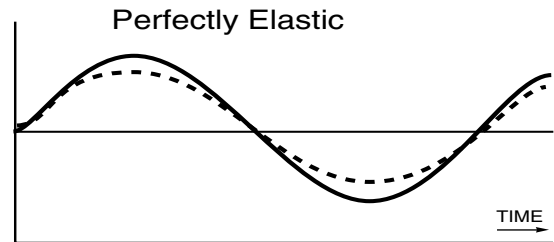
Figure 21. Modulus Values For Two Fluids

Both fluids would exhibit the same constant shear viscosity since the resultant vector of the two moduli is the same magnitude. However, viscoelastic measurement reveals that from a rheological standpoint, they are quite different. In the elastic fluid, viscosity is primarily due to its elastic modulus. On the other hand, the viscous fluid derives most of its viscosity from its viscous modulus. The elastic fluid would provide improved suspension of insoluble particles or a more stable emulsion. These differences can only be revealed through viscoelastic measurements.

Since viscoelasticity cannot be measured in the steady, uniform flow field found in viscometers, oscillatory methods of measurement must be used. The rheometer applies a sinusoidally varying strain (deformation) to the sample. An appropriate transducer measures the resulting stress (force). In samples that are highly elastic, the stress and strain sine waves are in phase. If the material is perfectly viscous (such as an oil) the stress and strain will be 90° out of phase (Figure 22). All materials have a phase angle somewhere between 0 and 90 degrees. Instruments commonly used for viscoelastic measurement include the Rheometrics, Weissenberg and some Haake and Contraves models.



Stress (solid line) and strain (dashed line) are 90° out of phase



Stress (solid line) and strain (dashed line) are in phase

Figure 22. Viscous and Elastic Responses to an Applied Strain.

MEASUREMENT METHODS

The technique described above for measuring viscoelastic properties can be used a number of ways. These are commonly called a frequency sweep, a strain sweep and a dynamic recovery test.

As the name implies, the frequency sweep varies the period or frequency of the sinusoidal oscillation as the test progresses. The amplitude or strain level is held constant. In many cases the frequency is expressed in radians/second (rad/s) to make comparison with steady shear data more straightforward. Because of the Cox-Merz rule, frequency in rad/s is equivalent to shear rate in sec^{-1} . In this way, the complex viscosity (η^*) can be compared to the shear viscosity (η) for materials that are not highly associated. Most often, a frequency sweep is run from 0.1 to 100 rad/s with a strain level in the linear viscoelastic region (to be explained in the next section). Fluids that are liquid-like have a G'' that is higher than G' at low to moderate frequency values. As a fluid becomes more gel-like and structured, G' increases faster than G'' ; therefore, G' predominates. In addition, these fluids tend to have G' curves that are relatively flat (independent of frequency).

In the strain sweep test, frequency is held constant while the amplitude of the oscillation (and thus the strain) is increased in some progression. The modulus values will remain relatively constant with respect to strain until a critical strain value is exceeded. This region, where modulus is constant, is referred to as the linear viscoelastic region. With these relatively small

strain values the associations between components in the system are not significantly disrupted. This is what leads to nearly constant modulus values. Systems that are highly loaded with suspended particles have a much smaller linear viscoelastic region than do solutions of pure polymer. In the same way, polymer solutions with robust intermolecular associations (like biopolymers) have a longer viscoelastic region than do more cross-linked fluids. If the linear viscoelastic region is not known, the strain sweep is run before the frequency sweep so that the latter can be run in the linear strain region.

The final common type of viscoelastic test is the dynamic recovery test. For this test, the sample is sheared at steady shear for some time to fully break down all molecular associations. Following this, the rotation is immediately stopped and a dynamic oscillation at some low strain level is begun. Points of association within the sample begin to reform and the elastic modulus (G') begins to increase. If the associations require extensive time to rebuild (as is the case with many particulate thickeners such as clays) the rate of increase of G' will be slow. On the other hand, biopolymer formulated systems will frequently show negligible loss of modulus from shear. Any loss measured is quickly rebuilt.

