

Gelation and Time-Dependent Rheological Behavior of Oil / Synthetic Based Drilling Fluids

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Abstract

Excessive pressure peaks inside the wellbore due to fluid gelation are observed typically at low wellbore temperatures in deep water wells. In such wells there is a narrow window between the pore pressure and the fracture gradient, causing the fracture pressure to be reached very easily. This study examines the transient stress response and gel-breaking pressure of time-dependent drilling fluids by taking into account how temperature and aging time influence structure development of the fluid. Aging time and temperature effects on fluid gelation in terms of stress and pressure responses of synthetic based fluids are investigated. An experimental study has been carried out to evaluate the rheological behavior and gelation properties of a synthetic-based fluid under a temperature range from 4 °C to 50 °C. A high-accuracy rheometer has been used to conduct steady state (equilibrium) and stress over-shoot (non-equilibrium) tests with vane geometries with shear rates in the range of 1.7 s⁻¹ to 85.5 s⁻¹. Results show that fluid gelation is severe at lower temperatures and longer static periods. Further, flow tests of a synthetic drilling fluid have been conducted in a flow loop with an annular test section in order to observe and simulate pressure peaks due to gel breaking pressure and surges occurring inside a wellbore upon displacing a gelled fluid with a non-gelled one. Finally, a mathematical model is developed based on the structure kinetic theory to describe the time-dependent rheological behavior of the fluid as a function of temperature, shear rate and aging time conditions. Model results and experimental observations of pressure peaks and stress conditions at the equilibrium state are in good agreement.

Introduction

Rheological properties of drilling fluids are adjusted to fulfill operational requirements and to affect the functionality of the mud. These properties have a direct impact on drilling hydraulics, cuttings transportation, and well bore stability. In addition, drilling fluids should prevent settling of cuttings and chemical additives by developing a solid or gelled structure during pump-off periods. Additional energy is required to decompose these structures to re-circulate the fluid in the drillstring and annulus. The magnitude of this energy increases

as the non-circulating duration increases. This extra energy directly influences the circulation pressure. One of the consequential concerns in deep water wells is to design and select the proper fluid system in order to maintain the ECD between pore pressure and the fracture gradient window of the formation to maintain wellbore stability. Insufficient ECD ranges may fracture the formation and cause either lost circulation or blowouts.¹

In this study, the transitional rheological behavior and pressure response of time-dependent fluids have been investigated by considering the yield stress and thixotropic phenomena. Besides the aging effect, effect of fluid temperature on gelation is investigated and a mathematical model has been proposed and compared with experimental results.

Background

Micro-structural existence in a fluid resists large rearrangements and is responsible for the yield stress. The destruction of such a microstructure while shearing is the source of thixotropy². Mujumdar et al.³ stated that thixotropy is a reversible breakdown of particulate structures under shear, which is frequently associated with a yield stress.

A continuous solid-like network can be formed in time-dependent fluids when the fluid is not flowing, and a minimum shear stress is required to initiate fluid movement. This minimum stress is known as the yield stress and varies with time and temperature⁴. Below the threshold of this stress value, elastic or plastic deformation is observed; on the other hand viscous deformation is encountered when the applied stress exceeds this critical value.² Yield stress is difficult to measure.⁵ In order to understand the yield stress concept and to obtain a true value of yield stress, dynamic and static yield stress terms should be taken into account and evaluated separately. Dynamic yield stress, known as a yield point, shows the stress value below which fluid deforms in an elastic range and structures in the system regain their original shapes when the applied stress is removed.⁶ Dynamic yield stress shows the stress which is needed to stop the fluid flow after the flowing or shearing process is over. Static yield stress, also known as gel strength, is the minimum stress required to start the fluid flow from a static condition⁷. Time-dependent

fluids can store some energy and form a weak gel structure. According to Herzhaft et al.⁸ colloidal interaction between solid additives in drilling fluids can lead to development of a structural network when the fluid is at rest. The strength of this gel network is sensitive to shearing history and temperature.

The rheology of most drilling fluids shows thixotropic character. Thixotropic fluids form a 3-D network as in gels and the 3-D structure is broken down to get a minimum thickness as in sols; hence thixotropy is referred to as the “sol-gel” transition phenomena.⁷ According to Barnes et al.⁹ the finite time needed for a reversible change of microstructures from one stage to another stage in liquid systems is known as thixotropy. Thixotropic systems consist of fine particles at the colloidal level and the interaction between these particles as they build-up microstructures. The build-up process is the association of particles to form a structural network due to shear-induced collision and a random movement of particles that are suspended in the liquid.⁵ In this process viscosity of the thixotropic fluid can increase as the build-up phenomenon continues. Contrarily, shearing force breaks the developed network due to viscous drag and is known as the structure break-down process. Apparent viscosity decreases during this process. When thixotropic materials are sheared, build-up and break-down kinematics compete with each other and eventually reach an equilibrium rate. Time-dependency or thixotropy is observed when the rates of the two kinematic processes are different¹⁰.

Time-Dependent Rheological Behavior of Thixotropic Systems with Yield Stress

Transitional pressure responses at the initiation of flow, when compared to steady flow cases, are observed in thixotropic fluids. Time-dependent rheological models are necessary to calculate the pressure peaks for a given duration of non-circulation. A mathematical form of the yielding condition in the thixotropic model is needed for constitutive equations, because both indicate the influence of an internal structure on the rheological behavior. Numerous studies found in the literature belong to time-dependent or transient stress equations based on structure kinetic theory with different approaches¹¹ or are based on curve-fitting methodology. Gandelman et al.¹² developed a gel stress equation with different aging time and temperature conditions to calculate the pressure peaks generated after a pump-off time. Bjorkwall et al.¹³ developed a gel strength equation based on the difference between initial stress peak and steady shear stress under different resting conditions. Mujumdar et al.³ used the indirect micro structural approach and proposed a time-dependent stress equation. They claimed that the total stress is the combination of elastic and viscous stresses in the system. Hernandez et al.⁴ researched the equilibrium and non-equilibrium rheological behavior of fresh cement paste and characterizes the time-dependent rheological response as agreeing with the Bingham Plastic model under equilibrium conditions. They stated that initial structure affects the yield stress term, and instantaneous structure level in the system

