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A Comparative Study on Thixotropic Behavior of Clay Based Drilling Fluids

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ABSTRACT

A series of steady shear flow and oscillatory dynamic shear tests were performed to characterize the gel structure, and thixotropy of unweighted and weighted bentonite and sepiolite drilling fluids. The effects of fluid type, temperature and density were analyzed. Results revealed that fluid type and its components have the significant influence in thixotropic behavior of mud samples. Sepiolite clay provided remarkable rate of buildup after breakdown the structure. Thixotropy is temperature dependent and while bentonite muds failed at high temperatures, sepiolite based mud yielded notable thixotropic response even at 150°C.

INTRODUCTION

Agreed by IUPAC terminology, thixotropy is defined as “the continuous decrease of viscosity with time when flow is applied to a sample that has been previously at rest and the subsequent recovery of viscosity in time when the flow is discontinued” [1]. Therefore it can be inferred that thixotropic materials are time depentent (non-newtonian) with shear thinning property [2–6].

A simple physical meaning of thixotropy was introduced by Roussel [7]. He defined the forces between particles as a potential energy well for each particle. Minimum required energy to set the particle free recognized as the depth of potential well. Applied energy should surpass this minimum requirement to get particle out of the well and flow starts. Long enough flowing (equilibrium state) will cause depth of energy well decreases to a lowest value. However, the particle will back to the well upon applied energy is reduced below the minimum value (material is at rest).

Drilling fluids are colloidal suspensions with solid particles. Inter-particle bonds (Waals attraction, electrostatic repulsion) between solid particles can be broken when fluid starts pumping to be circulated in wellbore. As a results, aggregation of suspended particles (flocs) breaks down and become smaller with increasing shear rate (pump speed). However, during tripping when circulation is stopped the particulate network will rebuild that causes flocs developing. Following the tripping process, again drilling fluid is pumped (structural network is broken again) to continue drilling. This is the similar story with thixotropy explanation by Roussel.

Thixotropy is an essential part of rheology. The thixotropic behaviors of drilling fluid could be an effective demonstration of its flow behaviors during drilling operation. The breakdown part of thixotropy can clarify the effect of its flow behavior on drilling fluid pumping and its buildup part can be a good indication of the ability of drilling fluid to leave particle in suspension and consequently hole cleaning efficiency. Therefore, deep understanding of thixotropic behaviors of drilling fluid is required to evaluate and predict flow behaviors of drilling mud in practice.

Even though numerous studies considered rheological properties of drilling fluids, there is limited information on thixotropic behaviors of drilling fluid because of the complexity of the issue and the lack of models describing it [6]. In general, thixotropy is not taken into account when dealing with drilling fluids in field applications [8], however few studies have recently considered thixotropy of drilling fluid [9–12].

This paper is an attempt to experimentally investigate thixotropic behavior of sepiolite based mud for the first time and compare it with frequently used bentonite drilling fluid. Sepiolite clay, an effective viscosifier, can be considered as the main component of drilling fluid system used for hostile drilling conditions such as high temperature, high salinity and deep well drilling (weighted mud) [13–18]. The effect of density and temperature on thixotropic behavior of both mud systems are also presented.

METHOD AND MATERIALS

Materials

This study covers examination of two clay based drilling mud systems. As a commonly used fluid system bentonite/polymer drilling mud was selected to be compared with new developed and thermally stable sepiolite drilling mud system [14]. Four states of these two fluid systems were considered to be investigated; without additive, unweighted, weighted, weighted and contaminated. A raw clay sepiolite clay sample, commercially named Turk Taciri Bej, was provided by AEM Company near Sivrihisar-Eskisehir district of Turkey. The process of preparing sepiolite clay such as crushing, grinding, sieve analysis, morphological properties and mineral characterization has been explained in a recent study [14]. Bentonite/polymer drilling fluid was also prepared in laboratory using Commercial Wyoming bentonite clay (QUIK-GEL) provided by Baroid Company. In addition, some other technical grade additives listed in Table 1 and 2, were used to improve rheological and filtration properties of formulated mud samples.

Table 1: Bentonite Based Mud Compositions.

Substance	Quantity (lbm/bbl)		
	Bentonite/Polymer Mud		
	Base SM1	Unweighted SM2	Weighted - Contaminated SM3, SM4*
Sepiolite	None	None	None
NaOH	None	0.06	0.06
Soda Ash	None	0.1	0.1
Bentonite	10	10	10
Polymer -	None	2	2
Polymer -	None	None	None
PAC-LV	None	2	2
Barite	None	None	378
*OCMA	None	None	50

Table 2: Sepiolite Based Mud Compositions.