While it is undoubtedly a sophisticated technique, viscoelastic measurement can add a great deal to the understanding and differentiation of polymer systems. It provides a useful addition to the traditional, steady shear measurements.

TYPICAL CURVES

Figure 23 shows viscoelastic results for the same polymers illustrated in Figure 4 previously discussed. The xanthan exhibits a higher degree of elasticity at the lower frequencies than the HEC or guar. In addition, the xanthan G' curve is much flatter than either the HEC or guar G' curves which indicates gel-like behavior in the xanthan solution. For xanthan, the G' curve is greater than the G'' curve giving further evidence for gel-like behavior which indicates good suspension properties.

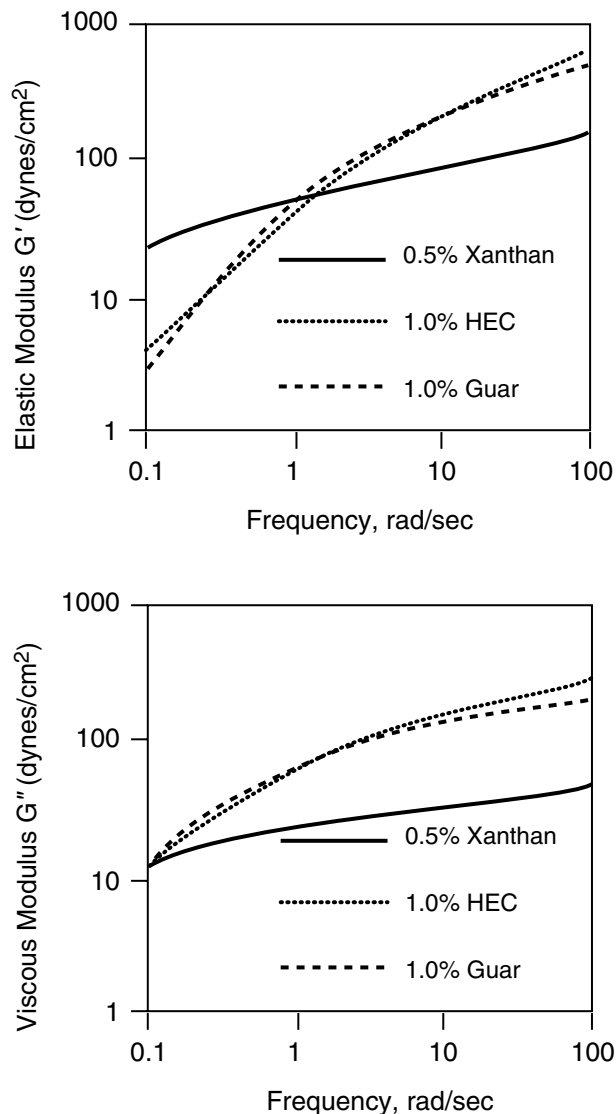


Figure 23. Viscoelastic Measurements for Xanthan, HEC and Guar in 3% KCl, 75°F

EXTENSIONAL RHEOLOGY

Extensional viscosity is a relatively new concept for many people; however, these types of flow are important in polymer processing and fiber spinning. Flow does not need to be complex to have extensional components. Pouring water from a container involves an extensional flow with gravity acting as the extensional force. Flow into expansions or contractions are other examples of extensional flow. The extensional component of this type of flow is one of the reasons why non-Newtonian fluids show higher pressure drops in flow through expansions and contractions than is observed for Newtonian fluids. The idea of stretching a liquid solution may seem unlikely and of little relevance; however, such measurements can be very revealing for many polymers. Diverse processes such as flow through porous media and drag reduction involve highly extensional flow.