affects the viscosity term. Their stress equation is:

$$\tau = \lambda_o \tau_{yo} + \lambda \mu_{po} \dot{\gamma} \quad (1)$$

where λ_o is the initial structure parameter, τ_{yo} is the yield stress at which all of the structure is assumed to be built-up in the system ($\lambda=1$), and μ_{po} is the plastic viscosity at which all the structure is assumed to be built-up in the system ($\lambda=1$). Tehrani et al.¹⁰ analyzed the thixotropic behavior of water-based drilling fluids and modeled their gelling properties. In their study, Moore's¹⁴ structure rate equation is used to explain the evolution in terms of a single structure parameter. This equation shows the time, or thixotropic effect on rheological parameters (both on the yield stress and apparent viscosity) at a molecular and particle level:

$$\frac{d\lambda(t)}{dt} = a[1 - \lambda(t)] - b\lambda(t)\dot{\gamma} \quad (2)$$

Cheng and Evans¹¹ use an equation of state that is similar to Hernandez et al.'s model⁴ to develop a time-dependent constitutive equation.

$$\tau(t) = \lambda(t)\tau_y + [\mu_\infty + c\lambda(t)]\dot{\gamma}^m \quad (3)$$

where $\tau(t)$ is the instantaneous stress, τ_y is the dynamic yield stress and μ_∞ is the infinite viscosity value (viscosity value at equilibrium conditions).

Theory

Classical time-independent rheological models such as the Bingham Plastic (BP) or the Yield Power Law (YPL), explain steady state stress behavior and are adequate for classical calculations in to characterize the rheological behavior of yield stress fluids. However, these models do not consider the transient deformation response because of thixotropy or aging. In more challenging and complex situations like pressure peaks after pump start-up, these steady-state models cannot be used since they do not take thixotropic behavior into account.⁸

For this study, the followings assumptions are made in developing a mathematical model for both stress and frictional pressure loss equations: i) the sample fluid is incompressible, ii) the system is homogenous, iii) steady-state rheological parameters, i.e., the dynamic yield stress, τ_y , the consistency index, K , and the fluid flow behavior index, m , are all time-independent and only change with temperature.

In order to take thixotropy into consideration, structure kinetic theory and the structure parameter, λ , which shows the ratio of unbroken links in the total network of the system, are used in the proposed model. The structure parameter was introduced and used by Moore¹⁴ to investigate ceramic paste flow. Moore's form of the structural rate equation is

$$\frac{d\lambda(t)}{dt} = f(\text{Buildup rate \& Breakdown rate})$$

or

$$\frac{d\lambda(t)}{dt} = U(1 - \lambda(t)) - D\lambda(t) \quad (4)$$

where U is the build-up rate coefficient and D is the break-down rate coefficient. Equation 5 states that, the rate of structure development increases with the increase in number of unstructured particles in the system, represented by $1 - \lambda(t)$ in the equation. Similarly, the rate of structure break-down is proportional to the instantaneous structure, $\lambda(t)$, which exists in the system at that time, and the applied shear rate. In dynamic equilibrium the rates of structure build-up and structure break-down are equal, i.e., $d\lambda(t)/dt=0$. The value of the structure parameter at equilibrium depends on the applied shear rate: the higher the shear rates, the higher the break-down rates, which decreases the value of λ_{eq} . Conversely, the lower the rate of strain, the higher the build-up rates, which brings on higher equilibrium values.⁵ Therefore, the equilibrium value of the structure parameter can be expressed as:

$$\lambda_{eq} = \frac{U}{U + D} \quad (6)$$

Equation 8 is solved with the help of two limiting conditions: i) at $t=\infty \Rightarrow \lambda(t) = \lambda_{eq}$ (which is the equilibrium structure parameter), and ii) at $t=0 \Rightarrow \lambda(t) = \lambda_o$ (which is the initial structure parameter). Finally, the rate equation becomes

$$\lambda(t) = \lambda_{eq} + (\lambda_o - \lambda_{eq})e^{-(U+D)t} \quad (7)$$

Structure effects on the rheological behavior of the fluid are considered only in the yield stress term. The consistency index, K , and the fluid behavior index, m , are assumed to be constant at a constant temperature. Based on these assumptions, the time-dependent constitutive equation can be expressed as

$$\tau(t) = \lambda(t)\tau_{yo} + K(\dot{\gamma})^m \quad (8)$$

where τ_{yo} is the maximum value of the static yield stress at which all the structure is completely built-up ($\lambda=1$). The first term on the right side of the equality, $\lambda(t)\tau_{yo}$, is the instantaneous static yield stress. Combining Equations 7 and 8 yields

$$\tau(t) = [\lambda_{eq} + (\lambda_o - \lambda_{eq})e^{-(U+D)t}] \tau_{yo} + K(\dot{\gamma})^m \quad (9)$$

The initial structure parameter, λ_o is found by using the structure kinetic equation (Equation 5). When the thixotropic

system is motionless, only the build-up process occurs in the system and the build-up dynamic determines the structure development. Therefore, the rate equation for the initial structure parameter in terms of resting time is redefined as

$$\frac{d\lambda_o(t_r)}{dt_r} = U(1 - \lambda(t_r)) \quad (10)$$

We then apply the limiting conditions: i) at $t_r=\infty \Rightarrow \lambda(\infty) = 1$ (which is the complete build-up structure) and ii) at $t_r=0 \Rightarrow \lambda(0) = \lambda_{eq}$ (which is the equilibrium value of the structure). Finally the rate equation for λ_o , which is a function of aging time, becomes:

$$\lambda_o(t_r) = 1 + (\lambda_{eq} - 1)e^{-(U t_r)} \quad (11)$$

Thus, the time-dependent constitutive equation (Equation 8) can be defined as a function of U , D , t and t_r by

$$\tau(t) = \left\{ \left[\frac{1 + (\xi - 1)e^{-U t_r}}{-\xi} \right] e^{-(U+D)t} + \xi \right\} \tau_{yo} + K(\dot{\gamma})^m \quad (12)$$

where $\xi = \frac{U}{U + D}$

This equation does not explain elastic and plastic region deformation, but models the transition region between elastic-plastic deformations to viscous flow. The major focus of this study is the pressure peaks due to gelation and the pressure responses with time.

