



An Experimental Study on Structural and Thermal Stability of Water-Based Drilling Fluids

Ali Ettehadi^{1*}

^{1*} Izmir Katip Çelebi University, Faculty of Engineering and Architecture, Department of Petroleum and Natural Gas Engineering, Izmir, Turkey (ORCID: 0000-0002-4213-7510), ali.ettehadi@ikcu.edu.tr

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Abstract

Thermal stability of water-based drilling fluids is an essential factor especially through drilling geothermal and deep oil and gas wells. The chemical and physical properties of a drilling fluid system are substantially affected by high temperature and consequently lead to excessive gelation and formation damage issues. As a result of high temperature, formation damage might result from high fluid losses and reaction with formation fluid salts and hydroxides. This study is an attempt to investigate the thermal stability of clay base drilling fluids using thermal cycle testing. This test is a part of stability testing that allows determining if a fluid system remains stable under various conditions. This type of test can be applied to the drilling fluid systems and puts the sample through a series of extreme and rapid temperature change encountering in during fluid circulation in a geothermal well. Less toxicity as well as commercial and economical availability of clays make them an inevitable component for drilling fluid systems. A type of sepiolite clay taken from Eskisehir in Turkey and Wyoming bentonite as the API reference clay were considered to prepare freshwater weighted, unweighted, and solid contaminated fluid systems. API recommended and oscillation amplitude sweep tests were firstly carried out to evaluate the mechanical stability of selected fluid systems. The samples were then subjected to five thermal cycles from 25° C to 150° C. The relative change of the viscosity value compared to the value at the start of the thermal cycles was used as a measure of the structural changes in the fluid systems. The sample that shows a small value for the relative structural change at the end of the thermal cycles has the lowest decrease in the viscosity and hence the highest thermal stability. Discovery Hybrid Rheometer (DHR-II) was used to apply the oscillation and thermal cycle testing.

Results revealed that sepiolite based muds formulated in this study tolerate stability problems resulted from high and rapid temperature variation. Obtained appropriate thermal rheological properties as well as thermal cycle test results were strong indicators for the effectiveness of sepiolite muds. This study can help the oil and geothermal industry to be more familiar with a high-temperature stable sepiolite clay to prepare high-performance drilling fluids.

Keywords: Drilling fluid, Thermal stability, Sepiolite, Thermal cycle.

Su Bazlı Sondaj Sıvılarının Yapısal ve Isıl Kararlılığı Üzerine Deneysel Bir Çalışma

Öz

Jeotermal, derin petrol ve gaz kuyularının sondaj operasyonlarında su bazlı sondaj çamurlarının termal stabilitesi (ısı kararlılığı) oldukça önemli bir parametredir. Sondaj akışkanının kimyasal ve fiziksel özellikleri, yüksek sıcaklıktan önemli ölçüde etkilenmekte ve sonuç olarak bu durum aşırı jelleşme ve formasyon hasarı sorunlarına yol açmaktadır. Yüksek sıcaklığın sonucu olarak, formasyon hasarı, yüksek sıvı kayıpları ve sondaj sıvısının formasyondaki tuzlu su ve hidroksitlerle etkileşiminden kaynaklanabilmektedir. Bu çalışmada, kil bazlı sondaj çamurlarının termal stabilitesi termal döngü testini kullanarak incelenmiştir. Bu test, akışkan yapısının farklı koşullar altında kararlı kalıp kalmadığını gösteren etkin bir stabilite (kararlılık) testi olarak bilinmektedir. Bu test, jeotermal ve derin petrol ve gaz kuyularında sondaj akışkanının karşılaştığı ani ve hızlı sıcaklık artışlarını canlandırmak için sondaj akışkanına bu çalışma

* Corresponding Author: ali.ettehadi@ikcu.edu.tr

kapsamında uygulanmıştır. Çok az toksik etki göstermesinden, ekonomik uygunluğundan ve kolay bulunabilirliğinden dolayı killer sondaj akışkan sistemleri için kaçınılmaz bir katkı maddesi olarak tanınmaktadır.

Eskişehir, Türkiye'den alınan sepiyolit kili ve API referanslı Wyoming bentonite kili, saf su kullanılarak; ağırlaştırılmış, ağırlaştırılmamış ve kirletilmiş çamur örnekleri hazırlanmıştır. Osilasyon genlik süpürme testleri öncelikle seçilen akışkan örneklerinin mekanik kararlılığını incelemek için uygulanmıştır. Ayrıca hazırlanan örnekler 25° C den 150° C ye kadar artarak oluşan beş termal döngü testine tabi tutulmuştur. Termal döngü testinin başlangıcındaki değere kıyasla viskozite değerinin göreceli değişimi, akışkan sistemlerindeki yapısal değişikliklerin bir ölçüsü olarak kullanılmıştır. Termal döngü testinin sonucuna göre, en az değeri göreceli yapısal değişim gösteren çamur örneği, viskozitede en küçük düşüşe ve dolayısıyla en yüksek termal stabiliteye sahiptir. Discovery Hybrid Rheometer (DHR-II) aleti, osilasyon ve termal döngü testlerini uygulamak için kullanılmıştır.

Bu çalışmanın sonuçlarına göre, formüle edilen sepiyolit çamurunun yüksek ve ani sıcaklık değişimlerine karşı stabilitesini (kararlılığını) koruduğu gözlemlenmiştir. Elde edilen uygun termal reolojik özellikler ve termal döngü test sonuçları, sepiyolit çamurlarının etkinliği için güçlü bir göstergedir. Bu çalışma, yüksek performanslı sondaj sıvıları hazırlamak için yüksek sıcaklığa duyarlı olan sepiyolit kilinin petrol ve jeotermal endüstrisinde daha fazla tanıtımına ve kullanılmasına yardımcı olmaktadır.

Anahtar Kelimeler: Sondaj Çamuru, Termal Stabilite, Sepiyolit, Termal Döngü.

