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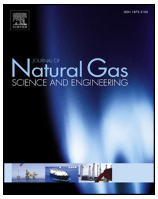

An improved test protocol for high temperature carrying capacity of drilling fluids exemplified on a sepiolite mud

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ABSTRACT

Drilling fluids with a high carrying capacity can improve the rate of penetration only if they maintain their suspending properties under downhole conditions. Testing, however, is generally performed by hot rolling fluids at the expected temperatures and measuring rheology after cooling which can produce misleading results. Therefore, a new test protocol was developed using a sepiolite water-based fluid and a xanthan gum-based reference system designed for drilling high temperature wells. First the static carrying capacity of the fluids was evaluated by suspending simulated cuttings and aging them statically before evaluating the samples visually. With conventional testing, both fluids seemed to retain their suspending capability after aging equally, however, only the sepiolite fluid maintained its excellent suspending property up to 150 °C (302 °F) in the static carrying capacity tests. This was confirmed by HTHP (150 °C/302 °F) viscosity measurements thus showing the importance of testing under actual conditions.

1. Introduction

Drilling fluids must fulfill a multitude of requirements thereby necessitating varying fluid properties. While the main function of drilling fluids is to remove cuttings from the borehole, they are also responsible for cooling and lubricating the drill bit and forming a filter cake on the wall of the wellbore to prevent excessive fluid loss (Finke, 2012; Bloys et al., 1994). When drilling in impermeable rock formations such as granite however, fluid loss control is generally not required. Managing borehole pressure is also one of the most important requirements for the drilling mud. The borehole pressure should always be maintained in the pressure window between pore pressure and fracture pressure (Caenn et al., 2011).

To achieve the desired rheological properties, thickening agents are required which allow the fluid to remain pumpable, but also provide enough viscosity to suspend cuttings, even at high temperature (Caenn and Chillingar, 1996; Wise et al., 2010). Xanthan gum is a commonly used drilling fluid additive which provides high low shear viscosity (= high yield point) and thus ensures excellent hole cleaning and carrying capacity for drill solids. It is routinely used as viscosifier when drilling geothermal wells in continental Europe. The water-soluble microbial biopolymer is prepared via fermentation using the bacterial strain of *Xanthomonas campestris*. The polysaccharide possesses a β-D-glucose backbone to which every second glucose unit, a trimer of mannose, glucuronic acid and mannose is linked (Fig. 1). The mannose unit closest to the backbone contains an anionic acetate group while the furthest mannose unit possesses an anionic pyruvate group, which explains the excellent solubility of xanthan gum in water.

The viscosifying mechanism of xanthan gum is based on the formation of a large network due to the entanglement of the individual hydrocolloid chains when present in sufficient concentration. As these chains are only weakly bound to one another, they flow easily when stress is applied (Morris, 2019). Loss of viscosity after aging at high temperatures is caused by radical degradation of the polymer. Random hydrolysis of the β 1 → 4 linkage of the glucose backbone results in a strong molecular weight decrease and therefore the viscosity is reduced. The breakdown of the backbone is accompanied by the removal of the pyruvate and acetate groups, which are most susceptible to hydrolysis (Lambert and Rinaudo, 1985). As the breakdown of the backbone is due to radical degradation, adding antioxidants such as sodium metabisulfite can increase the thermal stability of xanthan gum.

Sepiolite is a natural magnesium silicate clay belonging to the group of phyllosilicates. It is represented by the general chemical formula $Si_{12}Mg_8O_{30}(OH)_6(OH_2)_4{\cdot}8H_2O$ and forms needle like particles. Its crystal structure is shown in Fig. 2. When suspended in water, the non-swelling needles can aggregate and associate to form a random network which is responsible for the viscosifying effect. This effect is induced either through low pH values or at alkaline pH through the addition of

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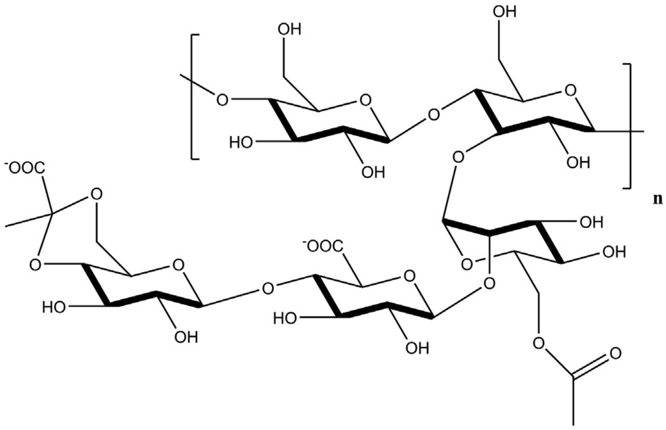


Fig. 1. Chemical structure of xanthan gum (Hamman, 2010).

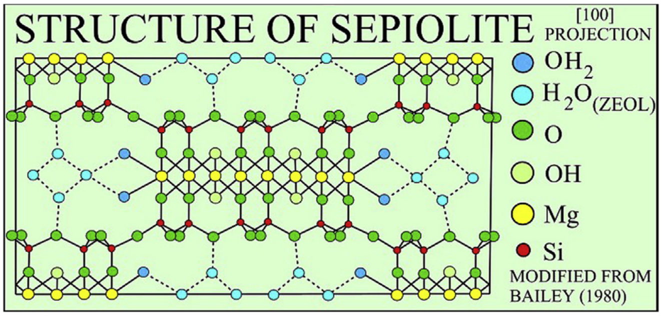


Fig. 2. Crystal structure of sepiolite (Poppe et al., 2001).

electrolytes which reduce the negative charge of the silanol surface groups allowing the individual particles to aggregate along their surfaces, thereby increasing the viscosity of the suspension. At high temperatures (260 °C), sepiolite undergoes a hydrothermal reaction resulting in the formation of stevensite, a smectite possessing a flaky (lamellar) morphology similar to montmorillonite or hectorite. As shown in Fig. 2, the individual sepiolite ribbons are connected to one another only along the Si–O–Si bonds at their corners. At elevated temperature, these ribbons can glide (c/2 glide mechanism) into a more stable configuration as additional bonds are established within the network resulting in a smectite structure. The reaction react is highly dependent on the temperature as well as the type and concentration of electrolytes (Güven, 1979).