Experimental Work and Determination of Model Parameters

Laboratory tests were conducted to characterize fluid rheological behavior with different aging times and temperatures using a rheometer. Also, flow-loop experiments were performed to observe the time effect on transitional pressure responses. A Synthetic-Based drilling fluid (SBM) was used in the experiments. The fluid has the physical properties of 11 ppg density and 75/25 oil/water ratio, with 17% water phase salinity.

In the first stage of experimental work, an Anton Paar Physica MCR 301 rheometer was used, with different measurement geometries, to determine the static yield stress and to characterize the fluid. After analyzing the test results, the coefficients of Equation 12 were determined. For transient rheological tests a vane measurement geometry was found to be the best.

The proposed model does not cover the elastic deformation part of the stress response, which is illustrated in Figure 1 (0.32 s⁻¹ shear rate, 4°C temperature, 60-minute aging time). In this figure, it is clearly stated that first elastic, then plastic

stress responses are observed against the applied rate of strain within the first second. The peak stress is observed after 0.611 seconds of shear application; hence, this time is assumed to be the starting time of shearing and time data are refined according to this consideration.

In the rheological tests strain-controlled measurement methodologies are selected, low and constant shear rate is applied to the sample and shear stress overshoot or peak stress is recorded as the static yield stress value; i.e., the stress value at which the creeping flow pattern changes to viscous flow.¹⁵ This technique is known as the “stress-overshoot test” (SOT). Figure 2 illustrates the SOT results at a constant 0.27 s⁻¹ shear rate and a 17°C constant temperature after 1, 5, 10, 20, 30, 40 and 60 minutes of aging times. The aim of this graph is to highlight the aging effect on shear stress readings. Therefore, this figure is limited to 2 seconds in order to demonstrate the early time-dependent stress response. All of the aging time graphs start from the peak points and then stress decay follows because the developed structural network is broken by shearing force. The threshold or peak values increase as the aging is increased. Finally, all the stress curves come to an equilibrium value because steady state shear stress is independent of aging period and is only affected by ambient temperature and applied shear rate, as illustrated in Figure 3. However the relaxation period, i.e., the time interval between t peak point and the equilibrium point, increases when the aging time is increased due to the higher network development possible during longer resting periods.

According to SOT results presented in Figure 4, structure development continues after 30 minutes of aging with a τ_{ys} value of 19.42 Pascal, which increases to 21.47 Pascal after 60 minutes. Therefore, the structure development or structural network increases when the duration of the aging period is extended, as shown in the gel strength or peak stress readings of the SBM.

Presented in Figure 5 is the stress measurement of SBM at 4, 10, 17, 24, 30, 40 and 50°C at the 0.84 s⁻¹ average shear rate application after 30 minutes of aging. Both the equilibrium stresses and the peak stresses increase when the fluid temperature is decreased, as seen in Figure 6. It can be observed that the gelation process or structural build-up is more significant at low temperatures. Also, the stress decline rates increase as the temperatures increase, as seen in Figure 5.

Determination of the Model Coefficients

Equation 12 contains coefficients which need to be determined via dynamic tests. These coefficients are all material-dependent and affected by temperature change in the fluid system.

Rheological parameters (the consistency index, K , dynamic yield stress, τ_y , and the flow behavior index, m) are obtained from the steady state experimental data at various shear rates and a constant temperature. The equilibrium shear stress data are plotted against the corresponding shear rate to construct a rheogram. This curve is fitted to a Yield Power Law model to obtain the three model parameters. As mentioned, these equilibrium rheological parameters are temperature sensitive;

the procedure must be repeated for different temperatures. There are three parameters to describe a YPL fluid: the dynamic yield stress or yield point, τ_y , the consistency index, K , and the flow behavior index, m . The model is

$$\tau = \tau_y + K \dot{\gamma}^m \quad (13)$$

In the literature numerous methods are used to get the rheogram. The rheogram is the plot of various shear stresses at the corresponding shear rates. Low shear rate readings in time-dependent or structured fluids show higher stresses compared to actual or steady state conditions, as illustrated in Figure 7. This is due to inadequate time for structures to reach equilibrium. This problem is eliminated by selecting the shear stresses at the corresponding shear rates on SOT at constant temperature test intervals. Time duration is adjusted for stress response to reach the steady state; i.e., equilibrium is reached at the structural level. For example, the equilibrium stress readings for a 4°C test at an applied constant 6.31 s⁻¹ shear rate are: 14.86, 14.68 and 14.443 Pascal for 10-, 20- and 30-minute resting times, respectively. The steady state stress value of this shear rate is found by taking the average of the seven aging period stabilized readings and is found to be 14.69 Pascal (see Figure 8). The procedure is repeated for every shear rate interval to sketch the rheogram. Table-1 illustrates the three parameters that were determined.

The definition of the maximum value of static yield stress (gel strength), τ_{yo} , is the static yield stress or peak stress measurement of a thixotropic fluid in which all the structures in the system are completely built-up ($\lambda=1$). The parameter τ_{yo} is affected by temperature and applied shear rate. Therefore, it is obtained by plotting the stress peaks of stress over-shoot data versus resting time at a constant shear rate and temperature. The value τ_{yo} is obtained by extending the plot of resting time to infinity. This procedure is repeated for different shear rates at each temperature to find the influence of shear rate. Experiments are repeated at different temperatures to observe the effect of temperature on τ_{yo} .