Substance	Quantity (lbm/bbl)		
	Sepiolite Mud		
	Base SM5	Unweighted SM6	Weighted - Contaminated SM7, SM8*
Sepiolite	17.5	17.5	17.5
NaOH	None	None	None
Soda Ash	None	0.1	0.1
Bentonite	None	None	None
Polymer -	None	2	2
Polymer -	None	3	3
PAC-LV	None	None	None
Barite	None	None	371
*OCMA	None	None	50

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

Methods

Steady Shear Rheological Properties (API suggested tests):

All drilling mud samples were prepared in laboratory based on API RP-13B Standard recommendation. Unweighted and barite-weighted sepiolite and bentonite mud systems were formulated based on 350 ml of water phase (distilled water) mixing with sepiolite, bentonite clays and different amount of commercially available additives (polymers). Adding 378 lb/bbl (1077 kg/m^3) and 371 lb/bbl (1057 kg/m^3) of barite to fluid system, contributed to prepare barite-weighted bentonite/polymer and sepiolite based drilling fluid samples, respectively. Contamination of drilling fluid during drilling operation was simulated by adding 143 kg/m^3 (50 lb/bbl) of standard evaluation clay (formerly named OCMA) to the mud system.

Furthermore, fluid samples were subjected to the temperatures of 25 and 150°C for 16 hours using roller oven and then were cooled down to ambient temperature. Some rheological parameters such as apparent viscosity (AV), plastic viscosity (PV), yield point (YP), gel strength (GS), shear thinning and thixotropy indexes were then determined after rheology measurement using Fann Model 35 Couette type viscometer that is a devise typically used in the field. The measurements will be carried out at 25°C (80°F) and 49 °C (120°F) to check the temperature dependency of the test fluids.

Determination of shear thinning and thixotropy indexes

The shear thinning of drilling fluid is defined as a viscosity decrease with increasing shear rate. Strong shear thinning improves cutting transport ability and consequently hole cleaning. The slope of the viscosity-shear rate plot can be demonstrated as the tendency of shear thinning. In addition, it can be quantified using a shear thinning index (STI) which is defined based on 3-rpm and 300-rpm readings from Fann 35A (Eq-1). Therefore the large STI value is an indication of strong shear thinning fluid.

$$STI = \frac{3\ rpm\ reading}{300\ rpm\ reading} \qquad (1)$$

Thixotropy is defined as a viscosity decrease over time at a constant shear rate. This parameter can be evaluated as an indicator for barite particle suspension ability of mud system during shut-in condition. Building good gel structure, fluid system will be more structurally stable, therefore the viscosity drop over time will be more obvious. Fann 35A viscometer will be used to evaluate fluid thixotropy. A sequence of procedures repeated in the same order as follows;

1. Mixing for 5 minutes at 300 rpm to break the gel structures
2. Resting for 5 minutes
3. Shearing for 5 minutes at 600 rpm and record maximum viscosity reached initially
4. Record minimum viscosity reached after 5 minutes of shearing at 600 rpm.

This procedure is repeated at 300 rpm. The thixotropy was determined at 25°C (80°F) and 49°C (120°F). Thixotropy is defined based on maximum and minimum viscosity observed at a given shear rate (Eq-2). Contrary to the STI the smallest the TI, the more thixotropic the fluid.

$$TI = \frac{Min\ Final\ Viscosity}{Max\ Initial\ Viscosity} \qquad (2)$$

Oscillation and Thixotropy Test Using Rheometer

Thixotropic properties of drilling fluid samples were measured based on flow ramp and oscillation tests. 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. Moreover, the results of thixotropy loop and dynamic time sweep (Three Interval Thixotropy Test) tests were used to analyze the thixotropic behavior of fluid samples.

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. 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 (Ø60 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.

Measuring methods to determine the thixotropy using Rheometer

1. Thixotropy loop (Hysteresis curve) test

In quality control the thixotropic behavior is determined by evaluating the surface area between the upwards and downwards parts of a flow curve. Flow loop tests can be used to show the magnitude of structural change during the wait time between the down and up curves.