The ability of a drilling fluid to remove cuttings from the borehole strongly impacts the rate of penetration (ROP) (Robinson and Morgan, 2004; Becker et al., 1991). When the carrying capacity of the fluid is too low, the cuttings will settle at the bottom and the drill bit then will waste considerable energy in grinding them. However, when the suspending property of the fluid is high, then the cuttings are removed more effectively from the borehole. As less energy is spent on grinding the drill solids, ROP is enhanced. Therefore, ideally the low shear rate viscosity and yield point of a drilling fluid should be high enough to suspend the cuttings once circulation is halted, while at the same time plastic viscosity should be low to avoid excessive pump pressures. However, many fluids show strong thermal thinning, i.e. they lose their suspending property downhole as the temperature increases. This leads to strongly reduced ROP as the thinned fluid exhibiting low viscosity values at low shear rates fails to properly remove the cuttings from the bottom of the well.

In order to develop drilling fluids which still sufficiently retain their hole cleaning property downhole, it is essential that laboratory testing can accurately simulate these conditions. However, conventional drilling fluid testing is generally performed by hot rolling the fluid at the expected temperatures, cooling it to near ambient and subsequently measuring rheology. The IADC drilling manual recommends viscosity testing and reporting at either 50 °C (120 °F) for standard wells or 66 °C (150 °F) for high temperature wells (IADC Drilling Manual, 2014). The rheological values obtained according to this method are taken as evidence for the behavior of the fluid at exposure to peak temperature. However, this test may produce grossly misleading results, as effects such as thermal thinning are not accounted for, because fluid viscosity might have dropped even to zero at peak temperature, but reconstituted while cooling down. Unfortunately, it is still common practice to test drilling fluid performance according to this method or even without aging in spite of its obvious shortcomings (Khalil and Jan 2012; Yan et al., 2013; Hamed and Belhadri, 2009; Song et al., 2016; Temraz and Hassanien, 2016).

So far, only few authors have realized this gap and expressed the need for an improved test method as well as conducting tests with high temperature rheometers (Zamora and Growcock, 2010; Shah et al., 2010; Amani et al., 2012; Santoyo et al., 2001). However, as these rheometers are very costly, there is a need for a simpler yet realistic and meaningful test method which allows for accurate evaluation of the suspending properties of fluids under actual downhole condition, as this property directly impacts hole cleaning and thus ROP.

The aim of this work is to highlight the importance of testing the rheology of drilling fluids under conditions which are representative of actual borehole situations, thus allowing a more realistic assessment of fluid performance. This is especially important at high temperatures, but also at low temperatures such as in natural gas hydrates as lower temperatures can too strongly increase the viscosity of the fluid. It is also important for directional drilling as well deviation makes hole cleaning more difficult as the cuttings tend to collect at the bottom, especially in 90° horizontal wells (Jenn Yue et al., 2019). To probe its hole-cleaning property, newly designed static carrying capacity tests were carried out to evaluate the ability of the fluid to suspend cuttings under static conditions. Moreover, the performance of a sepiolite fluid was compared with that of a xanthan gum-based fluid. The sepiolite clay system was developed within the framework of an EU funded project (ThermoDrill) which aimed at lowering drilling cost on geothermal wells in crystalline rock such as granite through a combination of a new jetting tool on the drill bit and a drilling fluid possessing optimized rheological properties. Rheology of the fluid was first measured following the conventional test protocol using both hot roll and static aging in a roller oven up to 150 °C (302 °F) and subsequent cooling to ambient. For comparison, static carrying capacity was tested at the same temperatures using suspended quartz particles as indicator for the carrying capacity under actual borehole conditions. To confirm the validity of the new test method, rheology was also captured on an HTHP rotational viscometer and the results were compared with those from the novel and simple static aging test using the quartz particles.

2. Experimental section

2.1. Materials

The sepiolite sample (Berkbent Marine) was provided by Tolsa SA (Madrid, Spain). Xanthan gum (Satiaxane® CX 90 T) was received from Cargill France SAS (Saint-Germain-en-Laye, France). The defoamer Tego® Antifoam MR 2132 was obtained from Evonik Nutrition & Care GmbH (Essen, Germany). Potassium chloride and potassium carbonate (both technical grade) were provided by Sirius - ES Handels GmbH (Wels, Austria). Anhydrous sodium carbonate (99.5%) was purchased from Bernd Kraft (Duisburg, Germany) while sodium metabisulphite (98%) came from Merck KGaA (Darmstadt, Germany). 1 – 2 mm sized quartz particles were attained by sieving CEN-Normsand EN 196-1 (Normensand GmbH, Münster, Germany) with 1 and 2 mm sized sieves.

2.2. Characterization of viscosifiers

TEM imaging of the sepiolite was conducted on a JEM 2010 microscope (JEOL, Eching, Germany) equipped with a LaB_6 cathode. Sepiolite samples were prepared by placing a drop of diluted aqueous sepiolite suspension on a carbon-coated copper grid.

The oxide composition of the sepiolite clay was determined by X-ray fluorescence (XRF) using an Axios instrument (PANalytical, Philips, Eindhoven, Netherlands).

Zeta potential of a 2 wt% sepiolite dispersion in DI water containing various KCl concentrations was calculated from the colloidal vibration current (CVI) determined via the electroacoustic method using a Model DT-1200, electroacustic spectrometer (*Dispersion Technology Inc.*, Bedford Hills, New York, U.S.A.) at room temperature. First, the ion background of the sepiolite was determined and then subtracted from the measured value to obtain the actual zeta potential.

Size exclusion chromatography (Alliance 2695 from Waters, Eschborn, Germany) equipped with RI detector 2414 (Waters) and an 18 angle dynamic light scattering detector (Dawn EOS from Wyatt Technologies, Santa Barbara/CA, USA) was used to separate the xanthan gum on a precolumn and two Aquagel-OH 60 columns (Polymer Laboratories, distributed by Varian, Darmstadt, Germany). Eluent was 0.2 M aqueous $NaNO_3$ solution (adjusted to pH = 9.0 with NaOH) at a flow rate of 1.0 mL/min. The value of dn/dc used to calculate M_w and M_n was 0.143 (value for guar gum) (Jumel et al., 1996).

The specific anionic charge amounts of xanthan gum was measured in DI water and in 1 wt% Na_2CO_3 (pH 11.2) at room temperature using a PCD 03 pH apparatus (Mütek Analytic, Herrsching, Germany). Charge titration was carried out with a 0.001 N solution of poly(diallyl-dimethyl-ammoniumchloride) laboratory grade (BTG Mütek GmbH, Herrsching, Germany) as cationic polyelectrolyte.