The numerical value of τ_{yo} is obtained by plotting the stress peak readings at constant temperature and constant shear rates at different aging times. Figure 9 is such a plot at 17°C and at 5.37 s⁻¹ shear rate. The stress peaks show a logarithmic increase with aging time and stress can be stabilized after 900 minutes. Therefore the limiting time for the structure growth or development in the system is assumed to be 900 minutes. Figure 10 presents an example of τ_{yo} values determined at 17°C and seven different shear rate results. From this graph, a constant 45.5 Pascal τ_{yo} is used to find the coefficients of U and D terms in Equation 12.

When the stress peak readings of SOT are encountered, shearing time is assumed to be zero ($t=0$) or shearing is assumed to start at the instant of stress-overshoot; i.e., inertia and elastic deformation are eliminated. The model proposed in Equation 12 is modified when $t=0$:

$$\tau_{peak}(t_r) = \left[\left(1 + \left(\frac{U}{U+D} - 1 \right) e^{-(U t_r)} \right) \right] \tau_{yo} + K \dot{\gamma}^m \quad (14)$$

The build-up rate coefficient, U , and break-down rate coefficient, D , are both rate functions, determined by conducting a curve-fitting procedure using the stress-overshoot experimental data for all shear rates with various aging times at a certain temperature. The parameter U is assumed to be independent of shear rate. On the other hand, D is assumed to be shear rate-dependent. Figure 11 shows the model prediction and experimental stress peak data at 30°C and 0.25 s⁻¹ shear rate with a 26.32 Pascal τ_{yo} value. U and D terms are determined using a curve-fitting procedure applied to Equation 14. Table-2 shows the U and D terms obtained using this approach.

Then, all the experimental data having different aging times and shear rates are applied to a curve-fitting procedure using Equation 12 at a constant temperature. It is assumed that D is shear-rate-dependent for shearing times greater than zero. This assumption is based on structure kinetic theory because the build structure is broken down (at a rate of D) into particles due to the shearing force. The D term is redefined as a function of shear rate, such that

$$D = d_1 + d_2 \dot{\gamma} \quad (15)$$

The numerical values of U , d_1 and d_2 coefficients at different temperatures based on this approach are presented in Table – 3.

Flow-Loop Experiments

Flow loop experiments were conducted on a flow loop system called the Dynamic Testing Facility at The University of Tulsa – Drilling Research Projects (DTF) (Figure 12). The purpose of the flow loop experiments is to validate the proposed model in terms of pressure peaks and time-dependent pressure response. A schematic drawing of the DTF test section is presented in Figure 13. The test system is composed of seven main components: mixing or recirculation tank, mud pump, temperature sensors, heating/cooling system with an annular heat exchanger, vertical pipe test section (2 in – 0.5 in), pressure sensors and a computer-based data acquisition system. The test procedure starts by circulating the SBM to get a homogeneous mixture and adjusting the test temperature. After this, circulation in the vertical annular section is bypassed and circulation continues in the horizontal pipe section by closing the valve and waiting for different resting times for the development of gelled structures. As the desired aging time is obtained, circulation is diverted to the vertical test section and to measure the gel breaking pressure is measured by circulating the non-gelled fluid. Circulation in the vertical section continues until steady pressure readings are observed.

Figure 14 is a typical example of DTF test results. In this

figure, the experimental results at 24°C with 4.7 gpm flow rate at different aging times emphasize the gelation or structure development effect on transitional pressure responses. After 1 minute of aging, the pressure peak starts at 0.08 psi/ft and reaches a 0.022 psi/ft steady state gradient after 3.5 seconds of peak observation. The 10-minute peak starts at 0.1 psi/ft and takes around 4 seconds to reach 0.0245 psi/ft steady state readings. This pressure decline takes more than 5 seconds, from 0.14 psi/ft to 0.021 psi/ft, when the fluid is stagnant for 30 minutes in the vertical section. Pressure readings show 0.174 psi/ft and drop to 0.026 psi/ft in around 5 seconds when the SBM is subjected to a 60-minute aging period. In this figure, all the pressure curves with different aging times come to the same steady state pressure gradient, 0.025 psi/ft. This is expected because aging does not affect the equilibrium pressure drop and if the temperature is kept constant at a constant flow rate, the steady state frictional pressure loss gradient should be constant.

The pressure peaks in Figure 14 increase as the resting period increases. This is due to stronger gel structures with longer aging times. For example, the maximum pressure gradient doubles when the aging time increases from 1 minute to 60 minutes and the pressure peak at 30 minutes is 40% higher than the pressure peak at 10 minutes. This is illustrated in Figure 15.

Presented in Figure 16 is the DTF experiment conducted at a constant 4.65 gpm flow rate at different temperatures. Similar to the pressure peaks, steady state pressure gradients tend to decrease when fluid temperature increases.

Frictional Pressure Loss for YPL Fluids

A calculation methodology for frictional pressure losses of YPL fluids in annular geometries is presented in Advanced Drilling and Well Technology.¹⁶ The authors assume that the inner pipe is concentrically located in the annular section, fluid is incompressible and flow is isothermal. Total flow area in the annular geometry is represented by the product of h and w . According to Newton's Law of Motion, the pressure gradient is defined as a function of wall shear stress, τ_w :

$$\tau_w = \frac{h}{2} \left(- \frac{dP}{dl} \right) \quad (16)$$

The flow behavior of a YPL fluid can be defined as:

$$Q = \left\{ \begin{array}{l} \frac{w h^2}{2 K^{\frac{1}{m}} \tau_w^2} \left(\frac{1}{1+2m} \right) (\tau_w - \tau_y)^{\frac{1+m}{m}} \\ \left(\tau_w + \frac{m}{m+1} \tau_y \right) \end{array} \right\} \quad (17)$$

It should be noted that Equations 16 and 17 are developed without considering thixotropy or time-dependent effects.