MEASUREMENT METHODS

Extensional flow devices for liquids can be divided into two broad categories. In the filament stretching device, a thin filament of the liquid is stretched, usually by pulling a vacuum. The flow rate and change of cross sectional area of the filament is used to determine the extensional strain rate. A force measuring device, such as a fiber spinning instrument, is used to determine the stress applied to the sample. A second class of instruments capable of providing extensional flow fields is represented by the stagnation point devices. These include the 4 roll mill, cross slot flow and opposing jet devices. With these, an area of intense extensional flow can be created without the need for a spinnable sample. That is, extensional measurements are possible for materials not able to sustain a continuous filament. In addition, stagnation point devices are capable of higher strain rates than are fiber spinning instruments. Stagnation point devices have a long history of use for optical rheological measurements. The area near the stagnation point shows birefringence due to chain alignment above a critical strain for most polymers.

A schematic of the opposing jets device is shown in Figure 24. The sample is held in a jacketed beaker with a temperature probe and two jets immersed in the fluid. The left arm and jet are mounted on a force rebalance transducer. The right arm and jet are mounted on a moveable mechanical stage to control jet spacing and alignment. Fluid is drawn into the two jets simultaneously by dual syringe pumps. Flow rate is controlled by a computer driven stepper motor. When desired, temperature control is achieved with a circulating water bath and jackets around the beaker and syringes. This device has the advantage of producing an extensional flow field in a relatively small volume of liquid. It is readily adaptable to the water-soluble polymer solutions used in the oil field.

The equations required to calculate extensional stress (σ) and extensional strain rate ($\dot{\epsilon}$), as well as extensional viscosity ($\bar{\eta}$), are very simple for the opposing jet device where F_j is the force at the jet, A is the cross sectional area of the jet, Q is the volumetric flow rate, G is the gap between the jets and D_j is the diameter of the jets:

$$\sigma = \frac{F_j}{A} \quad \dot{\epsilon} = \frac{8Q}{\pi G (D_j)^2} \quad \bar{\eta} = \frac{\sigma}{\dot{\epsilon}}$$

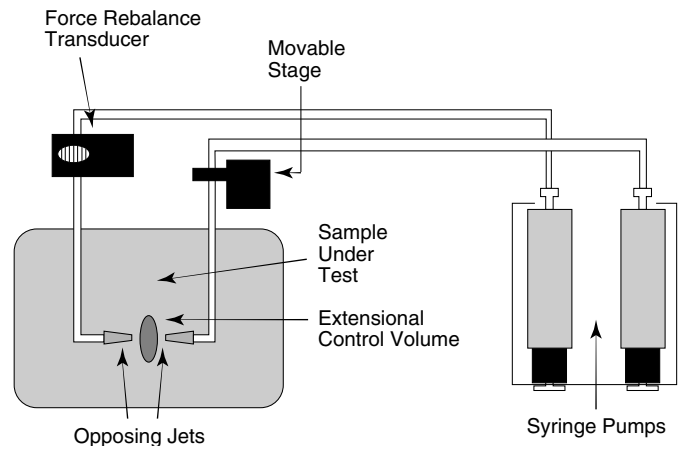


Figure 24. Schematic of the Opposing Jets Device.

Effective strain rate may be varied by two methods: altering the flow rate or by changing the jet area. Larger jets and/or lower flow rates reduce the effective strain rate, $\dot{\epsilon}$.

The Rheometrics Fluids Extensional Rheometer (RFX) measures extensional viscosity by means of an opposing jet design. Strain rates from 10^{-1} to 10^4 may be achieved for materials of sufficient viscosity, as illustrated in Figure 25.

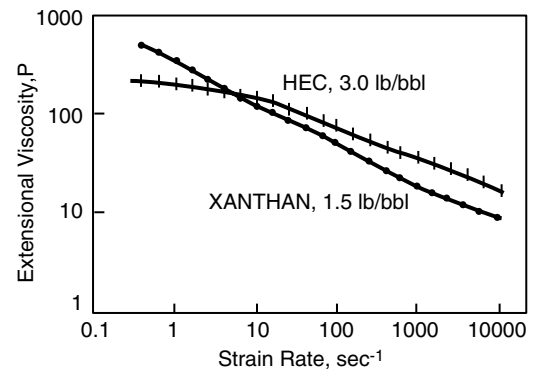


Figure 25. Extensional Viscosity of Xanthan and HEC, 3% KCl, 75°F

The Trouton ratio is the extensional viscosity over the shear viscosity, $\bar{\eta} / \eta$ and is used as a reference for extensional viscosity measurement. It is an excellent way to check the validity of a technique, since the extensional viscosity should be three times the shear viscosity for simple Newtonian materials.