2.3. Preparation of drilling fluids

The sepiolite drilling fluid was prepared by mixing the sepiolite with tap water at 4,000 rpm for 5 min in a blade-type laboratory blender obtained from Waring Products (Torrington, CT, USA). Salt (KCl or K_2CO_3) was then added, and the drilling fluid was mixed for another 2 min at 4,000 rpm. For the KCl fluid, the pH value was adjusted to ~10.5 using Na_2CO_3. The recipe used for the sepiolite fluid is shown in Table 1.

The xanthan gum based drilling fluid was prepared by dissolving the sample (Satiaxane® CX 90 T) at 400 rpm using a propeller type mixer (EUROSTAR power control visc stirrer, IKA Werke GmbH & Co. KG, Staufen, Germany) for 1 h in tap water. Salt (KCl or K_2CO_3) was then added while mixing for 1 h at 400 rpm. Sodium metabisulfite was used as an oxygen scavenger to decrease the rate of degradation of xanthan gum at elevated temperatures and the pH value was adjusted to ~10.5 using Na_2CO_3. A defoamer (Tego® Antifoam MR 2132) was employed to reduce air entrainment. The recipe used for the xanthan gum fluid is shown in Table 2.

2.4. Conventional drilling fluid test

Conventional drilling fluid testing was carried out according to the API norm RP 13B-1, *Recommended Practice for Field Testing Water-based Drilling Fluids* (API RP 13B-1 2004). Here, the fluids were thermally conditioned for 16 h in a roller oven (OFI Testing Equipment, Houston, TX). 400 mL of the drilling fluid were poured into a 500 mL Teflon liner which was transferred into a stainless steel ageing cell and sealed. A N_2 pressure of 35 bar was applied to the cell. The sample was then heated up to the desired temperature (27, 80, 100, 120, or 150 °C) while rotating at 25 rpm in the roller oven and left rotating for 16 h. Thereafter, the cells were removed from the oven, cooled to ambient in a water bath and remixed for 5 min at 400 rpm using a propeller type mixer before the fluid properties (rheology, pH value and density) were determined.

Table 1
Sepiolite drilling fluid recipe.

Component	Amount (g)
Tap water	500
Sepiolite	7.5 or 10
KCl or K_2CO_3	88 or 60.5
Na_2CO_3	3.4 or 0

Table 2
Xanthan gum drilling fluid recipe.

Component	Amount (g)
Tap water	500
Xanthan gum	3 or 5
KCl or K_2CO_3	88 or 60.5
Na_2CO_3	5 or 0
$Na_2S_2O_5$	1
Defoamer	0.5 mL

After 16 h thermal conditioning, cooling and subsequent re-mixing, the fluid was poured into the measuring cup of a coaxial cylinder viscometer (Chandler 3500LS, from Chandler Engineering, Tulsa, Oklahoma, U.S.A.). Dial readings were taken from the Chandler viscometer every 10 s, and the rotational speeds changed in the following sequence: 600–300–200–100–60–30–20–10–6–3 rpm. The gel strengths were determined after shearing for 10 s at 600 rpm, followed by a 10 s, 10 min or 30 min rest period before shearing again at 3 rpm until a maximum value was reached. The maximum value reached presents the 10 s, 10 min or 30 min gel strength respectively. The dial readings from the Chandler 3500LS instrument (torsion spring factor F = 0.2) were converted into lbf/100 ft^2 by multiplying with 0.213.

Plastic viscosity (PV, Eq. (1)) and yield point (YP, Eq. (2)) of the fluids were calculated using the shear stress values (in lbf/100 ft^2) obtained at 300 (θ 300) and 600 (θ 600) rpm. This calculation assumes a Bingham type flow behavior.

$$PV(cp) = \theta 600 - \theta 300 \quad 1$$

$$YP(lbf/100ft^2) = \theta 300 - PV \quad 2$$

In addition, the Bingham model parameters yield point (YP_M in lbf/100 ft^2) and plastic viscosity (PV_M in cp*s) were determined via linear regression and compared with the PV and YP values calculated using Eqs. (1) and (2). The plastic viscosity values determined via linear regression were converted from lbf/100 ft^2*s to cPs. The Power Law model parameters flow consistency index (K, lbf/100 ft^2*s^n) and flow behavior index (n, dimensionless) were also calculated via linear regression as the model has shown good correlation with xanthan gum based fluids in previous work (Tabzar et al., 2015). A coefficient of determination (R^2) value of 1.00 refers to samples with a correlation > 0.995.

2.5. Novel carrying capacity test

To determine the actual carrying capacity, the drilling fluids were aged statically for 16 h at the desired temperature (27 – 150 °C). At first, the fluid was poured into 50 mL glass vials before small quartz particles (1 – 2 mm in diameter) were added to the fluid. The vials were closed and shaken to ensure that the particles or cuttings were evenly distributed throughout the fluid. The samples were then transferred into roller oven cells and a N_2 pressure of 35 bar was applied to the cells. They were then placed upright for 16 h in the oven. Thereafter, the cells were removed from the oven and cooled in a water bath. The vials were removed and photographed to document the distribution of the particles and the ability of the fluid to keep the particles suspended during aging at high temperature.

2.6. HTHP rheometry

High temperature rheology of the drilling fluids was determined using a Couette type coaxial cylinder rotational viscometer (model PVS Rheometer from Brookfield Engineering Laboratories, Middleboro/MA, USA). For each measurement, 12.5 mL of the fluid were poured into the rheometer cup. A B1 type bob was used and a N_2 pressure of 500 psi applied. The fluid was then heated under stirring at 30 rpm to 150 °C (302 °F), sheared for 20 s at 300 rpm and then the shear stress values (N/m^2) at shear rates of 600–300–200–100–60–30–20–10–6–3 rpm were taken. The system was then stirred again at 30 rpm for 60 min until the next measurement was taken to assess the long-term viscosity stability of the fluid. Every 2 h the torque value was recalibrated.

3. Results and discussion

3.1. Characterization of sepiolite and xanthan gum

Sepiolite is a natural magnesium silicate clay which forms needle like particles with a length of around 1 µm and a diameter of ~20 nm, as is shown in the TEM images (Fig. 3). Its oxide composition as well as the loss of ignition (LOI) were determined via XRF (Table 3).