1. Introduction

Thermal stability of drilling fluids is totally dealing with their rheological properties at high temperature along with active solid intrusion that are the major problems frequently encountered during geothermal drilling operations. Furthermore, drilling at high temperature are expected to provoke formation damage and gelation problems stemmed from chemical and physical changes in the structure of the drilling fluid (Dahab 1991). These problems cause loss circulation, stuck pipe, wellbore instability, difficulty in cement jobs and wellbore diameter reduction that increase the well cost by an average of at least 15% in geothermal wells (Carson and Lin 1982). Therefore, the rheological properties of the drilling fluids should be characterized in details to avoid these problems. Bentonite based mud is mainly used to drill the overburden well sections with high temperature gradients and formations exhibiting instability, particularly collapse tendency. Increasing in viscosity of bentonite based mud after subjected to high temperature changing from 150 to 200 °C, triggers pipe sticking as a very severe problem and results in increasing non-productive time. Polymer based drilling fluids can be used as another drilling fluid system to support the well and provide efficient cutting transport. However, most of commercially available polymer additives are limited temperature of almost 90 °C (Otte, Pye, and Stefanides 1990). Synthetic and oil based muds are used at high temperatures, where bentonite and polymer based muds are not sufficient. Amani et al. (2012) made a comparative study of using water based mud and oil based mud in order to find the most appropriate mud type in high temperature and high pressure (HTHP) fields. The experimental results demonstrated that the tolerance of oil based mud is more suitable than water based mud in HTHP fields (Amani, Al-Jubouri, and Shadravan 2012; Abduo et al. 2016). Even though oil based mud systems are more suitable in terms of technical performances, the challenges of using oil based mud systems are stated as inconvenient usage, environmental problems and high expenditures (Ahmadu et al. 2019). In contrast to the oil based drilling fluids, the water based drilling fluids are inexpensive and environmental friendly. The gap of alternative and effective water based drilling fluid system at high temperatures encouraged researchers to perform more study. Sepiolite based mud was introduced as a temperature and salinity resistant fluid system in some special studies. Numerous investigator have conducted various studies on water based mud to investigate rheological behavior and filtration properties in high temperature and high saline environments. The common point of the all research is that several additives were added to the sepiolite based mud in order to obtain appropriate viscosity and

filtration properties of mud samples (Carney and Meyer 1976; Carney and Guven 2007, 1982; Hilscher and Clements 1982; Moussa and Al-Marhoun 1985; Guven, Panfil, and Carney 1988; Zilch, Otto, and Pye 1991; U Serpen, Hacıislamoglu, and Tuna 1992; U. Serpen 1999; Umran Serpen 2000).

The effects of grain size, mixing time, mixing speed and gelling time in water based drilling fluid prepared with sepiolite clay at high temperatures were investigated (Altun and Serpen 2005; Altun, Osgouei, and Serpen 2010). Altun et al. (2015) experimentally investigated the rheological and filtration properties of drilling fluids prepared with sepiolite clay. The experiments were carried out with and without some commercial additives for different temperatures and pressures in different salinity (Altun et al. 2015). Needaa et al. (2016) studied the effects of sepiolite nanoparticles on bentonite-based mud in terms of rheological properties and loss filtration under different temperature and pressure. They observed that particularly under high temperature and high pressure conditions, adding sepiolite nanoparticles to bentonite based mud made rheological properties more stable. Sepiolite nanoparticles reduced fluid loss and permeability under reservoir pressure and temperatures. Additionally, sepiolite nanoparticles were stated as an ideal additive for bentonite based mud (Needaa et al. 2016). In spite of existing several studies in literature about high temperature rheological properties of drilling fluid, thermal in-situ stability of drilling fluid has not been well known enough. The rheological properties measured by conventional viscometer might cause some sort of deceptive results that were far from reality. The reason may stems from the preparation process when the mud samples were required to be cooled down in order to be ready for viscometer measurements. It is well-known fact that the viscosity of liquid phase of mud samples changes with temperature variations. The viscosity of most water base fluids will be decreased with increasing temperature. However, it was not possible to measure the in-situ thermal rheological properties of mud samples. In this study, Discovery Hybrid Rheometer (DHR-II) was used instead of conventional viscometer (such as Fann VG viscometer), to characterize most down-to-earth thermal properties of a drilling fluid.

The objective of this study is to investigate rheological properties of water-based drilling fluids at high temperature and pressure conditions using DHR-II. The experiments were carried out on sepiolite and bentonite muds based on Thermal Cycle Testing method, determining whether the fluid system is thermally stable or not under various conditions.

2. Material and Method

In this experimental study, bentonite and sepiolite based drilling fluids were prepared in laboratory under four different states as without additive, unweighted, weighted, and weighted-contaminated. Formulated Sepiolite based fluid systems (Altun and Osgouei, 2004) and the bentonite/polymer fluid systems were compared in an attempt to determine the thermal stability of the drilling fluids. Regarding comparative studies, the main objective is to develop the thermo-structural properties of drilling fluid that can operate under high-temperature conditions.

The raw sepiolite clay (commercial product identified as Turk Taciri Bej) was obtained from AEM Company (AEM 2014) in Eskisehir / Turkey. Several studies have shown that sepiolite clay, which is a thermally stable viscosifier, can be used under harsh drilling conditions due to its essential properties such as providing sufficient rheological properties, exhibiting less gelation tendency, and reducing fluid loss when used with some suitable additives (Carney and Meyer 1976; U Serpen, Hacıislamoglu, and Tuna 1992; Altun and Serpen 2005, Altun and Osgouei, 2014). Additionally, commercial Wyoming bentonite clay (QUIK-GEL) supplied by Baroid Company was used as the primary additive for bentonite/polymer drilling fluid. QUIK-GEL viscosifier is an easy-to-mix, finely ground (200-mesh), premium-grade, high-yielding Wyoming sodium bentonite. QUIK-GEL viscosifier imparts viscosity, fluid loss control and gelling characteristics to freshwater-based drilling fluids. The commercial Wyoming bentonite might contain some surface active agents (Url-2021). Table.1 summarizes other additives in terms of technical grades, used in the formulation of both type of drilling fluid systems.

2.1. API Recommended Measurements

The drilling fluid samples were subjected to the composition of 350 ml of distilled water including sepiolite, bentonite clay, and various concentrations of commercially additives through both unweighted and barite-weighted systems via API RP-13B Protocols. In order to prepare 14 lb/gal barite-weighted bentonite/polymer and sepiolite-based drilling fluid samples, amounts of 1077 kg/m³ (378 lb/bbl) and 1057 kg/m³ (371 lb/bbl) barite were added to mud systems, respectively. Throughout all experiments, 143 kg/m³ (50 lb/bbl) standard evaluation clay (OCMA) was used to simulate active clay invasion during drilling operations. The compositions of sepiolite and bentonite drilling fluids in four states are shown in Table 1.

Prepared drilling fluid samples were hot rolled for 16 hours at 25 and 150° C. Afterwards, the hot rolled sample cells were cooled down at room temperature. Rheological properties such as apparent viscosity (AV), plastic viscosity (PV), yield point (YP), and gel strength (GS) were then measured with Fann Model 35 Couette type viscometer at 25° C (80° F) and 49° C (120° F). The static filtration properties of the samples were measured using a high temperature high pressure (HTHP) filter press.

2.2. Oscillation and Freeze-Thaw Cycle Test Using Rheometer

Mechanical and thermal stability of drilling fluid samples were measured based on oscillation and Freeze-Thaw Cycle Test. All measurements in this study were carried out with TA Discovery Hybrid Rheometer (DHR II) equipped with a Peltier plate, 60 mm parallel plate geometry, and pressure cell unit.

Mechanical properties of the fluid samples were evaluated in terms of oscillation amplitude test results. The samples were tested in an amplitude sweep at temperature of 25°C and 150°C for the mechanical properties analysis and yield stress measurements. Moreover, the results of thermal loop test based on Freeze-Thaw Cycle Test were used to analyze the thermal stability of fluid samples.