Dynamic yield stress is constant in the equations; i.e., they are shear rate or velocity-independent. The first term in the proposed model, Equation 12, represents the instantaneous yield stress, which is a time-dependent property of the thixotropic system, where

$$\tau_y(t) = \left\{ \left[(1 + (\xi - 1)e^{-(U t_r)}) - (\xi) \right] e^{-(U+D)t} + (\xi) \right\} \tau_{y0} \quad (18)$$

$$\text{where } \xi = \frac{U}{U + D}$$

If the dynamic yield stress, τ_y , is replaced by the instantaneous yield stress, $\tau_y(t)$, in Equation 17 and flow rate is converted to average velocity, the flow equation of a YPL fluid in an annular geometry for a given average velocity in structured fluids becomes

$$\frac{12v}{D_o - D_i} = \frac{(\tau_w - \tau_y(t))^{1+m}}{K^m \tau_w^2} \left(\frac{3m}{1+2m} \right) \left(\tau_w + \frac{m}{1+m} \tau_y(t) \right) \quad (19)$$

Equation 19 must be solved iteratively to find shear stress at the wall.

Model Validation

The stress equation, Equation 12, is validated with the predicted and measured shear stress values. In addition to shear stresses, the calculated frictional pressure loss gradient, based on the stress equation, is compared with results obtained from the flow loop experiments.

Figure 17 compares the model prediction and SOT at a 0.81 s^{-1} shear rate and a test temperature of 17°C . In this analysis, SBM is subjected to a 30-minute aging period. The model gives a 30.47 Pascal value when the measured value is 30.92 Pascal. The stress curve obtained from the prediction shows a faster stress decline response compared to measured data. This may be due to the assumed linear relationship between the shear rate and break-down rate coefficient shown in Equation 15.

Figure 18 shows the calculated frictional pressure loss gradients, based on the stress equation, and the measured values at a 4.7 gpm flow rate and 24°C temperature with 60 of minutes resting. The pressure peak encountered in the experimental work is 0.198 psi/ft and a 0.194 psi/ft pressure gradient is calculated based on the time-dependent constitutive equation. The predicted and measured equilibrium pressure gradient values are both 0.038 psi/ft. These results validate the model's applicability for practical applications.

Conclusions

The aim of this study is to analyze the gelation and time effects on thixotropic fluid rheology and to estimate the pump

start-up pressure and transitional pressure behavior. A time-dependent constitutive equation was proposed in order to address these points. Rheological and flow experiments were conducted to characterize the deformation behavior of a test fluid and to show the applicability of the equation. From this study, the following conclusions can be reached:

- The static value of yield stress (gel strength), τ_{ys} , increases as aging time increases. Also, it increases as the fluid temperature decreases.
- The energy required to break the gel structure and to initiate fluid flow shows a response similar to the stress behavior in thixotropic fluids. Pressure peaks increase with increasing resting time.
- Experimental results show that the magnitude of the measured pressures at peak points is significantly higher than the pressure values after equilibrium conditions are established.
- The model can estimate pressure gradients for both peak and equilibrium conditions with reasonable accuracy.

Nomenclature

a	= Build-up constant, s^{-1}
b	= Break-down constant, s^{-1}
c	= Constant in Equation 3
d	= Curve fitting parameter in Equation 1
D	= Breakdown rate coefficient in Equation 8, s^{-1}
G	= Shear modulus, Pa
h	= Height, in
H	= Constant in Equation 1, Pa
K	= Consistency index, $\text{Pa}\cdot\text{sm}$
l	= Length, in
m, n	= Flow behavior index
P	= Pressure, Pa
Q	= Flow rate, m^3/s
t	= Time, seconds
t_r	= Resting (aging) time, minutes
t_{ref}	= Reference time, seconds
U	= Buildup rate coefficient in Equation 5, s^{-1}
v	= Velocity, m/s
w	= Wide, in
y	= Clearance from center of slot, in

Greek letters

γ_e	= Equilibrium strain
γ	= Shear rate, s^{-1}
γ_w	= Wall shear rate, s^{-1}
μ_{po}	= Plastic viscosity, P
λ	= Structure parameter
λ_e	= Equilibrium structure parameter
λ_o	= Initial structure parameter
τ	= Shear stress, Pa
τ_g	= Gel strength, Pa
τ_o	= Stress overshoot peak, Pa
τ_y	= Dynamic yield stress, Pa
τ_{y0}	= Limiting value of static Yield stress, Pa
τ_{ys}	= Static yield stress, Pa
τ_w	= Average wall shear stress, Pa

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Table – 1 Rheological properties determined using equilibrium stress values

Temperature (°C)	τ_y (Pa)	K (Pa-sec ^m)	m
4	2.59	8.55	0.18
10	5.73	1.83	0.39
17	3.64	1.20	0.45
24	2.76	1.36	0.36
30	2.12	1.40	0.34
40	1.91	1.12	0.36
50	2.71	0.61	0.42

Table – 2 U and D values to be used for determining stress peaks

Temperature (°C)	U (min ⁻¹)	D (min ⁻¹)
4	0.013511	0.056527
10	0.014923	0.032492
17	0.015806	0.021119
24	0.015766	0.019933
30	0.015714	0.024955
40	0.015440	0.027556
50	0.016539	0.022459

Table – 3 Coefficients of U and D for determining time-dependent shear stress

Temperature (°C)	U (sec ⁻¹)	d_1 (sec ⁻¹)	d_2 (sec ⁻¹)
4	0.864	14.954	-0.142
10	1.325	8.751	0.005
17	1.554	11.117	0.462
24	0.915	7.798	0.295
30	1.256	12.747	0.564
40	1.788	17.890	0.784
50	1.500	11.392	0.379