The zeta potential of a 2 wt% sepiolite suspension containing increasing amounts of KCl is shown in Table 4. Sepiolite shows a negative zeta potential in DI water, which increases towards zero with increasing KCl concentration. The potassium cation can reduce the negative charge of the silanol surface groups allowing the individual particles to aggregate along their surfaces, thereby increasing the viscosity of the suspension.

The polysaccharide xanthan gum was characterized using size exclusion chromatography. Its values for weight and number average molecular weight (M_w, M_n), polydispersity index (PDI), radius of gyration ($R_{g(z)}$) and hydrodynamic radius ($R_{h(z)}$) are shown in Table 5. The specific anionic charge density in DI water was 165 C/g and 194 C/g when measured in DI water containing 1 wt% Na_2CO_3 (pH 11.2).

3.2. Fluid properties from conventional test

The drilling muds mixed with salts (12.1 wt% K_2CO_3 or 17.6 wt% KCl) to achieve a density of 1.1 g/L (see Tables 1 and 2) were hot rolled for 16 h at temperatures up to 150 °C (302 °F), and their rheology and pH was determined after cooling to ambient (~25 °C (80 °F)). This test presents the standard industry procedure to determine the thermal stability of a drilling fluid system. Retention of viscosity after aging is thus considered as an indication that the fluid possesses this viscosity also downhole.

Shown in Table 6 are the rheological properties after hot roll of a 1.5 wt% sepiolite drilling fluid weighted with 17.6 wt% KCl. It is obvious that this fluid exhibits high low shear rate viscosity (3 and 6 rpm readings) which is indicative for effective hole cleaning property. This property is also manifested in a high yield point (~20–30 lbf/100 ft^2, depending on the temperature). At the same time, the plastic viscosity is always low (3–5 cp), thus signifying a highly shear-thinning fluid with a high ratio of YP:PV of ~5–6:1 Such fluids are known to be easily pumpable while under static condition they exhibit exceptional hole cleaning and suspension property. Measurement of the 10 s, 10 and 30 min gel strengths indicated that up to 100 °C the sepiolite drilling mud does not show any gelation. An increase of the 10 and 30 min gel strength values especially was determined after aging at 150 °C.

The observed decrease of the pH value with increasing aging temperature is due to the partial dissolution over time in alkaline environment of some the Si tetrahedra resulting in the consumption of hydroxyl ions. Subsequently the pH decreases, though only a small amount of Si tetrahedra are effected and no significant structural change occurs (Martinez-Ramirez et al., 1996).

The rheology plot of the 1.5wt% sepiolite fluid weighted with KCl after aging at 27 °C and 150 °C is shown in Fig. 4 together with the fitted Power Law model while the calculated parameters for Power Law and

Table 3
Oxide composition of sepiolite sample as determined by XRF.

SiO_2 (wt. %)	MgO (wt. %)	Al_2O_3 (wt. %)	Fe_2O_3 (wt. %)	K_2O (wt. %)	Na_2O (wt. %)	CaO (wt. %)	LOI (wt. %)
54.7	21.2	2.4	0.5	0.6	0.6	0.3	19.6

Table 4
Zeta potential of a 2 wt% sepiolite suspension in DI water.

KCl dosage wt.%	Zeta potential [mV]
0%	−20,3
0.25%	−13,7
0.5%	−13,5
1%	−7,5
2%	−2,7

Table 5
Molar masses (M_w, M_n), polydispersity index (PDI), radius of gyration ($R_{g(z)}$), and hydrodynamic radius ($R_{h(z)}$) of the xanthan gum sample.

M_w (g/mol)	M_n (g/mol)	PDI (M_w/M_n)	$R_{g(z)}$ (nm)	$R_{h(z)}$ (nm)
2.1×10^6	1.9×10^6	1.1	235 ± 47	37 ± 6

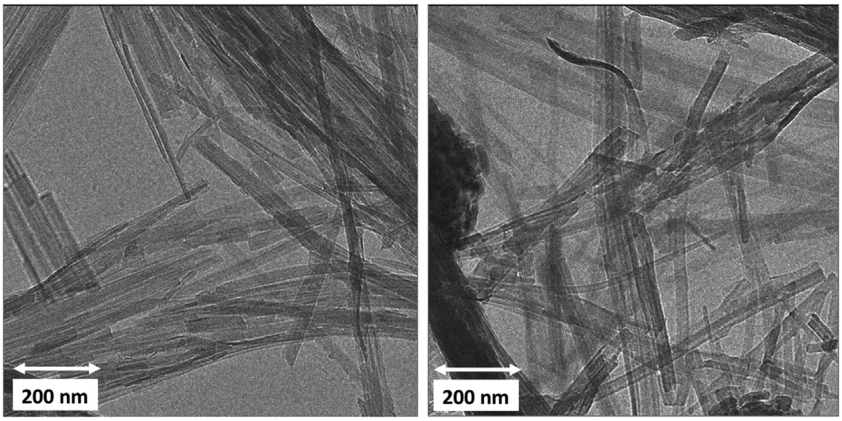


Fig. 3. Transmission electron microscopic (TEM) image of sepiolite particles.

Table 6
Rheological properties and pH of a 1.5 wt% sepiolite drilling mud holding 17.6 wt% KCl after 16 h hot roll, measured at room temperature.

Temp.	pH	Shear stress (lbf/100 ft^2)										
		600 rpm	300 rpm	200 rpm	100 rpm	6 rpm	3 rpm	10 s GS	10 min GS	30 min GS	YP	PV (cp)
27 °C (80 °F)	10.4	40	35	32	27	13	11	12	11	11	30	5
80 °C (176 °F)	10.3	34	30	27	23	11	10	9	10	10	26	4
100 °C (212 °F)	10.3	34	29	27	23	12	9	9	10	10	24	5
120 °C (248 °F)	10.1	29	26	24	21	12	10	9	11	11	23	3
150 °C (302 °F)	9.9	17	14	14	12	11	11	13	19	22	11	3

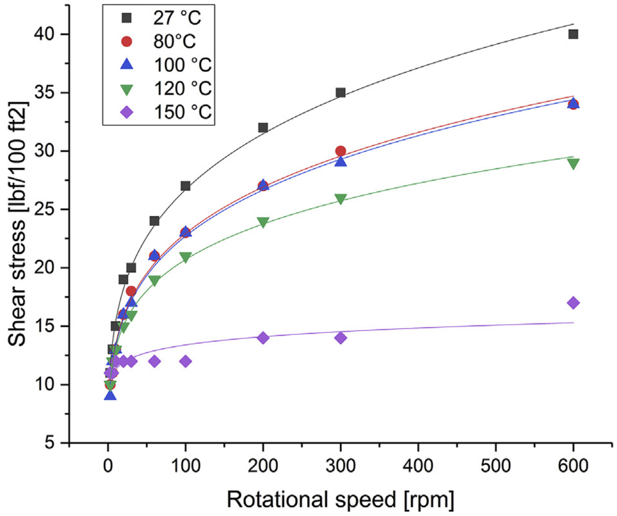


Fig. 4. Rheology and Power Law model fit of 1.5 wt% sepiolite fluids holding 17.6 wt% KCl aged at 27 – 150 °C (80 – 302 °F).