2.1.1. Oscillation Amplitude Sweep Tests

Strain amplitude measurement based on the storage and loss moduli (G' , G'') is the first step in characterizing visco-elastic behavior. In an amplitude sweep test, the amplitude of deformation is varied while the frequency is held constant. Sample will behave visco-elastically under small strain, when the material internal structure is not disrupted. Sample structure will be deformed by increasing strain to a critical strain. Therefore sample structure deformation changes from linear viscoelastic response to nonlinear viscoelastic response. A strain sweep test is carried out to determine the extent of the sample's linearity. Ramping strain moduli decline, G'' exceeds G' eventually and the sample becomes progressively more fluid-like. Loss factor [$\tan \delta = (G''/G')$] represents the strength of the colloidal forces. A loss factor less than 1 indicates that interaction forces between colloidal particles are highly dominated (viscoelastic solid) and sedimentation could occur. A high loss factor at given concentration publishes that the particles are mainly unconsolidated (viscoelastic fluid). An intermediate loss factor is desired for a stable system. The amplitude sweep is used to determine the Linear Visco-Elastic region (LVE) of the sample. It is also used to identify structural stability and dynamic yield point. The amplitude sweep test was carried out at an angular velocity of 10 rad/s at strains from 0.001% to 1000% using parallel plate ($\varnothing 60$ mm) at 25°C. This test was performed at same amount of angular velocity and strain interval using pressure cell unit at 150 °C to avoid evaporation. All samples were conditioned by pre-shearing at the rate of 400 (sec-1) for 120 second prior to test.

2.1.2. Freeze-Thaw Cycle Testing

Fluid circulation is initiated by pumping drilling mud from surface to the borehole and completed when it backs to the surface through annuli. Freeze-Thaw Cycle test is a thermal loop test that can be conducted to evaluate thermal stability of drilling fluid system. The drilling fluid samples were subjected to five thermal cycles from +25 °C to 150 °C to simulate the thermal stresses while drilling different formations. Pressures cell unit of DHR-II was used to perform this test.

During the cycles, the samples were measured at frequency of 1 Hz (angular frequency of 10 rad/s) and a shear rate of 200 (sec-1). The heating and cooling rate was set to 3 °C/min which is a relatively high value. To adjust the data after the each ramp 600 sec was replaced as soak time with an oscillation time step. A degree of the structural changes in the fluid sample can be defined as the relative change of the value of the viscosity (η) compared to the value at the start of the thermal cycles. All necessary calculations were performed with the rheometer software. The thermal cycles can also be carried out with the rheometer software using the internal loop. The parameters have to be set for five cycles according to the requirements for a particular sample. The temperature is checked at the start of the test to ensure that the measurement is started only after a thermal equilibrium has been reached.

Table 1. Compositions of drilling fluid samples.

Substance	Quantity (kg/m ³)					
	Bentonite Mud			Sepiolite Mud		
	Base SM1	Unweighted SM2	Weighted -Contaminated SM3, SM4*	Base SM5	Unweighted SM6	Weighted - Contaminated SM7, SM8*
Sepiolite	None	None	None	50	50	50
NaOH	None	0.17	0.17	None	None	None
Soda Ash	None	0.286	0.286	None	0.286	0.286
Bentonite	28.5	28.5	28.5	None	None	None
Polymer - 1	None	5.7	5.7	None	5.7	5.7
Polymer - 2	None	None	None	None	8.6	8.6
PAC-LV	None	5.7	5.7	None	None	None
Barite	None	None	1077	None	None	1075
*OCMA	None	None	143	None	None	143

* Standard evaluation clay (formerly OCMA) added only in weighted - contaminated muds (SM4, SM8)

3. Results and Discussion

3.1. API Recommended Test Results

Rheological and filtration properties of bentonite and sepiolite based muds were listed in Table 2 and 3, respectively. Two important parameters of drilling fluids that should be monitored while drilling operation are yield point (YP) and gel strength (GS). Yield stress indicates the ability of drilling fluid to carry cuttings to the surface during dynamic condition and frictional pressure is directly related to the yield stress (Barnes 1999; Moller et al. 2009). A drilling fluid with high yield stress leads to high pressure loss when mud is being circulated in the wellbore. During geothermal drilling, at high temperatures (> 150° C) frequently fluid systems tend to be flocculated and high amount

of yield stress will be appeared. Moreover, active clay contamination makes yield stress more excessive and rises the frictional pressure losses up.

The YP values measured at 49° C for all mud samples were shown in Figure 1 demonstrating the variation of YP versus mud density. At 25° C, unweighted bentonite and sepiolite based mud (without additives) provided a relatively high YP. Adding additives to the mud systems decreased YP at the rate of around 40% for the both fluid systems. With increasing density (from 8.6 to 14 ppg) the value of YP for both systems were not drastically changed at 25° C indicating minor effect of inert weighting agent material (barite). Both system at 25° C provided acceptable YP values within the upper and lower defined (Bourgoyne et al. 1991) ranges for clay base muds (Figure 1).

Table 2. Rheological and filtration properties of bentonite based mud.

Fluid system	Bentonite fresh water		Bentonite polymer (8.6 ppg)		Bentonite polymer (14 ppg)		Bentonite /polymer (14 ppg) contaminated		
Aging Temperature, °C	25		25		25		150		
Dial reading @ measurement temperature	Dial reading@ 24°C 49°C		Dial reading@ 24°C 49°C		Dial reading@ 24°C 49°C		Dial reading@ 24°C 49°C		
Rotor Speed (rpm/min)	600	54	52	63	50	97	68	42	34
	300	46	43	41	32	60	40	22	18
	200	42	40	32	25	45	30	16	13
	100	38	36	21	16	29	19	10	8
	6	29	24	6	4	6	4	3	2
	3	28	16	5	3	5	3	2	2
Plastic viscosity, Pa.s	0.008	0.009	0.022	0.018	0.037	0.028	0.02	0.016	0.016
Yield Point, Pa	21.98	20.55	19.59	15.29	28.66	19.11	10.51	8.60	8.60
*Gel strength, 10min./ 1min./ 10min.	23/26/37	15/15/18	6/9/20.	4/6/17.	6/10/26.	4/6/17.	2/3/3.	2/2/2.	2/2/2.
Apparent viscosity, Pa.s	0.027	0.026	0.0315	0.025	0.0485	0.034	0.021	0.017	0.017
pH	8.1	8.1	8	8	8.6	8.6	7.9	7.9	7.9
Water loss, cc (7.5/30min)	3/8.		1.8/3.6		2/4.		excessive		
Density (kg/m ³)	1024	1024	1030	1030	1677	1677	1737	1737	1737

* Multiply dial reading value by 0.478026 to obtain Pascal

Table 3. Rheological and filtration properties of sepiolite based mud.