The samples were pre-sheared before conducting any rheological measurements to erase any shear history during sampling preparation and fluctuation allowed to rest for few minutes to reach equilibrium. A pre-shear was applied at a shear rate of 5 s^{-1} for 30 seconds to provide the same flow history. Stress is ramped up linearly for 100 second from the initial (zero) to the final stress, and then back down, over the same duration. The final stress value was determine using steady state flow ramp test conducted from 0.1 to 1000 1/sec for each sample. The further the up ramp and down ramp curves differ, the larger the area between the curves, the higher the thixotropy of the material.

2. Three Interval Thixotropy Test (3 ITT)

The 3 Interval Thixotropy Test (3 ITT) is more convenient method to measure thixotropic properties. This test type can be used to simulate the conditions of application processes. The test involves of the following three intervals:

1. Interval at rest with a constant, very low shear load (rest interval).
2. Interval with a constant high shear load (load interval).
3. Interval for regeneration of the structure with the same test settings as in the interval at rest (regeneration interval).

The strain and angular frequency are set in an oscillatory test. The setting values must be selected inside the linear viscoelastic range for the interval at rest and interval for regeneration in oscillatory test. The amplitude sweep is performed for determining the LVE range as stated before. For load interval the strain value was selected from nonlinear region for each sample. For this study the strain of 400 % was set in load interval as this value is quite high to destroy the structural network of all fluid sample. The same setting values must be applied in the rest and regeneration intervals.

RESULTS AND DISCUSSION

API Recommended Test Results

Rheological parameters measured using conventional viscometer (Fann 35A) were tabulated in Table 3 and 4 for bentonite and sepiolite mud systems, respectively. The values of plastic viscosity, yield point, gel strength and apparent viscosity of examined fluid systems should be evaluated to understand drilling fluid performance during static and dynamic conditions. On the other hand the impact of thixotropy on pressure drops during mud circulation can be indicated using shear thinning index and thixotropy index. The most two important rheological parameters indicating carrying capacity and suspension ability of drilling fluid are yield point and gel strength, respectively. YP decreased from 38 to 19 lb/100 ft^2, and from 40 to 18 lb/100 ft^2 for bentonite and sepiolite fluid systems, respectively, by adding polymeric additives to the base mud system at 25°C. As barite is a kind of inert solid, increasing density of fluid systems has not significant effect on the value of YP for both mud system at 25°C.

Table 3: Bentonite based drilling fluid properties.

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	Dial reading@		Dial reading@		Dial reading@		Dial reading@	
@measurement temperature	25°C	49°C	25°C	49°C	25°C	49°C	25°C	49°C
Plastic viscosity, Cp	8	9	22	18	37	28	20	16
Yeild Point, lb/100 ft^2	38	34	19	14	23	12	2	2
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.
Apparent viscosity, Cp	27	26	31.5	25	48.5	34	21	17
pH	8.1	8.1	8	8	8.6	8.6	7.9	7.9
Shear thinning index (STI)	0.61	0.37	0.12	0.09	0.08	0.08	0.09	0.11
Thixotropy index (TI)@600 rpm	0.98	0.96	1.00	1.00	0.96	0.97	1.08	0.93
Thixotropy index (TI)@300 rpm	0.98	0.98	0.98	1.00	0.97	0.98	1.00	1.00

Table 4: Sepiolite based drilling fluid properties.

Fluid system	Sepiolite fresh water		Sepiolite polymer (8.7 ppg)		Sepiolite polymer (14 ppg)		Sepiolite /polymer (14 ppg) contaminated	
Aging Temperature, °C	25		25		25		150	
Dial reading	Dial reading@		Dial reading@		Dial reading@		Dial reading@	
@measurement temperature	25°C	49°C	25°C	49°C	25°C	49°C	25°C	49°C
Plastic viscosity, Cp	6	5	20	15	36	26	34	27
Yeild Point, lb/100 ft^2	40	27	18	12	23	14	24	19
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, Cp	26	18.5	29	21	47.5	33	46	36.5
pH	7.5	7.5	8	8	8.1	8.1	8.1	8.1
Shear thinning index (STI)	0.50	0.53	0.11	0.11	0.07	0.08	0.10	0.13
Thixotropy index (TI)@600 rpm	0.97	0.90	0.98	0.95	0.97	0.93	0.98	0.99
Thixotropy index (TI)@300 rpm	0.97	0.91	0.97	0.96	0.96	0.94	0.98	1.02

Drastic decrease was observed in YP value of weighted bentonite polymer mud system (from 12 lb/100ft^2 at 25°C to 2 lb/100ft^2 at 150 °C) with increasing temperature and intrusion of active clay. On the contrary, the YP of weighted sepiolite mud system was increased (from 14 lb/100ft^2 at 25°C to 19 lb/100ft^2 at 150°C) indicating a noticeable thermal stability for this mud system.