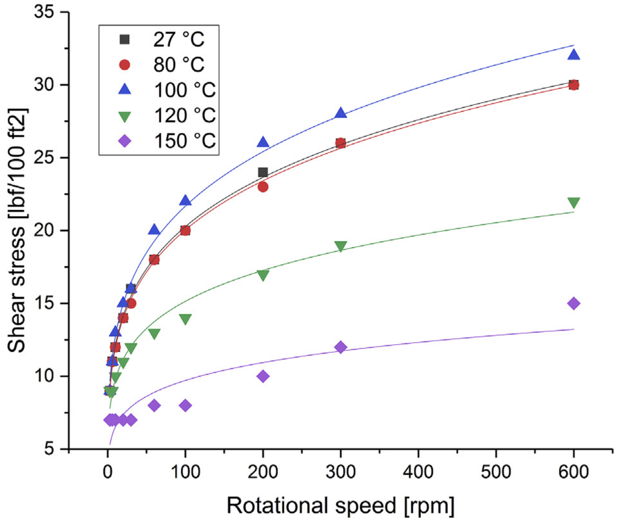


Fig. 5. Rheology and Power Law fit of 1.5 wt% sepiolite fluids holding 12.1 wt % K_2CO_3 aged at 27 – 150 °C (80 – 302 °F).

Bingham are displayed in Table 9. Before aging at high temperature, the correlation with the Bingham model was low due to the strong shear thinning property of the sepiolite fluid at high shear rates. After aging, the plastic viscosity was very low throughout the tested shear rate region, therefore the Bingham model correlates well with the measured values and to the YP calculated via Eqs. (1) and (2). Therefore, the fit for the Power Law model is initially very high, and decreases for the sample aged at 150 °C. The flow behavior index (n) decreases with increasing aging temperature, while the flow consistency index (K) remains around 8–9 lbf/100 ft^2*s^n.

Similar results were obtained when the sepiolite fluid was prepared with K_2CO_3 as weighting agent. Table 7 shows the rheological values measured at room temperature of the 1.5 wt% sepiolite weighted with 12.1 wt% K_2CO_3. Again, a highly shear-thinning fluid characterized by high YP and low PV values was obtained. However, when comparing the results from the KCl and K_2CO_3 formulated fluids it becomes obvious that as a viscosifier sepiolite performs slightly better in the KCl-based fluid (Table 6). As with the KCl weighted fluid, the K_2CO_3 fluid did not display increased gel strength values after aging at low temperature. After hot rolling at 120 °C a slight increase was observed while after aging at 150 °C a strong increase of the gel strength values was determined.

Shown in Fig. 5 is the rheology plot for the fluid weighted with 12.1% K_2CO_3 after aging. As with KCl, the fit of the Bingham model is better after aging and YP_M and YP (Table 9) are nearly identical as the fluid maintains a constant PV throughout the tested shear stress region. The same trend as with KCl is observable with the Power Law model, the fit is very high and decreases slightly for the sample aged at 150 °C. K and n are also lower after high temperature aging.

In comparison, the xanthan gum fluid weighted with 17.6 wt% KCl also showed excellent low shear rheology (3 and 6 rpm readings and yield point) measured from the fluid after cooling down to room temperature (Table 8). The data suggests that xanthan gum retained its viscosity very well up to ~120 °C (248 °F), only at 150 °C (302 °F) a minor drop is observed. Generally, the xanthan gum fluid is a non-gelling fluid, as the 10 s, 10 min and 30 min gel strength values were always similar, the gel strength values of the samples aged at low temperature increased only slightly overtime while after aging at 150 °C the values were identical. Similar results were obtained for the xanthan gum fluid weighted with 12.1 wt% K_2CO_3 to S.G. of 1.1 g/mL (values

Table 7
Rheological properties and pH of a 1.5 wt% sepiolite drilling mud holding 12.1 wt% K_2CO_3 after 16 h hot roll, measured at room temperature.

Temp.	pH	Shear stress (lbf/100 ft^2)										
		600 rpm	300 rpm	200 rpm	100 rpm	6 rpm	3 rpm	10 s GS	10 min GS	30 min GS	YP	PV (cp)
27 °C (80 °F)	11.6	30	26	24	20	11	9	7	7	8	22	4
80 °C (176 °F)	11.5	30	26	23	20	11	9	7	8	8	21	5
100 °C (212 °F)	11.5	32	28	26	22	11	9	8	9	9	24	4
120 °C (248 °F)	11.4	22	19	17	14	9	9	8	11	12	16	3
150 °C (302 °F)	11.3	15	12	10	8	8	8	16	32	34	9	3

Table 8
Rheological properties and pH of a 0.6 wt% xanthan gum drilling mud holding 17.6 wt% KCl after 16 h hot roll, measured at room temperature.