Fluid system	Sepiolite fresh water		Sepiolite polymer (8.7 ppg)		Sepiolite polymer (14 ppg)		Sepiolite /polymer (14 ppg) contaminated		
	Dial reading@ 24°C	Dial reading@ 49°C	Dial reading@ 24°C	Dial reading@ 49°C	Dial reading@ 24°C	Dial reading@ 49°C	Dial reading@ 24°C	Dial reading@ 49°C	
Aging Temperature, °C	25		25		25		150		
Dial reading @measurment temperature	24°C		49°C		24°C		49°C		
Rotor Speed (rpm/min)	600	52	37	58	42	95	66	92	73
	300	46	32	38	27	59	40	58	46
	200	43	30	30	22	44	30	45	36
	100	37	26	20	15	28	19	30	24
	6	23	17	5	4	5	4	8	7
	3	23	17	4	3	4	3	6	6
Plastic viscosity, Pa.s	0.006	0.005	0.02	0.015	0.036	0.026	0.034	0.027	
Yeild Point, Pa	21.99	15.29	18.15	12.90	28.18	19.11	27.71	21.98	
*Gel strength, 10min./ 1min./ 10min.	22/24/31.	21/22/32.	3/3/5.	2/3/5.	4/5/10.	3/4/7.	7/9/15.	7/8/12.	
Apparent viscosity, Pa.s	0.026	0.0185	0.029	0.021	0.0475	0.033	0.046	0.0365	
pH	7.5	7.5	8	8	8.1	8.1	8.1	8.1	
Water loss, cc (7.5/30min)	50/122		2.2/5		2.1/4.8		3.1/8		
Density (kg/m ³)	1018	1018	1042	1042	1677	1677	1725	1725	

* Multiply dial reading value by 0.478026 to obtain Pascal

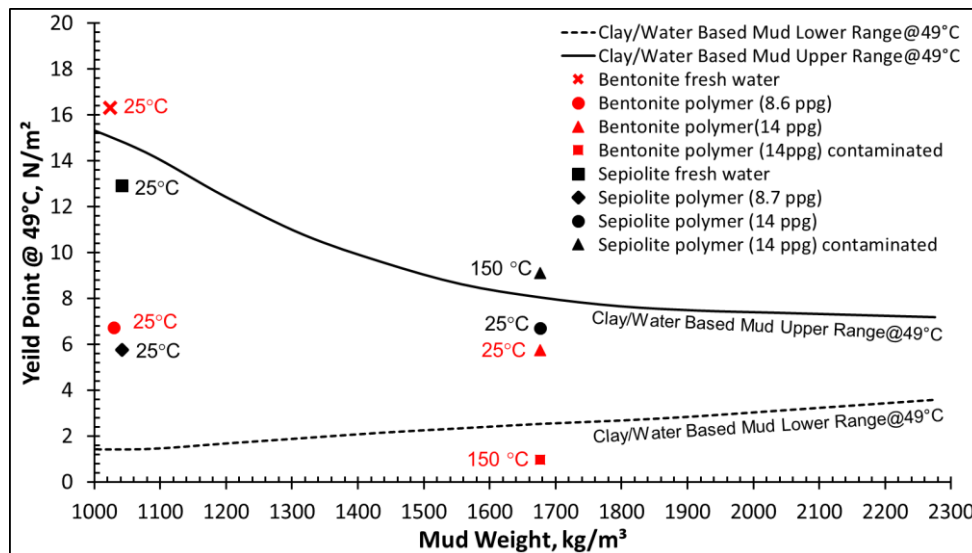


Figure 1. YP values measured for sepiolite and bentonite mud samples and compare with defined the upper and lower range for clay/water base muds (Bourgoyne Jr et al. 1986).

Increasing temperature to 150° C along with active clay intrusion made a noticeable difference in YP values of two fluid systems. The YP value for weighted bentonite mud system was remarkably decreased from 12 lb/100ft² at 25° C to 2 lb/100ft² at 150° C despite of active clay contamination. Gel strength of fluid system demonstrates the ability of the drilling fluid to suspend drill solids and weighted materials when circulation is ceased. If gel strength appears to be too progressive, the mud may require excessive pump pressures to break the gel and initiate the circulation (Tehrani 2007). Similar to the yield stress, high temperature and active contamination have deleterious effects on the drilling fluid gel strength. Generally, oil industry uses the peak value of dial reading in standard viscometer while a 3 rpm of rotor speed is directly applied to the mud sample to measure the initial 10 second and 10 minute “gel strengths”. Obtained results indicate that the initial 10 second and 10 minute of bentonite and sepiolite freshwater mud samples are relatively high requiring enormous pump power to break formed gel. Bentonite polymer muds (8.6 ppg and 14 ppg) provided noticeably high 10

minute gel strength (17 lb/100ft²) comparing to the sepiolite polymer based muds (8.7 ppg and 14 ppg) gel strength (5 and 7 lb/100ft², respectively). However, in the case of active clay intrusion at high temperature (150° C) the gel structure of bentonite polymer mud was broken down resulting in weak gel strength (2 lb/100ft²). Amount of 12 lb/100ft² gel strength for sepiolite base mud clarifying stable gel structure in spite of active clay contamination at 150° C. Fluid loss is also another determined parameter to evaluate efficiency of drilling fluid. The rheological properties should be considered along with fluid losses to be more realistic and accurate in drilling fluid performance. API standard test procedure based on cooling process after hot rolling might cause to measure fallacious rheological properties for a drilling fluid. Therefore, DHR rheometer was used to determine in-situ mechanical stability of both fluid systems.

3.2. Mechanical stability and flow point

Figures 2 and 3 picture the results of oscillation amplitude sweep test for bentonite and sepiolite mud samples, respectively.

Linear Visco-Elastic (LVE) range limit is increased from 10% in bentonite fresh water based mud without additives (SM1) to 99% in SM2 containing polymeric additives.