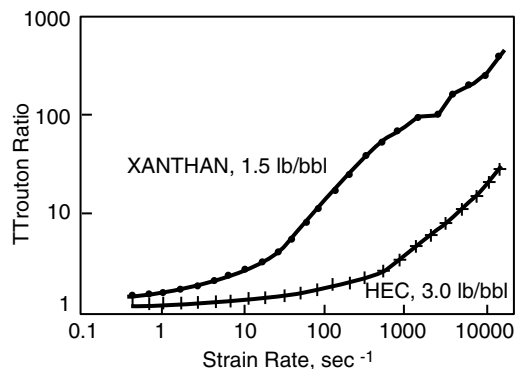


Figure 26. Trouton Ratio of Xanthan and HEC, 3% KCl, 75°F

Trouton ratios are frequently a good indicator of molecular conformation. When the extensional viscosity increases relative to the shear viscosity, the Trouton ratio increases. This increase is indicative of an increase in hydrodynamic size due to molecular stretching. If the Trouton ratio is constant and low (theoretical minimum is 3) then the time scale (inverse of the strain rate) is too long to influence the molecule's shape. If the Trouton ratio is constant and high, it is an indicator that the molecule is fully stretched. The absolute magnitude of the Trouton ratio in the upper plateau (if measurable) is an indicator of how much the molecule can be stretched out. A large difference between the ratio at low and high strain rates indicates that the molecule is very flexible and can be extended to a high degree. A large molecule will show a greater Trouton ratio than a small one. Figure 26 provides a comparison of Trouton ratios for xanthan (1.5 lb/bbl) and HEC (3.0 lb/bbl). Since the xanthan molecule is stiffer and easier to stretch, Trouton ratios for the xanthan increase at lower strain rates relative to the HEC. The increased Trouton ratios observed for xanthan are also indicative of the high degree of inter-molecular associations, typical for this concentration of xanthan.

SUMMARY

The ability of a fluid to perform a specific function is dependent on its rheological properties. Establishing correlations between rheological parameters and fluid functionality is the first step toward fluid design and optimization for any application.

Mathematical equations describing the rheological properties of fluids have been used to predict flow behavior under a variety of conditions. When modeling fluid flow, extreme care should be taken to validate the model for each fluid and to use the model only for conditions under which it has been shown to be valid. There is no universal model for fluids. All the models have limitations; for example, the power law model cannot be used to accurately predict low shear rate behavior of fluids having a lower Newtonian region.

Measurement of rheological properties is a difficult process which is complicated by the presence of solids, viscoelasticity, and the need to measure rheological properties over a wide range of shear rates. Current instrumentation, usually a concentric cylinder viscometer, does not adequately describe the rheological behavior of fluids in the low shear rate range that characterizes hole cleaning and suspension properties of the fluid. For example, traditional measurements derived from the Fann model 35 viscometer including yield point and viscosity at 3 rpm (5.1 sec⁻¹) do not fully describe or predict performance of polymer based fluids. This has mandated the adoption of more sophisticated instruments. Even the use of the Brookfield LV series viscometers is an improvement because they allow data to be acquired at much lower shear rates. These instruments provide a means of evaluating fluid behavior at low shear rates and verifying the accuracy of extrapolations made from high shear rate measurements. Until these and other instruments, which provide data over a wider shear rate range, are adopted, there will continue to be limited understanding of fluid functionality.