Temp.	pH	Shear stress (lbf/100 ft^2)										
		600 rpm	300 rpm	200 rpm	100 rpm	6 rpm	3 rpm	10 s GS	10 min GS	30 min GS	YP	PV (cp)
27 °C (80 °F)	10.1	43	34	30	24	14	13	13	15	16	25	9
80 °C (176 °F)	10.3	48	38	33	27	16	14	14	16	17	28	10
100 °C (212 °F)	10.1	43	34	30	25	15	14	13	16	17	25	9
120 °C (248 °F)	10.1	35	28	25	21	13	12	12	15	16	21	7
150 °C (302 °F)	9.6	28	23	21	18	10	9	10	10	10	18	5

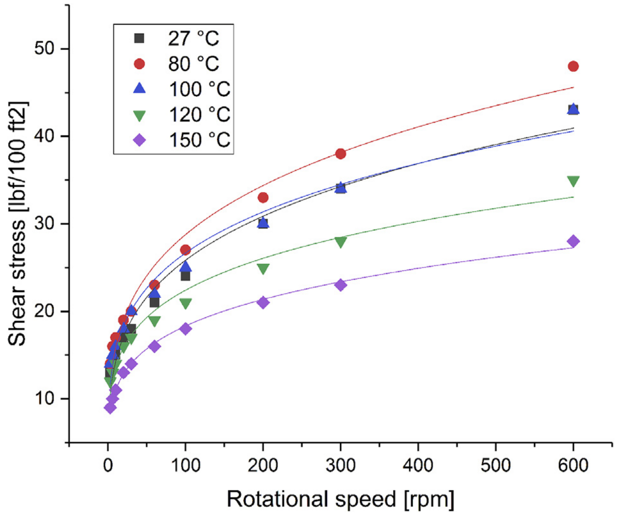


Fig. 6. Rheology and Power Law model fit of 0.6 wt% xanthan gum fluids holding 17.6 wt% KCl aged at 27 – 150 °C (80 – 302 °F).

not shown here). The decrease of the pH value observed for the xanthan gum drilling fluid is the result of increasing consumption of hydroxide ions during the hydrolysis of the polysaccharide with increasing aging temperature.

The rheological plot along with the fitted Power Law model is shown in Fig. 6 for the 0.6 wt % xanthan gum fluid weighted with 17.6 wt% KCl. Again, the correlation for the Power Law model was good and n decreased with increasing aging temperature. No clear trend wasobservable for K though it appears to decrease after aging at 150 °C. For the Bingham model, unlike for the sepiolite fluid, the rheology plot does not correlate better after aging at high temperature. The correlation was actually better before aging. As with the YP value, the YP$_M$ value for the xanthan gum fluid aged at 150 °C was higher than for the compared sepiolite fluids (Table 9).

The fluids aged at 27 and 150 °C were fitted using the Bingham and Power Law models. The correlation of the Bingham model for the sepiolite fluid aged at 27 °C was low due to its strong shear thinning character at higher shear rates. However, the fit was very good for the fluid aged at high temperature as the plastic viscosity remained low throughout the tested shear rate regime. It appears that the sepiolite fluid transitions from a Herschel Bulkley fluid to a Bingham fluid with a high yield point after aging at 150 °C (Table 9). This was confirmed due to the high correlation of the sepiolite fluid with the Power Law model. Generally, the correlation of all fluid was better with the Power Law model than with the Bingham model.

The results from the conventional hot roll test suggest that only 0.6 wt% of xanthan gum can provide similar rheological properties (i.e. carrying capacity and pumpability) than a fluid containing 1.5 wt% of sepiolite (Table 6–8). Furthermore, it suggests that both fluids exhibit comparable temperature stability with respect to their rheology. This is however contradicted by actual field experience from wells using xanthan gum where frequent replenishments of the viscosifying biopolymer are known to be necessary. Thus, some doubts arise as to whether this standard industry test represents the thermal stability of a drilling fluid accurately. To probe further into this subject, a new test protocol was developed to accurately present the static carrying capacity of a drilling fluid under actual high temperature conditions.

3.3. Results from new test method

To visualize the actual suspending properties of statically aged drilling fluids at high temperature, small quartz particles (Ø 1 – 2 mm) were added to the fluids, and the samples were aged statically for 16 h at 27 – 150 °C (80 °F – 302 °F). The concept was that if the fluid retained high values of low shear (= high yield point) during aging, then the particles would not have settled at the bottom when the samples were removed from the oven. However, if viscosity was lost during the aging, then the quartz particles would have precipitated at the bottom.

Fig. 7 displays the samples as obtained for the 1.5 wt % sepiolite containing 17.6 wt% KCl or 12.1 wt% K_2CO_3 after the static aging test. Both fluids showed excellent carrying capacity up to at least 150 °C (302 °F). All quartz particles were kept in suspension during the 16 h static aging period, even at 150 °C. Hence, the results obtained from the conventional mud test were confirmed also by this modified, more sophisticated method.

Next the fluid holding 0.6 wt% xanthan gum and 12.1 wt% K_2CO_3 was looked at. Here, a completely different result than in the conventional fluid test was obtained (Fig. 8). As is presented in Fig. 8, even at 27 °C (80 °F) the fluid exhibits limited carrying capacity as all quartz particles have settled at the bottom. Moreover, starting at ∼100 °C (210 °F), severe decomposition of the biopolymer is evidenced by the development of a more intense color of the fluid. Apparently, the xanthan gum fluid loses its carrying capacity rather quickly during aging (= severe fluid thinning), but at ambient rapidly reconstitutes to a fluid with a high yield point as was shown in the conventional test (Table 8). This experiment clearly demonstrates the limited validity of the conventional test. It also highlights the difference in behavior between an inorganic and a biopolymer-based viscosifier.

To test whether the poor performance of xanthan gum was the result of an insufficient dosage, another test series was carried out in which the xanthan gum dosage was raised from 0.6 wt% to 1.0 wt% (Fig. 8). At this higher dosage, the suspending properties were maintained up to 50 °C (122 °F), but also here were completely lost at 80 °C and above. Hence, these tests reveal that by using higher xanthan gum dosages and making frequent replenishments to the drilling fluid, a xanthan gum mud can hold up to temperatures as high as ∼100 °C (212 °F), but it will not tolerate such temperatures for longer exposure periods, and the fluid will not be stable, as is clearly the case for the sepiolite fluid.

The results clearly highlight a major discrepancy between the two tests. First, they demonstrate that during aging, thermal thinning of the fluid can occur which makes cuttings to settle at the bottom. Obviously, this is very detrimental for the drilling process as it can lead to a lower ROP or even stuck pipe. Second, our experiments demonstrate that measuring rheology after hot roll at ambient can produce a very misleading impression of the fluid's capability to suspend cuttings. Here,

Table 9
Comparison of the Bingham and Power Law model parameters for the different drilling fluids aged at 27 – 150 °C (80 – 302 °F) as calculated by linear regression and determined via Eqs. (1) and (2) respectively.