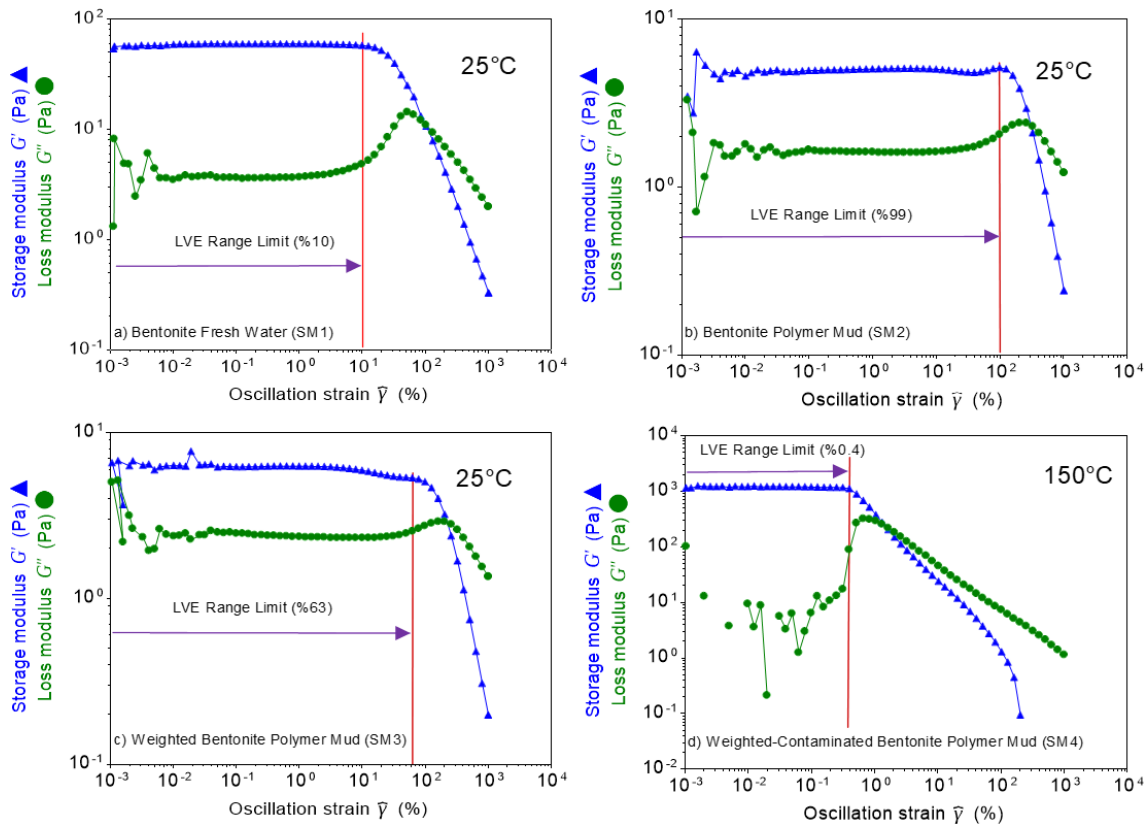


Figure 2. Oscillatory Amplitude Sweep Test Result (G' and G'' vs. γ). a) Bentonite fresh water (SM1), b) Bentonite polymer (SM2), c) Weighted bentonite polymer (SM3) @25° C, d) Weighted contaminated bentonite polymer mud (SM4)@150° C.

This is a clear evidence for the fact that adding polymer additives to the mud systems increases the structural stability of mud system. Structural stability of bentonite mud system decreases by adding barite as the LVE range limit decreases from 99% to 63% of strain. The LVE range limit decreased remarkably (0.4%) with increasing temperature and active clay intrusion to the mud system. This definitely indicates deterioration of mechanical stability of bentonite based mud at high temperature.

The samples coded as SM5 and SM8 behave visco-elastically under small strain and more solid-like. The limiting strain of the LVE range is about 0.5% and 6% for SM5 and SM8 sepiolite mud samples. Adding additives (selected polymers) to the sepiolite mud system makes it almost totally fluid-like (viscoelastic fluid) so that the amount of loss modulus G'' is a little bit more than storage modulus (G') for SM6. Adding barite increased the elasticity slightly as it is a kind of inert material (SM7). Similar to the bentonite mud systems, LVE range increases in sepiolite based mud by adding polymeric additives from 0.5% to 15.8% of strain. The limiting strain of the LVE range is about 5% for weighted sepiolite mud sample (SM7). Adding barite and other polymeric additives to the sepiolite mud system increased the length of the LVE range about 10 times (0.5% to 5%). Increasing temperature and intrusion of active clay to the sepiolite mud system (SM8) also increased the LVE ranges.

The LVE limits are illustrated for all mud samples through Figures 2 and 3. The elasticity of bentonite base mud samples are noticeably higher than sepiolite based mud samples. The linearity limits in bentonite fluid systems are much higher than sepiolite fluid systems at 25° C indicating higher structural stability against small strains. It means that too much pump power is required to break down the gel structure of bentonite based muds compare to sepiolite based systems at 25° C. In contrary, sepiolite based mud provided strength structure stability at 150° C (LVE limit 6%) compare to the bentonite based mud (LVE limit 0.4%).

The yield point (or yield stress) is the critical stress at which irreversible plastic deformation occurs. There are some conventional methods for the yield point evaluation. It can be calculated as the stress values where the value of the storage modulus has decreased by 5% compared to the value in LVE range. However the accurate method is to use of onset point of the storage modulus (G') curve. The flow point is the stress value at the crossover of storage and loss modulus. Figure 4 and Figure 5 show the storage and loss modulus as a function of the shear stress as well as the calculated flow point values for bentonite and sepiolite based muds, respectively. Table 4 lists the amount of yield stress and flow point values for all mud samples.