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Symbols

- A - surface area
d - diameter or thickness
 d_c - diameter of a tube or capillary
f - friction factor
F - force
G - modulus of elasticity
G' - elastic storage modulus
G'' - viscous loss modulus
K, K' - consistency index
 L_c - length of a tube or capillary
 n, n' - flow behavior index
 N_{Re} - Reynolds number
 Δp - pressure drop
Q - flow rate
s - displacement
v - velocity

Greek Symbols

- α - exponent usually equal to 1/n
 $\dot{\gamma}$ - shear rate
 γ - shear strain
 ϵ - extensional strain
 η - shear viscosity
 η_a - apparent viscosity
 η - limiting high shear rate viscosity, upper Newtonian region
 $\bar{\eta}$ - extensional viscosity
 η^* - complex viscosity
 μ - Newtonian viscosity
 ρ - fluid density
 ρ_s - solid density
 σ - extensional stress
 τ - shear stress
 τ_0 - yield stress
 $\tau_{1/2}$ - stress at which the initial viscosity drops by half
 ϕ - porosity

APPENDIX

Definition of Terms

Rheology - The science of the deformation and flow of matter.

Rheological properties - Rheological properties of fluids are usually derived from measurements using instruments with carefully defined flow geometries.

Normal Stress - A stress that acts perpendicular to a face.

Shearing Stress - A stress that acts tangentially to a face.

Shear Stress (τ) - The shear stress is defined as the force per unit area.

$$\text{Shear Stress } (\tau) = \frac{\text{shear force}}{\text{area fraction}} = \frac{A}{F}$$

Shear Strain (γ) - The shear strain is defined as the displacement divided by the height of the fluid element.

$$\text{Shear Strain} = \frac{\text{shear displacement}}{\text{height of fluid element}} = \frac{s}{h}$$

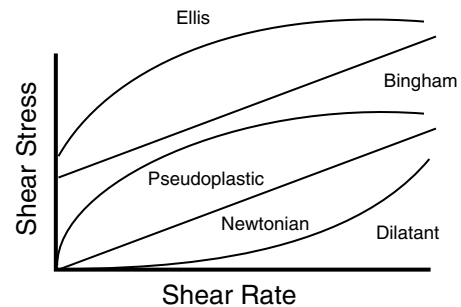
Shear Rate ($\dot{\gamma}$) - The shear rate is also called the shear strain rate. It is the time rate of change of the shear strain.

$$\text{Shear Rate } (\dot{\gamma}) = \frac{d(s/d)}{dt} = \frac{d\gamma}{dt}$$

Viscosity (η or μ) - The resistance to flow. For Newtonian fluids when shear stress is plotted against shear rate, a straight line results. The mathematical relationship that describes this line is $\tau = \eta \dot{\gamma}$. The slope of the line η is the constant of proportionality between shear stress and shear rate, and is known as the viscosity.

Apparent Viscosity (η_a) - Fluids that exhibit a non-linear relationship between shear stress and shear rate will exhibit a spectrum of viscosities depending upon the shear rate. To describe the behavior of these fluids, an apparent Newtonian viscosity has been defined as the slope of the line that connects the origin with a point on the curve at the shear rate of interest. Normally, this value is reported along with the corresponding shear rate.

Non-Newtonian Fluid - fluids that exhibit a non-linear relationship between stress and strain are termed non-Newtonian fluids. There are a number of different non-Newtonian fluids. The basic types of non-Newtonian response curves are shown below:



Laminar Flow - is a smooth flow that is devoid of flow disturbances such as eddies.

Turbulent Flow - is a chaotic flow characterized by intense mixing caused by eddies.

Reynolds Number (N_{Re}) - a dimensionless number that is the ratio of the inertial to the viscous forces,

$$N_{Re} = \frac{v\rho d}{\mu}$$

where v is velocity, ρ is the fluid density, d is a diameter and μ is the viscosity.

Velocity Profile - when a fluid flows in a confined region, the velocity of the fluid will vary with position. The shape of the velocity profile is a function of non-Newtonian nature of the fluid, and flattens as the power law n value decreases.