System	Temp.	Eqs. (1) and (2)		Bingham model			Power Law		
		YP (lbf/100 ft^2)	PV (cP)	YP$_M$ (lbf/100 ft^2)	PV$_M$ (cP*s)	R^2	K (lbf/100 ft^{2}*s^{n})	n	R^2
1.5 wt% sepiolite + 17.6% KCl	27 °C	30	5	17.6	21.7	0.79	8.9	0.24	1.00
	80 °C	26	4	15.3	18.2	0.78	7.9	0.23	1.00
	100 °C	24	5	15.1	18.2	0.79	7.8	0.23	1.00
	120 °C	23	3	14.6	13.9	0.78	8.3	0.20	1.00
	150 °C	11	3	11.5	4.5	0.95	9.5	0.07	0.74
1.5 wt% sepiolite + 12.1 wt% K_2CO_3	27 °C	22	4	13.7	15.6	0.81	7.3	0.22	1.00
	80 °C	21	5	13.4	15.8	0.83	7.1	0.22	1.00
	100 °C	24	4	14.5	17.2	0.78	7.5	0.23	1.00
	120 °C	16	3	10.7	10.5	0.89	6.3	0.19	0.98
	150 °C	9	3	6.9	6.8	0.98	4.4	0.17	0.81
0.6 wt% xanthan gum + 17.6% KCl	27 °C	25	9	16.3	23.9	0.93	7.9	0.26	0.98
	80 °C	28	10	18.1	26.8	0.93	8.8	0.26	0.97
	100 °C	25	9	17.4	22.5	0.93	9.0	0.23	0.97
	120 °C	21	7	15.1	17.7	0.92	8.3	0.22	0.98
	150 °C	18	5	12.3	14.4	0.86	6.6	0.22	1.00

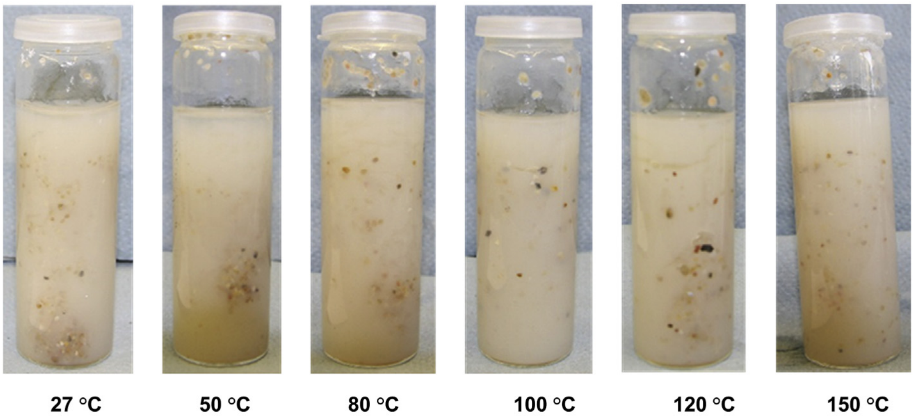
the results indicate that under static conditions the hole cleaning ability of the xanthan gum fluid was limited to ~75 °C (170 °F) and depends on the dosages used. In contrast, the sepiolite fluid retains its excellent suspending properties throughout the entire exposure period to high temperature. Apparently, the newly developed test method allows a more realistic fluid assessment under actual field conditions.

In order to determine whether the results attained with this simple and easy to carry out test are meaningful, the fluid samples were further subjected to measurements in an HTHP rotational rheometer.

3.4. HT rheometry

To probe into the validity of the static carrying capacity test, rheology measurements were carried out on fluids which were heated up to 150 °C (302 °F) in the Brookfield rheometer and kept there for up to 7 h. During this period, the time – dependent development of the

a) KCl fluid



b) K_2CO_3 fluid

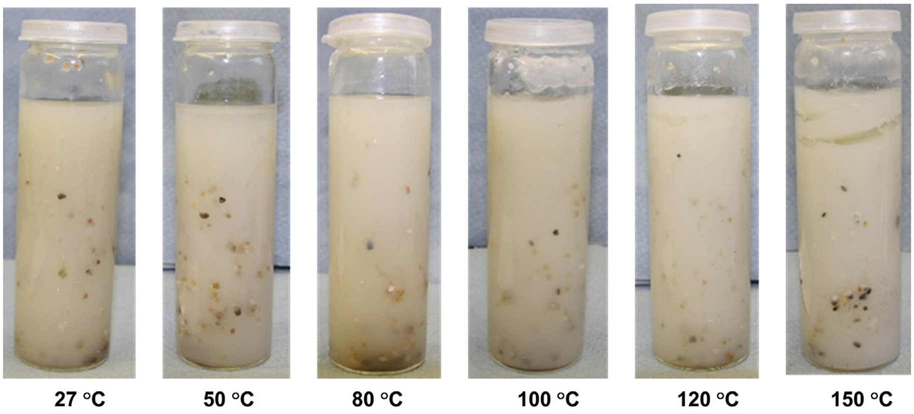
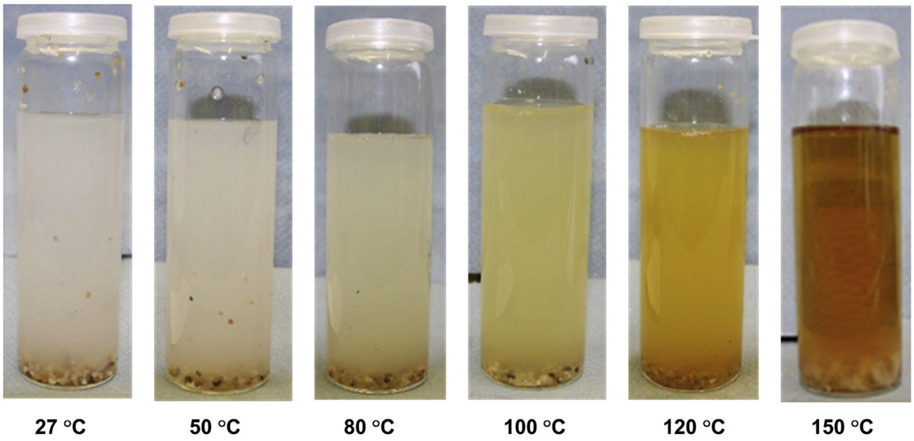


Fig. 7. Appearance of 1.5 wt% sepiolite fluid samples holding 17.6 wt% KCl (top) or 12.1 wt % K_2CO_3 (bottom) after 16 h static aging at 27 – 150 °C (80 – 302 °F).