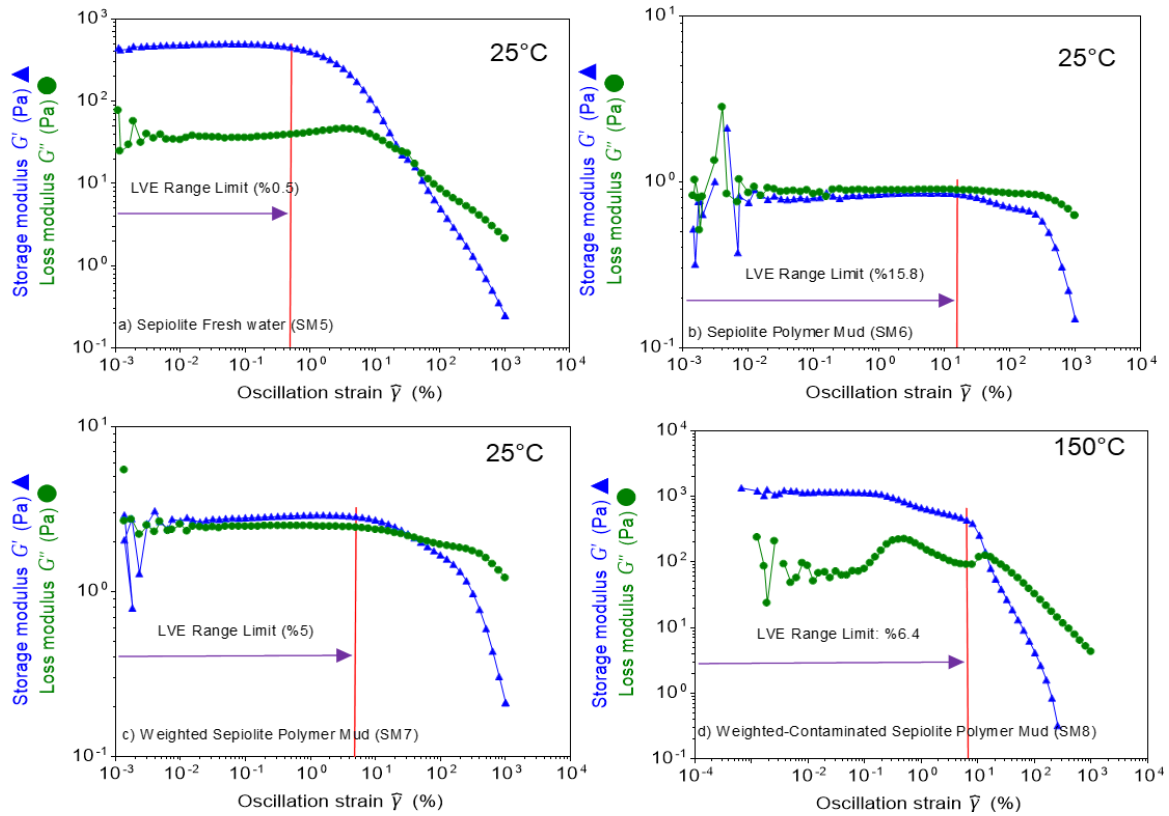


Figure 3. Oscillatory Amplitude Sweep Test Result (G' and G'' vs. γ). a) Sepiolite fresh water (SM5), b) Sepiolite polymer (SM6), c) Weighted sepiolite polymer (SM7) @25° C, d) Weighted contaminated sepiolite polymer mud (SM8)@150° C.

Yield stress of bentonite fresh water (SM1) decreases from 12.37 Pa to 9.42 Pa by adding polymeric additives to the mud system in bentonite polymer mud (SM2). Adding weighting agent (barite) to the mud system has a little effect on mechanical stability and measured parameters. However, mechanical parameters (yield stress and flow point) were decreased to 4.75 Pa with increasing temperature (150° C) in spite of large amount of active clay intrusion (143 kg/m³) in weighted –contaminated bentonite polymer mud (SM4). Flow point are also varied in the same trend with yield stress. This means that the bentonite polymer mud lose its colloidal structural strength during drilling formation with high temperature.

Adding polymeric additives to the sepiolite fresh water system (SM5) severely decreases mechanical stability and measured parameters. Yield stress and flow point decrease from 5.4 and 8.5 Pa in sepiolite fresh water mud system (SM5) to 0.28

and 1.2 Pa in weighted sepiolite polymer mud (SM7). Adding barite has not influenced the mechanical stability in sepiolite mud as expected. Unlike bentonite polymer mud system, the sepiolite polymer mud provides exceptional mechanical stability at high temperature in conjunction with high active clay intrusion. Yield stress and flow point of contaminated sepiolite polymer mud increases drastically to 31.5 Pa and 36 Pa, respectively at 150° C. Therefore, it can be inferred that sepiolite polymer mud can provide efficient hole cleaning while subjected to high temperature and high active solid contamination. Loss modulus (G'') in sepiolite polymer mud (SM6) exceeded storage modulus (G') even in LVE region indicating a more liquid behavior (viscoelastic liquid). It means that there are no such strong bonds between the individual molecules to provide gel structure, therefore no flow point was observed in the case of sepiolite polymer mud (SM6).

Table 4. Yield stress and flow point values for all mud samples.

Mud code	Discription	Temperature (°C)	Yield stress (Pa)	Flow point (Pa)
SM1	Bentonite fresh water	25	12.37	15.3
SM2	Bentonite polymer	25	9.42	10.25
SM3	Weighted bentonite polymer	25	8.89	9
SM4	Weighted-contaminated bentonite polymer	150	4.75	4.98
SM5	Sepiolite fresh water	25	5.4	8.5
SM6	Sepiolite polymer	25	0.28	-
SM7	Weighted sepiolite polymer	25	0.28	1.2
SM8	Weighted-contaminated sepiolite polymer	150	31.5	36

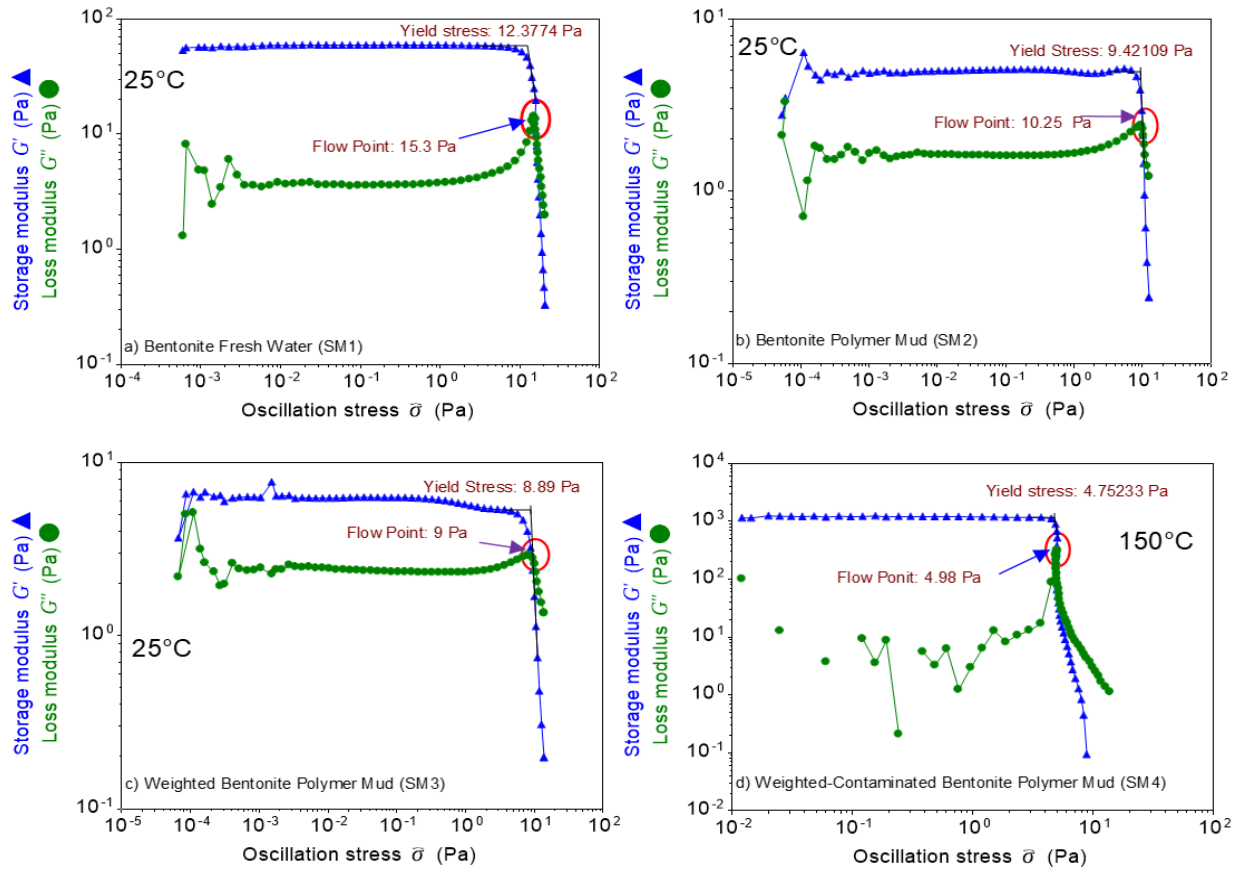


Figure 4. Oscillatory Amplitude Sweep Test Result (G' and G'' vs. τ). a) Bentonite fresh water (SM1), b) Bentonite polymer (SM2), c) Weighted bentonite polymer (SM3) at 25°C, d) Weighted contaminated bentonite polymer mud (SM4) at 150°C.

