1. Introduction

As the demand for energy increases, harsh and extreme environments are explored for hydrocarbon; and deeper wells have been drilled to reach targets in formations with very high temperatures and pressures. To drill successfully, safely, and economically in such harsh environments, a drilling fluid whose rheological properties remain stable when exposed to high temperatures and that can retard shale problems is required. Regarding their technical performance, oil-based mud (OBM) systems can be used successfully in such environments [1]. However, environmental concerns, cost, and difficulty in handling pose a challenge to the use of oil-based mud systems. More so, industrial regulators are becoming stricter on the use of environmentally-friendly drilling fluids. For example, the Environmental Protection Agency in the United States of America prohibited any discharge of OBMs or their cuttings from offshore platforms [2]. In addition, the attendant high cost of litigation action and fines from made-made environmental pollution, have engendered the search for environmentally friendly additives in the formulation of drilling fluids [3]. Even though water-based fluid systems are environmentally-friendly, their instabilities when exposed to high temperatures remain a big challenge [4]. High temperature affects the hydration of components, fluid loss, clay dispersion, and the degradation of additives in water-based fluids [5]. Biopolymers are commonly used in water-based muds and degrade at high temperature, thereby leading to the reduction or complete loss of drilling fluid rheological properties as well as fluid loss and suspension properties [6]. To avoid the problem associated with the use of biopolymer at high temperatures, they are used in formations with temperatures lower than 300°F (150^{o}C) [7]. The knowledge concerning the mechanisms of biopolymer degradation at high temperatures is, therefore, essential for their stabilisation in drilling fluids. Acid-catalysed hydrolysis, oxidation-reduction reactions, and early onset of the helix-coil conformation transition are some of the mechanisms, which cause biopolymer degradation. These mechanisms can be counteracted, neutralised, or retarded to stabilise water-based drilling muds at high temperatures [8, 9, 10]. These processes are, therefore, exploited in his work to stabilise water-based drilling fluids at high temperatures by screening additives (formate salts, antioxidants, and polyglycol) that can counteract, neutralise, or retard them. Numerous research works have been done to stabilise water-based fluids and to retard problems associated with reactive shales in high-temperature drilling operations using formate salts, antioxidants, and polyglycol. For example, formate brines are used in high-temperature drilling operations to stabilise biopolymers as they act as antioxidants [11, 12, 13, 14, 15, 16]. They stabilise biopolymers at high temperatures by raising their melting (transition) temperatures (T_m) and providing free-radical properties, which retard oxidative processes [13]. At the transition temperature (T_m), a biopolymer undergoes an order-disorder conformational change; the order-disorder conformational change is followed by a loss in viscosity and by an increase in the rate of hydrolytic degradation by two orders of magnitude [14]. As potent antioxidants, formate salts could only extend the stability temperature of biopolymers up to around 175^{o}C at best [11, 15]. It is, therefore, not advisable to use only formate salts to stabilise water-based fluids above 175^{o}C. In addition, polyglycols are used to stabilise biopolymers in water-based fluids above 150^{o}C [15]. Biopolymers and polyglycols in solutions associate by intermolecular hydrogen bonding and hydrophobic interactions. These association and interactions stabilised biopolymers at high temperatures [15]. Apart from being environmentally friendly, polyglycol muds inhibited sensitive shale formations, lubricated drill pipe, and cleaned and deposited a thin and impermeable mud cake on wellbore [17]. Polyglycols are also deployed to stabilise reactive shale formations. They absorb strongly on clay rock, thereby reducing clay hydration, but are not very effective in salt-free or low-salt fluids [18]. Some users of biopolymers also used buffering agents and antioxidants to prevent their degradation at high temperatures. This method is not very successful as changes in molecular conformation in biopolymer at elevated temperature are not taken into consideration [8]. In addition, oxidation-reduction reactions which involve free radicals are the probable cause of biopolymer degradation in drilling muds and its associated viscosity loss [8]. Without the presence of dissolved oxygen and based on pH, acid-catalysed hydrolysis and base-catalysed formation reactions have significant effects on biopolymer degradation at high temperatures [9].

Apart from the detrimental effects of high temperatures on the rheological properties of drilling fluids, suspension, and fluid-loss control, there are also wellbore instability problems associated with fluid interaction with reactive shale formations. About 70% of wellbore instability problems is caused by shale [19]. As shale is made of kaolinite, illite, smectite, and montmorillonite, it has an affinity for water. The shale-fluid interaction causes shale instability [20]. Therefore, if a shale formation is drilled with a non-inhibitive water-based mud, the shale instability problems occur [21, 22, 23].

Consequently, water-based fluids are formulated with organic or inorganic additives to improve shale inhibition properties[24,25,26]. The organic inhibitors are classified into three categories [24]: (1) monomeric amine shale inhibitors (they have a low level of inhibition and high ammonia odour), (2) oligomeric amine shale inhibitors (they provide more permanent shale stabilisation), and (3) polyamine shale inhibitors (they are a more permanent shale inhibitor and less attractive due to their incompatibility with other anionic additives). The inorganic shale inhibitors are salts such as sodium chloride, calcium chloride, and potassium chloride. Potassium chloride is a commonly used inorganic additive for shale inhibition. However, high concentrations of potassium chloride in drilling fluids are an environmental concern [27]. Another inorganic shale inhibitors are silicate additives [22]. Nevertheless, silicates have the potential to damage formation, and the control of rheological properties is a major problem with the use of a silicate-based mud system [28].