a) 0.6 wt.% xanthan gum fluid



b) 1.0 wt.% xanthan gum fluid

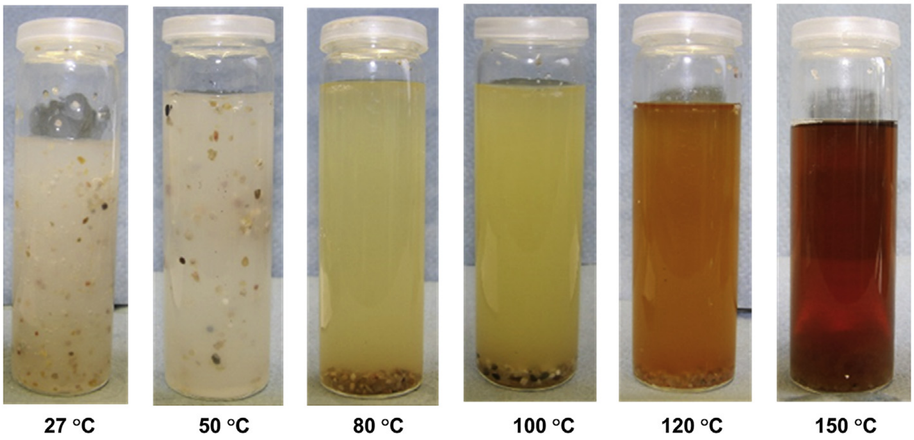


Fig. 8. Appearance of 0.6 wt% (top) or 1.0 wt% (bottom) xanthan gum fluid holding 12.1 wt% K_2CO_3.

rheology was determined. From the data, the yield point was calculated according to Eq. (2) and plotted against aging time.

The results displayed in Fig. 9 clearly demonstrate a superior thermal stability of the sepiolite fluid over the xanthan gum mud. For example, thermal thinning of the 1.5 wt% sepiolite fluid reduces its initial yield point from 17 to 14 lbf/100 ft^2, whereas the xanthan gum fluid encounters a much more pronounced drop of its YP from 13 to 3 lbf/100 ft^2, thus signifying strong degradation. Increased xanthan gum dosages cannot prevent this trend. For example, at 1 wt% of xanthan gum, YP drops from 30 to 14 lbf/100 ft^2 within 7 h, while a 2.0 wt% sepiolite fluid exhibits a reduction from 28 to 23 lbf/100 ft^2 only.

Another lesson from the HTHP rheology measurements is that under dynamic (stirred) condition as they exist in the Brookfield rheometer, for xanthan gum the drop in yield point occurs slower than under static aging conditions.

The results clearly suggest that the sepiolite fluid retains its excellent suspending property and therefore hole cleaning properties even over extended periods of time at 150 °C (302 °F). This behavior is exceptional and most advantageous to achieve high rates of penetration. It also confirms the results from the static carrying capacity tests displayed in Fig. 7 for sepiolite and Fig. 8 for the xanthan gum fluid. Similar results were obtained for the sepiolite and xanthan gum fluids based on 12.1 wt% K_2CO_3 (graphs not shown here).

The HTHP rheology measurements underline the importance of

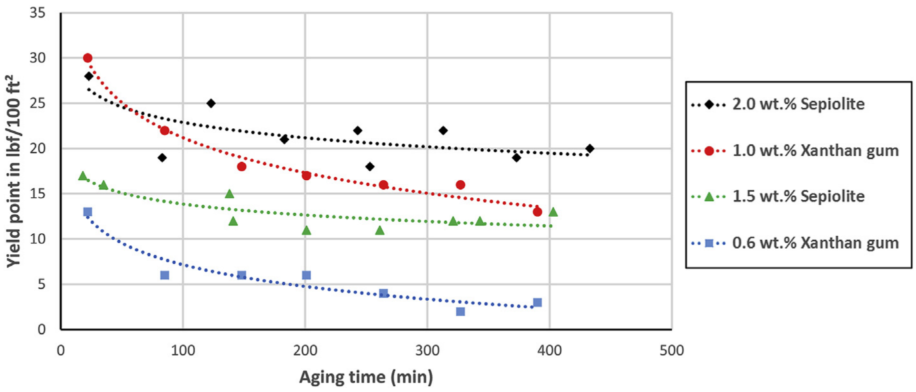


Fig. 9. Time-dependent yield point of the sepiolite and xanthan gum fluids holding 17.6 wt% KCl, measured at 150 °C (302 °F) in the Brookfield rheometer over 6 h.

testing fluid properties at *actual* temperature conditions, high or low, instead of *after* thermal aging at ambient as is practiced most often as neither the tests nor subsequent modelling of rheology could accurately predict this behavior. While maintaining a high low shear rate rheology and yield point after aging can be a good indicator of carrying capacity, this is not necessarily valid for all fluids and should be tested individually as low of hole cleaning ability can decrease drilling rates and lead to stuck pipe. Furthermore, the tests confirmed that the sepiolite fluid offers excellent yield point retention over time at high temperature and is therefore an excellent fluid for drilling high temperature or deviated wells. Most importantly, the validity of the static carrying capacity test was confirmed by HTHP rheology measurements. Hence, the static carrying capacity tests offer a simple, yet extremely practical alternative to HTHP rheology testing which requires an expensive instrument.

4. Conclusion and outlook

The newly proposed test protocol allows for a better assessment of fluid performance under actual downhole conditions such as when drilling in temperature granite and emphasizes the importance of testing under these conditions. When drilling in natural gas hydrates, testing should also be performed at low temperature, while drilling in shale would require fluid loss testing as has been reported in literature (Jaffal et al., 2017; Salih and Bilgesu, 2017). This test procedure, which is based on static aging of the fluid containing simulated cuttings reveal huge differences to the current, commonly practiced roller oven tests. While in the conventional test the xanthan gum fluid displayed similar yield point and low shear rate values than the sepiolite fluid, it performed significantly worse than the sepiolite fluid when tested at actual high temperature, under dynamic and even much more under static conditions. In addition, the proposed static carrying capacity test presents a viable and highly practical alternative to HTHP rheology measurements as it allows for an effective assessment of the high temperature suspending properties of a fluid as is especially required in deep or directional drilling. Our results for different rheological test methods demonstrate the importance of testing under realistic field conditions (temperature, well deviation, salinity etc.), as otherwise misleading data might be obtained.

The sepiolite fluid is characterized by a low plastic viscosity and high low shear rheology (= yield point) indicating a high carrying capacity, which was confirmed through static testing, and therefore good hole cleaning properties regardless of temperature and the kind of salt used as weighting agent.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jngse.2019.102964.

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