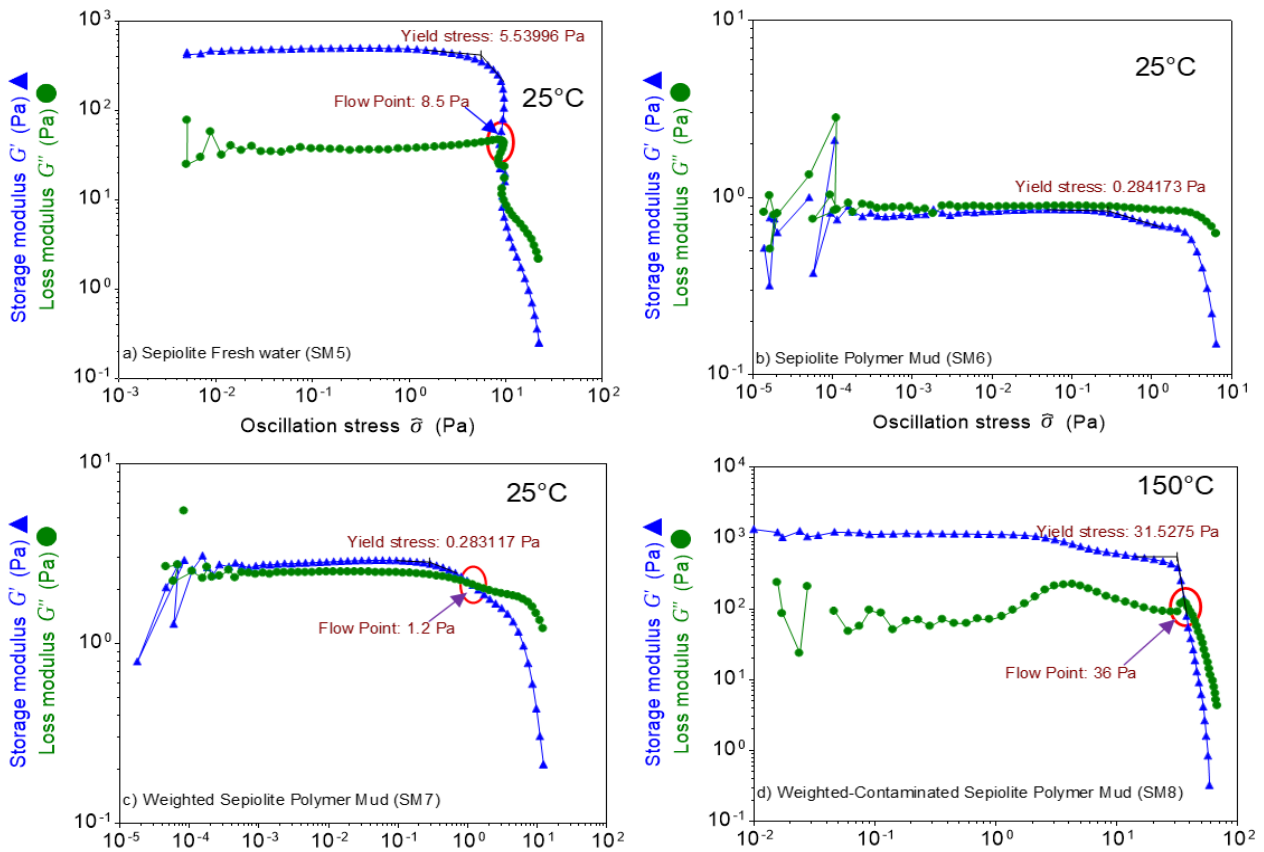


Figure 5. Oscillatory Amplitude Sweep Test Result (G' and G'' vs. τ). a) Sepiolite fresh water (SM5), b) Sepiolite polymer (SM6), c) Weighted sepiolite polymer (SM7) at 25°C, d) Weighted contaminated sepiolite polymer mud (SM8) at 150°C.

3.3. Thermal stability

Figure 6 and Figure 7 show the temperature and viscosity change over time in a part of the thermal cycle test for weighted-contaminated bentonite polymer mud (SM4) and weighted-contaminated sepiolite polymer mud (SM8), respectively. The change in both minimum and maximum viscosity values from cycle to cycle should be monitored to understand structural changes in samples. Each cycle has the maxima that is measured by rheometer and can be normalized by dividing the maximum viscosity value from the first cycle by maximum viscosity value

of each cycle. Therefore, the relative structural change Δ is then calculated as:

$$\Delta = \frac{\eta_{Max,1}}{\eta_{Max,i}} \quad \text{with } i = 1 \text{ to } 5 \quad (1)$$

where Δ is the relative structural change, $\eta_{Max,1}$ is the maximum viscosity value from the first cycle, and $\eta_{Max,i}$ is the maximum viscosity value of i^{th} cycle.

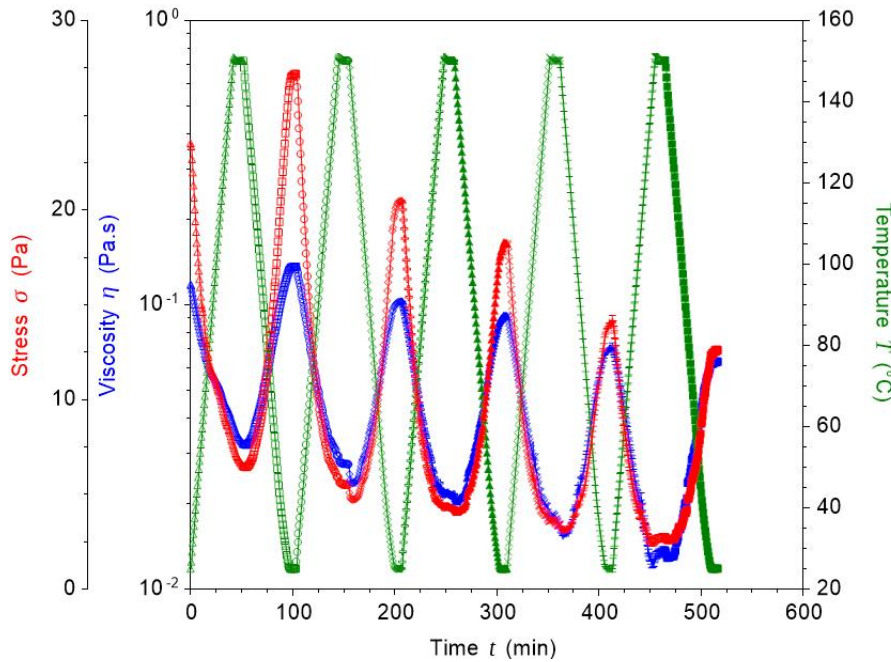


Figure 6. Thermal stability (cycle test) for weighted-contaminated bentonite polymer mud (SM4).