Formation of proper gel strength that is not progressively increased is desirable for drilling fluid system[8]. Temperature and reactive solid contamination have an essential influence on gel strength and consequently suspension ability of drilling fluid. While sepiolite mud (SM8) provides 12 lb/100ft^2 of gel strength, a relatively low 10 minute gel strength as 2 lb/100ft^2 was measured for bentonite mud system (SM4) when subjected to high temperature and reactive solid contamination (Table 3 and 4). Weak gel strength, a broken structural network were clearly inferred for bentonite drilling fluid system at high temperature during drilling clay rich formations.

Bentonite based mud (without additives, SM1) demonstrated more shear thinning properties (STI = 0.61) compare to sepiolite based mud, SM5 (STI = 0.50) at 25°C. But on the contrary, with increasing temperature to 49°C sepiolite base mud, SM5 (STI = 0.53) exhibited stronger shear thinning properties than bentonite base mud, SM1 (STI = 0.37). However, sepiolite based mud (without additives, SM5) is slightly more thixotropic (lower TI, 0.90) than bentonite based mud, SM1 (higher TI, 0.96). Upon adding polymers to the base mud system, shear thinning index was extremely decreased at the rates of around 80 % (from 0.61 to 0.12) and 78 % (from 0.50 to 0.11) for unweighted bentonite (SM2) and sepiolite polymer

(SM6) mud systems, respectively. The thixotropy index was increased in both fluid systems but sepiolite polymer mud system was still more thixotropic (lower TI). Adding weighted agent to fluids also causes some impacts on shear thinning and thixotropy. The extra solids (barite) made the both fluid systems less shear thinning (lower STI) but more thixotropic (lower TI). Both fluid systems became more shear thinning (higher STI) and less thixotropic (higher TI) with increasing temperature and active clay contamination. However, higher thixotropy (lower TI) was observed in the case of weighted contaminated sepiolite mud system (SM8) than those of weighted contaminated bentonite (SM4) at 150°C.

API standard test procedure does not represent the actual rheological properties as it cannot simulate hostile wellbore conditions. Therefore, it might lead to report fallacious results especially at high temperature along with contamination. A kind of sophisticated rheometer should be used to provide representative and in-situ properties of drilling fluid system.

Thixotropy loop (Hysteresis curve) test results

Table 5 lists the results of the data evaluation elements of the Thixotropy loop test as shown in Figure 1 and 2 for bentonite and sepiolite based mud samples, respectively. This includes the hysteresis area between the ramp up and ramp down curve of the shear stress and the yield stress derived from a curve fit according to the Herschel Bulkley model.

As seen from Table 5 and Figure 1 and 2 the amount of thixotropy for bentonite based mud (SM1) decreases at the rate of 38% by adding additives, SM2 (polymers). An increase of 30% was observed by increasing density of mud system from 8.7 to 14 ppg (SM3). The effect of temperature and active solid intrusion were so impressive and cause to 58% of decreasing in thixotropy value (SM4).

Table 5. Thixotropy loop (Hysteresis curve) test data.

Mud code	Hysteresis area (Pa/s)	Yield Stress (Pa)
SM1	5241.4	-17.8
SM2	3228.4	4.76
SM3	4587.3	4.47
SM4	1934.9	0.28
SM5	6374.2	9.13
SM6	1649.1	1.26
SM7	3154.9	2.51
SM8	10924.8	-35924

In the case of sepiolite mud the rate of decrease in thixotropy value (74%) is much more than those of bentonite mud sample by adding additives. Adding weighting agent to the sepiolite/polymer mud system (SM7) has more effect on increasing thixotropy (91%). Unlike bentonite mud sample, sepiolite mud sample exhibited a remarkable increase in the hysteresis area (10924.8 Pa/s) and corresponded thixotropy while circulating mud in wellbore at high temperature along with active solid intrusion (SM8).