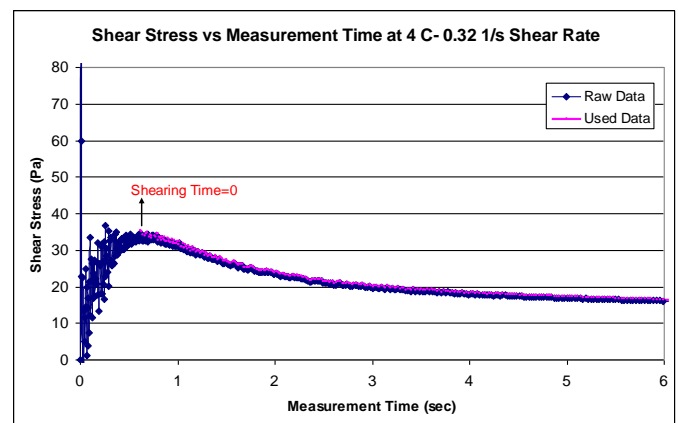


Figure 1: Example of data processing

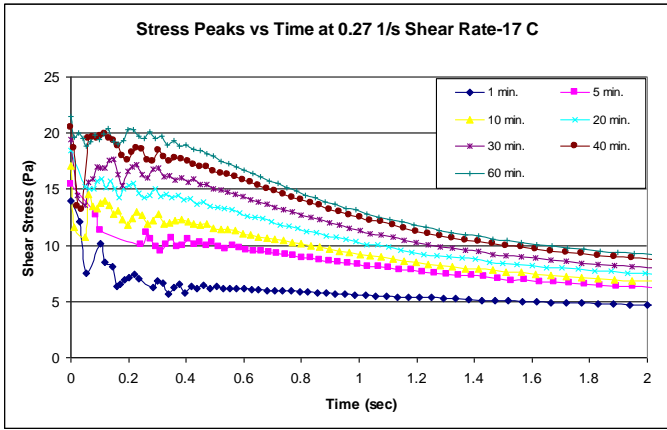


Figure 2: SOT Results (limited time scale)

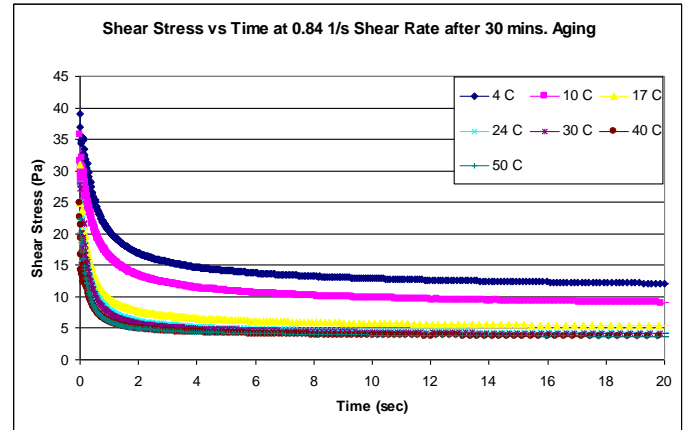


Figure 5: Temperature Effect on SOT Results at 0.84 s^{-1} – 30 minutes aging

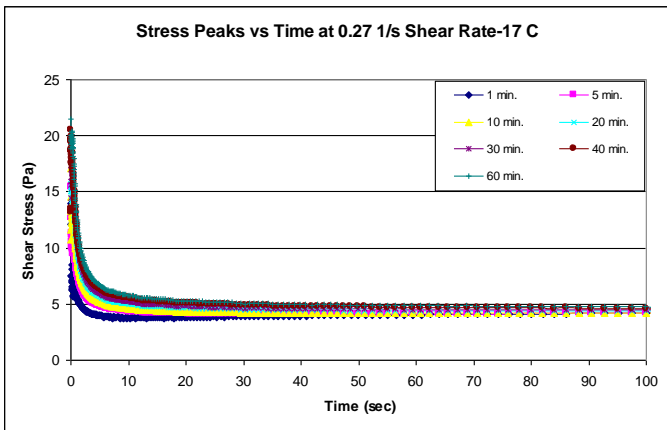


Figure 3: SOT results (full version)