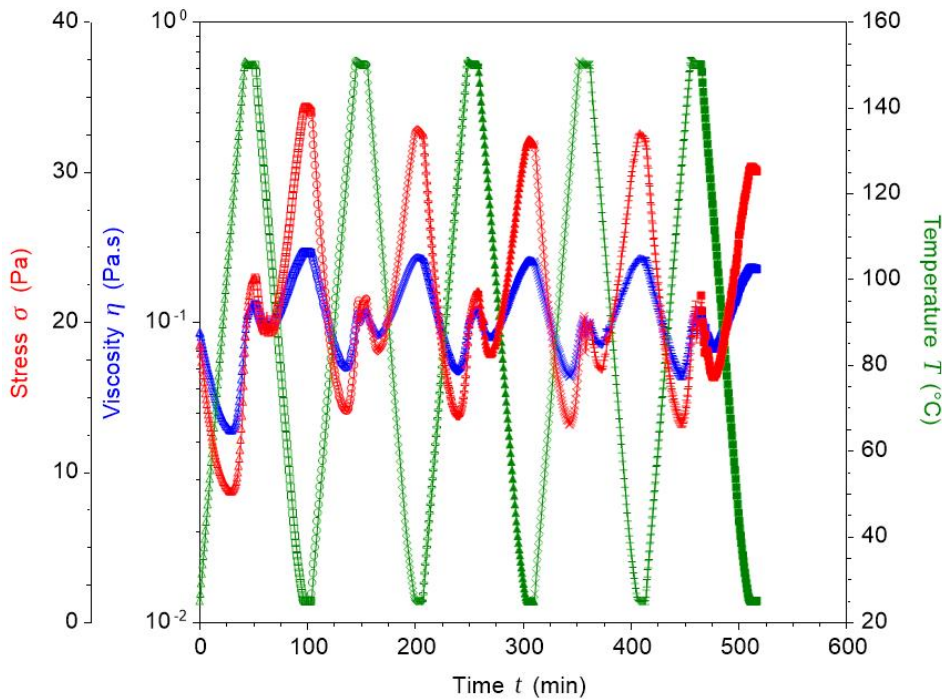


Figure 7. Thermal stability (cycle test) for weighted-contaminated sepiolite polymer mud (SM8).

Results revealed that both minimum and maximum viscosity values decreases from cycle to cycle in weighted-contaminated bentonite polymer mud (SM4). Therefore, the relative structural change values increases continuously over the duration of the measurement (Figure 6). However, as shown in Figure 7, no distinct changes is observed in both minimum and maximum viscosity values as a results of thermal cycle test for weighted-contaminated sepiolite polymer mud (SM8).

The structural change of samples is shown in Figure 8. The sample that shows a small value for the relative structural change at the end of the thermal cycles, has the smallest decrease in the

viscosity and therefore the highest thermal stability. Relative structural change in weighted-contaminated bentonite polymer mud (SM4) is almost two fold of what measured for weighted-contaminated sepiolite polymer mud (SM8) at the end of five thermal cycles. Moreover, thermal structural change in weighted-contaminated sepiolite polymer mud (SM8) is almost constant during the period of measurement (Figure 8). Therefore, results depicted in Figure 8 revealed that the weighted-contaminated sepiolite polymer mud (SM8) has significantly smaller structural change than the weighted-contaminated bentonite polymer mud (SM4). This is the robust indication for thermal stability of sepiolite mud while using at harsh drilling conditions.

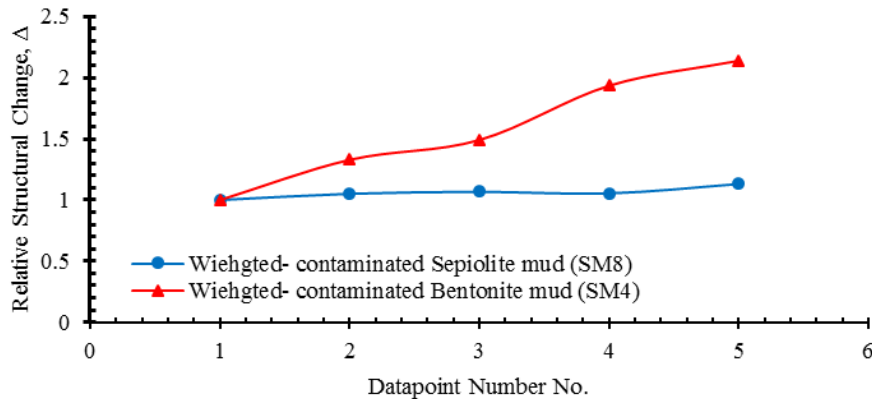


Figure 8. Relative change of the structural strength during 5 thermal cycles.

4. Conclusions and Recommendations

In this experimental study, in-situ mechanical and thermal stability of sepiolite and bentonite based muds, each in four states, were investigated using DHR-II. Following findings were revealed and confirmed;

- Mechanical parameters (yield point and gel strength) based on API recommended tests were measured and according to the results: both bentonite and sepiolite fluid system without additives provided quite high YP and GS at 25° C. Polymeric additives decreased the values of these two parameters considerably for both fluid systems. Breaking down the gel structure of bentonite polymer mud at 150° C despite of active clay intrusion, confirmed obvious failure in mechanical and thermal stability of this fluid systems. Weighted sepiolite polymer mud provided acceptable YP and GS at 150° C with active clay contamination.
- Oscillation amplitude sweep test results demonstrated that adding polymer additives to the mud systems increases the structural stability of mud system. The LVE limit was significantly reduced with increasing temperature and active clay intrusion to the mud system.
- The linearity limit value of bentonite mud is much higher than sepiolite mud at 25° C. Therefore, bentonite mud has higher structural stability in small strains. As a result, excessive amount of pump power is required to break the gel structure of bentonite mud in compare to sepiolite mud at 25° C.
- Unlike bentonite polymer mud system, sepiolite polymer mud can provide effective hole cleaning due to exceptional mechanical stability with high active clay intrusion at high temperature.

- Subjected to the thermal cycle test, weighted-contaminated sepiolite polymer mud has significantly smaller structural changes than the weighted-contaminated bentonite polymer mud.

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