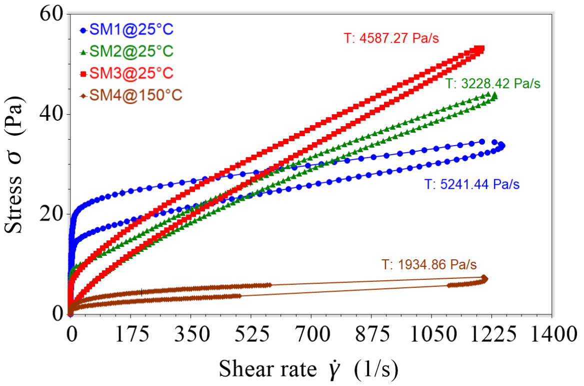

Figure 1. Thixotropy loop (Hysteresis curve) test results for bentonite mud samples.

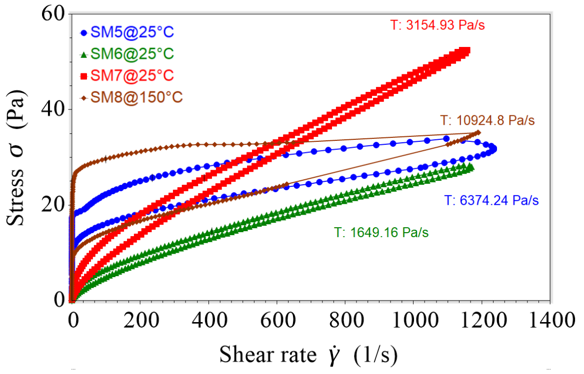

Figure 2. Thixotropy loop (Hysteresis curve) test results for sepiolite mud samples.

Thixotropy loop (Hysteresis curve) test was performed at four different temperatures (25, 50, 100, 150°C) for weighted (14 ppg) and contaminated mud samples (SM4, SM8) to investigate the effect of temperature in detail. Figure 6 shows the Thixotropy loop (Hysteresis curve) test results at four above mentioned temperatures. Similar trend in thixotropy change was observed up to 100°C for both weighted mud samples. The value of thixotropy was firstly decreased up to 50°C and then it was increased while temperature keeps rising up to 100°C in both systems.

The difference in thixotropy of both mud systems was came into the picture above 100°C. The negative effect of temperature on the values of thixotropy was observed in bentonite mud system at 150°C. Hysteresis area of thixotropy loop was decreased dramatically from 3260 Pa/s at 100°C (SM3) to 1934.8 Pa/s at 150°C, SM4 (almost at the rate of 40%). On the other hand, significant increase in the amount of thixotropy (10924 Pa/s) at high temperature is a strong indicator of being effective carrier fluid for weighted sepiolite mud system (SM8) in hole cleaning.

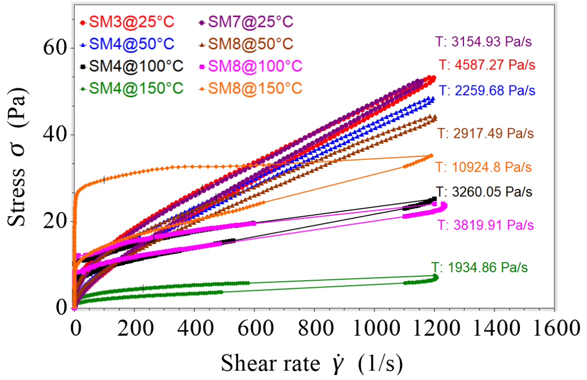

Figure 3. Thixotropy loop (Hysteresis curve) test results at different temperatures.

Three Interval Thixotropy test (3 ITT) results

Figure 4 and 5 show the results of 3-interval thixotropy test (3ITT) for bentonite and sepiolite mud systems, respectively. In addition, the data analysis of the structure recovery after 10 sec and time for 70% recovery ratio were tabulated in Table 6. The results indicated that sepiolite fresh water mud (without additives, SM5) provides higher storage modulus than bentonite fresh water mud (SM1) in the first resting interval. It is a clear indicator of stronger gel strength, a more stable network and corresponding elastic character of sepiolite fresh water mud (SM5). Sepiolite mud samples with additives (SM6) is more fluid and easier to pump wellbore as provided lower storage modulus in second interval.

Weighted-contaminated sepiolite mud sample (SM8) provided more elasticity than those of bentonite mud sample (SM4) at 150°C (load interval). This is a clear evidence that sepiolite mud can tolerate active solid intrusion at high temperature and remains more stable than bentonite mud at the same conditions.