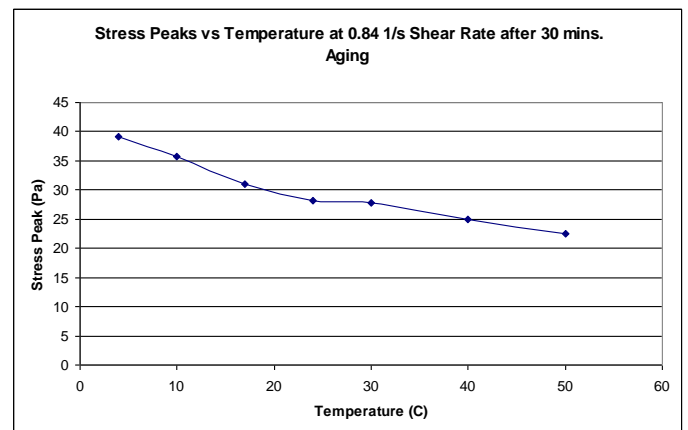


Figure 6: Temperature effect on stress peaks at 0.84 s^{-1} – 30 minutes aging

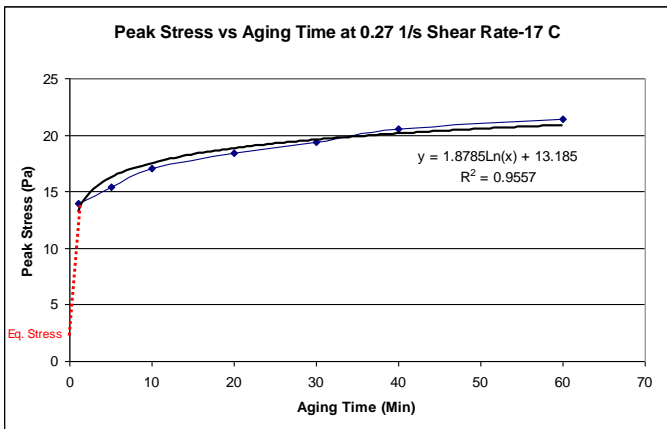


Figure 4: Aging effect on Stress Peaks at 0.27 s^{-1} and 17°C

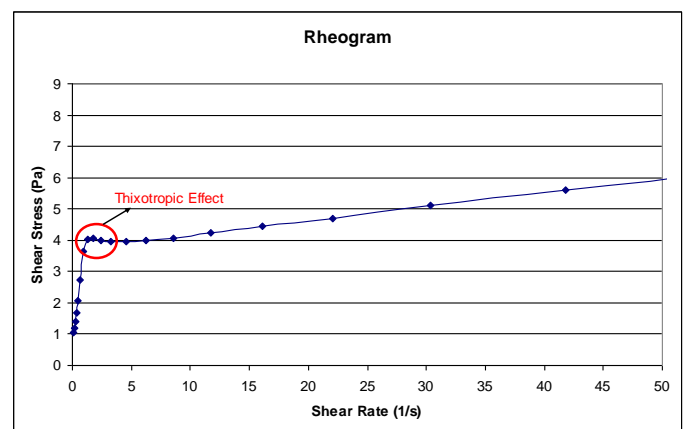


Figure 7: Thixotropy effect on rheogram

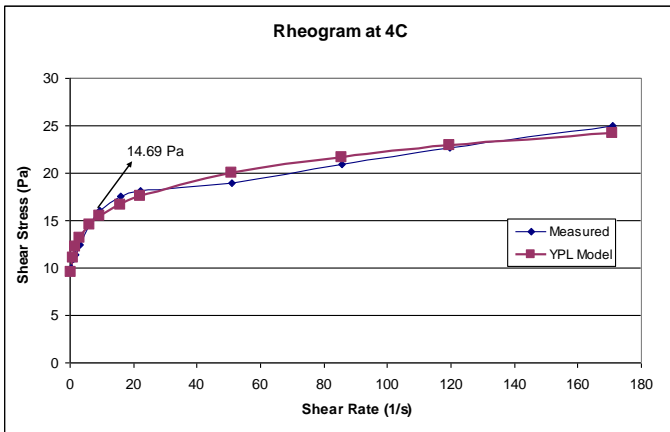


Figure 8: Rheogram at 4°C

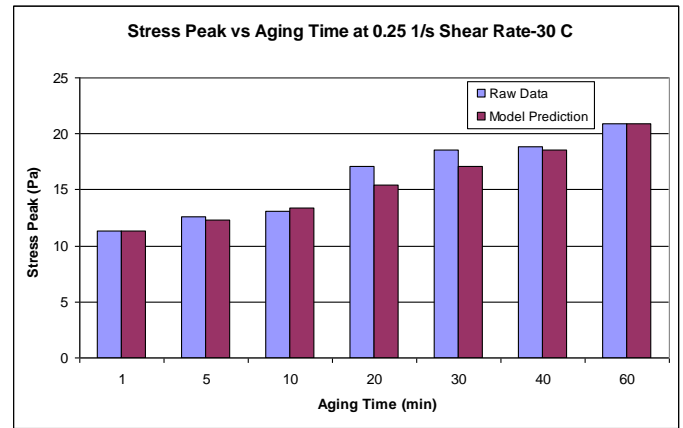


Figure 11: Experimental and model stress peak comparison at 0.25 s⁻¹ and 30°C

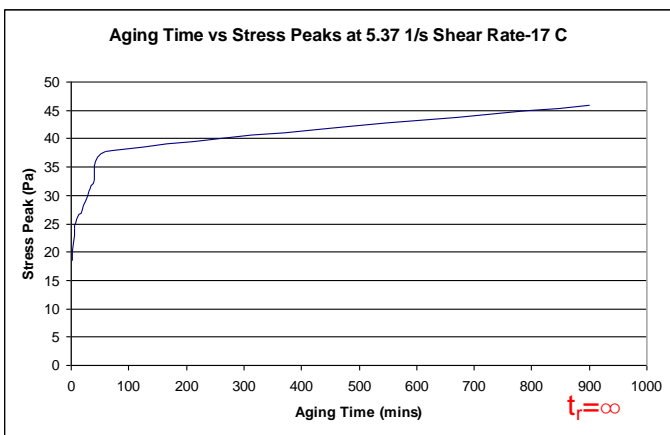


Figure 9: Stress peaks at 5.37 s⁻¹ and 17°C



Figure 12: Dynamic Testing Facility (DTF)