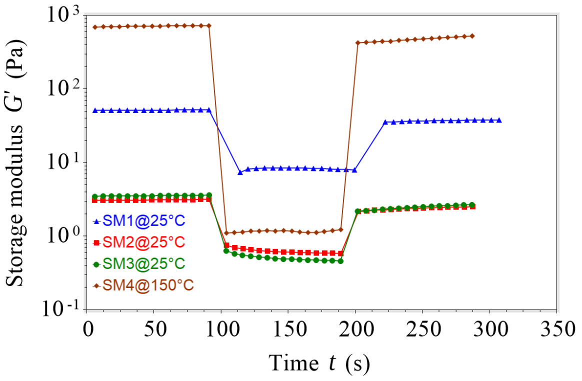

Figure 4. Three-interval thixotropy test results for bentonite mud samples.

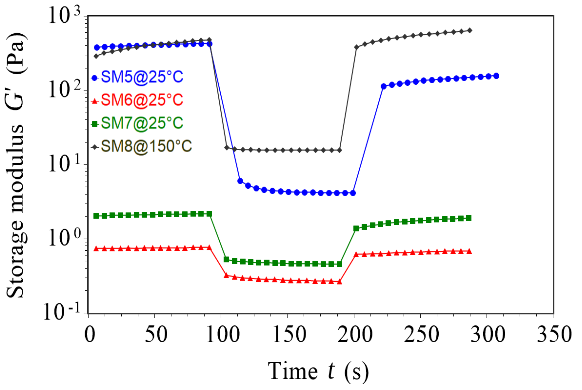

Figure 5. Three-interval thixotropy test results for sepiolite mud samples.

The amount of gel strength in drilling fluid is an important factor that is an indication of how fast the fluid structure recovers and significantly reduces the settling of solid particles. It is measured as the amount of shear stress at low shear rate after the mud stays in static condition for 10 seconds and 10 minutes in API standards. Therefore this study considers the structure recovery after 10 seconds for all mud samples. The structure of sepiolite fresh water mud (SM5) recovers 50% which is lower than structure recovery rate in bentonite fresh water (SM1) mud (68%) after 10 second. However, adding additives has significant effect on structure recovery in sepiolite mud system. 81% in structure recovery after 10 second was observed in sepiolite mud system after adding additives (SM6) that is higher than bentonite mud, SM2 (69%). Adding weighted agent causes to decrease in structure recovery after 10 seconds for both mud systems. While 80% recovery in structure after 10 seconds was observed for weighted contaminated sepiolite mud (SM8), the structure of weighted contaminated bentonite mud (SM4) recovered only at the rate of 58% at 150°C. Time for 70% recovery ratio in sepiolite mud samples is much less than bentonite mud samples (Table 6).

Table 6. The structure recovery analysis data

Mud code	**Analysis**	
	Structure recovery after 10 sec	**Time for 70% recovery**
SM1	68%	41 sec
SM2	69%	19 sec
SM3	61%	61 sec
SM4	58%	79 sec
SM5	50%	not reach
SM6	81%	<10 sec
SM7	63%	25 sec
SM8	80%	<10 sec

79 seconds are required to recover 70 % of the structural network for weighted bentonite mud (SM4) circulated in wellbore at 150°C when it subjected to active solid intrusion. At the same wellbore conditions weighted sepiolite mud (SM8) is able to recover 70% of its structural bonds in less than 10 seconds. Therefore, it is inferred that sepiolite mud can provide a stable gel structure and quickly recover it after pumping at hostile drilling conditions such as high temperature and active solid contamination.

CONCLUSION

The thixotropy is associated with the rebuilding of network structure over time and is an important parameter for the drilling fluid to avoid the settling of cuttings in static condition. Three approaches were considered to evaluate thixotropic behavior of two clay base drilling fluids. The results of this comparative study revealed that sepiolite mud provides an exceptional thixotropic characteristics even at high temperature with active solid contamination. Therefore, at hostile drilling conditions, sepiolite based mud can be a practical alternative for frequently used bentonite drilling fluid system to provide effective hole cleaning.

ABBREVIATIONS:

Mud code	Mud Type
SM1:	Bentonite fresh water
SM2:	Bentonite polymer, 8.6 ppg
SM3:	Weighted Bentonite polymer, 14 ppg
SM4:	Weighted - Contaminated Bentonite polymer, 14.5 ppg
SM5:	Sepiolite fresh water
SM6:	Sepiolite polymer, 8.6 ppg
SM7:	Weighted Sepiolite polymer, 14 ppg
SM8:	Weighted - Contaminated Sepiolite polymer, 14.5 ppg

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