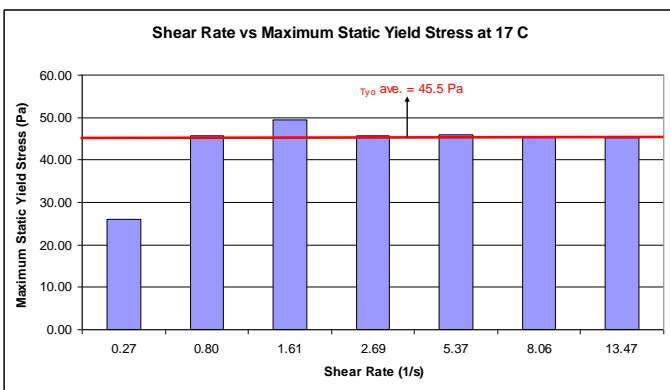


Figure 10: Shear rate effect on τ_{yo} at 17°C

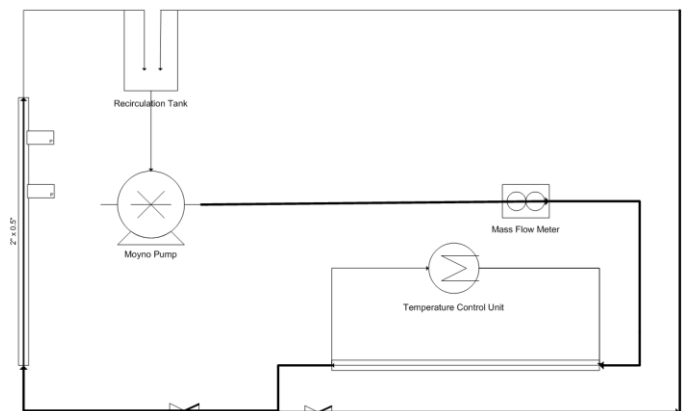


Figure 13: Schematic of experimental section of DTF

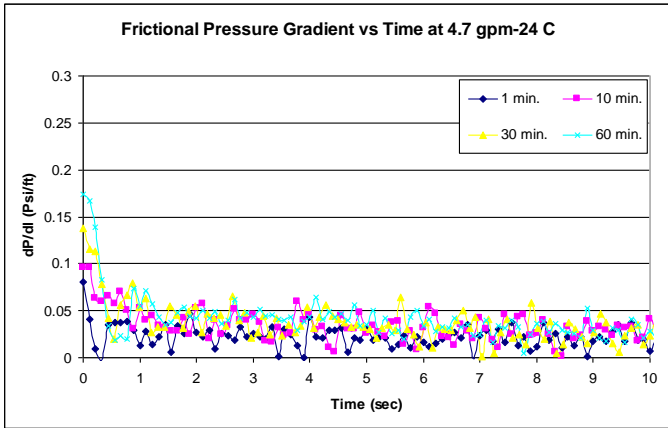


Figure 14: DTF results at 4.7 gpm and 24°C

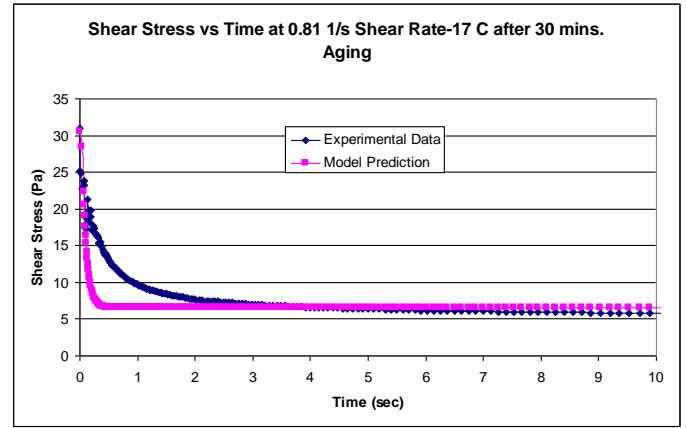


Figure 17: Stress model-experimental data comparison at 0.81 s^{-1} and 17°C - 30 minutes aging

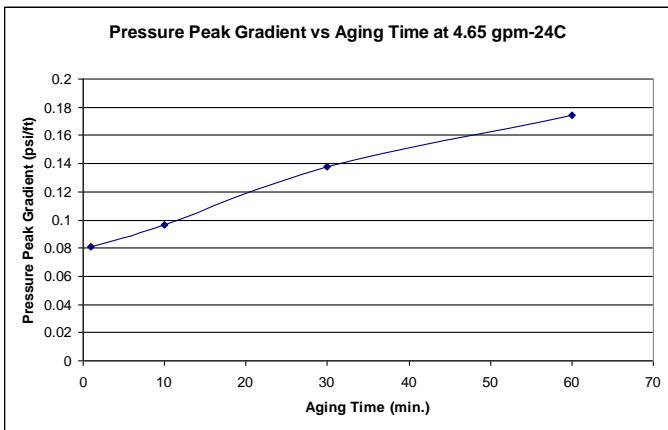


Figure 15: Aging effect on pressure peaks at 4.65 gpm and 24°C

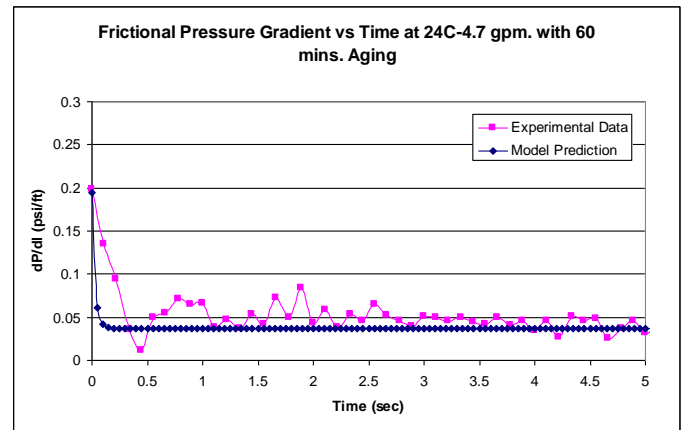


Figure 18 Frictional pressure gradient, model-experimental Data comparison at 4.7 gpm and 24°C - 60 minutes aging

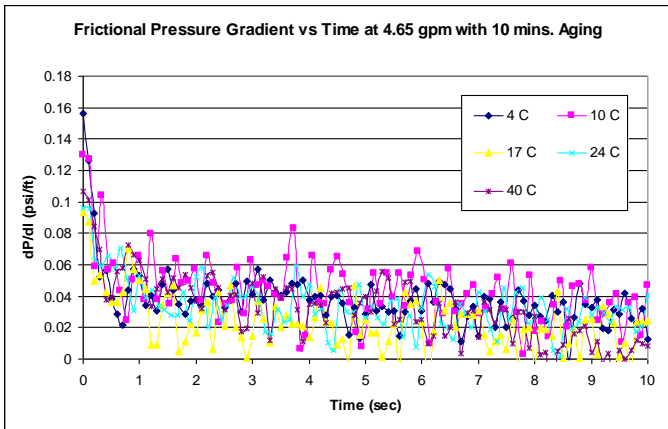


Figure 16: Temperature effect on DTF results at 4.65 gpm-10 minutes aging