All these efforts of researcher have brought to the fore the attempt made in stabilising water-based fluids at high temperatures and in inhibiting shale hydration in reactive shale formations. None has, however, considered the combination of additives used in this work. The objective of this work, therefore, was to examine whether the combination of antioxidant, formate salts, polyglycol, and pH control agents could increase the stability temperatures of diutan gum (a biopolymer) in water-based drilling fluids and retard shale dispersion at high temperatures. This work goal was to determine the rheological properties of fluid formulations experimentally and to find the drilling fluid additives that can stabilise biopolymers in water-based drilling fluids and inhibit shale dispersion at high temperatures.

2. Experimental

2.1. Materials

The materials used in the mud formulations were bentonite, diutan gum, potassium formate, sodium erythorbate, polyethylene glycol (PEG), sodium carbonate, sodium bicarbonate, defoamer, and Marcellus shale rock. Bentonite was purchased from Hilton Instrument, Aberdeen; CP Kelco, USA provided diutan gum powder; Potassium formate, sodium erythorbate, polyethylene glycol, sodium carbonate and sodium bicarbonate were purchased from Fisher Scientific, United Kingdom. The defoamer, Dispelair Se574, was provided by Blackburn Chemicals Limited, Blackburn, United Kingdom. Marcellus shale was purchased from Kocurek Industries INC. Table

1 shows the additives used in this work and their primary function.

Table 1

Additives screened for their effects on biopolymers at high temperatures and shale rock.

Additives	Primary function	Amount
Potassium formate (Ibm)	Temperature stabilizer/Anti-oxidant	1 – 3
Sodium erythorbate (Ibm)	Antioxidant/ Oxygen scavenger	1 –3
Polyethene glycol 8000(PEG)(Ibm)	Shale stabiliser	1 –3
sodium carbonate (Ibm)	Buffer	1 – 3
sodium bicarbonate (Ibm)	Buffer	0.1–1.2
Dispelair Se574 (bbl)	Defoamer	0.003 – 0.03

The above additives were screened for their effects on diutan gum in drilling muds at high temperatures. PH preserving agents were added to the drilling fluid samples to counteract acid-catalysed hydrolysis. Potassium formate was deployed as an antioxidant and temperature stabiliser. Sodium erythorbate served as antioxidant and oxygen scavenger. An antioxidant protects a biopolymer at high temperatures from thermal degradation, by preventing oxidative processes and reacting with hydroxyl free radicals [15].

2.2. Experimental procedure

2.2.1 Drilling fluid formulations

The electronic scale was used to measure the specified amount of additives and graduated cylinder was used to measure 350ml of water and a specified volume of defoamer. The Hamilton Beach mixer (Fig. 1) was used to stir all the drilling fluid formulations at high speed. The Model 1100 viscometer was used to measure the rheological properties of the mud formulations. With speed (RPM) accuracy of 0.001, the Model 1100 viscometer (Fig. 2) is fully automated and operated with ORCADA® software portable [29]. Before testing was carried out, the Model 1100 viscometer was calibrated.

Bentonite was first added to 350 ml (one lab barrel) of freshwater in a mud cup.

The cup contents were placed under the Hamilton Beach mixer spindle and stirred for 30 minutes at room temperature. After 30 minutes of stirring, the bentonite-water suspension was aged for 16 hours at room temperature to hydrate fully. After that, the control mud formulation was prepared by adding diutan gum to the pre-hydrated bentonite-water suspension and aged at room temperature for sixteen hours before its rheological properties were measured at 25°C, using Model 1100 viscometer. Three more control mud samples were also prepared but pressurised in aging cells (Fig. 3) to prevent them from boiling at 100°C and above, and aged dynamically in a roller oven (Fig. 4) at different temperatures for sixteen hours. After aging, the mud samples were allowed to cool to 25°C; thereafter, the rheological properties of the mud samples were measured at 25°C, using Model 1100 viscometer. The other mud formulations were prepared by adding the additives as shown in Table 1 and diutan gum to the pre-hydrated bentonite-water suspension and stirring for 30 minutes at room temperature using the Hamilton Beach mixer. The additives and diutan gum were gradually added to the pre-hydrated bentonite-water suspension while mixing. After 30 minutes of stirring, the mud formulations were pressurised in aging cells to prevent them from boiling at 100°C and above. The pressurised muds were after that aged dynamically at different temperatures for 16 hours in a roller oven to mimic the interaction between drilled cuttings and drilling fluids in a wellbore annulus. After aging for 16 hours at different temperatures, the mud formulations were allowed to cool to 25°C before testing. After that, the rheological properties of the mud samples were measured at 25°C, using Model 1100 viscometer. Eleventh drilling fluids were formulated in total to measure their rheological properties and to determine their shale inhibitive characteristics. The additives used in the mud formulations were kept within acceptable limit [30]. Bentonite in the mud formulations was kept low as excess solids in drilling fluids cause pipe sticking and increase drag and torque [31]. 10.5 lbm of bentonite was used in all the mud formulations (3% w/v concentration). The Newtonian viscosities of the additives measured were not more than 1cP. Table 2 shows drilling fluid formulations with the biopolymer and the additive package.

Fig. 1. Hamilton Beach Mixer

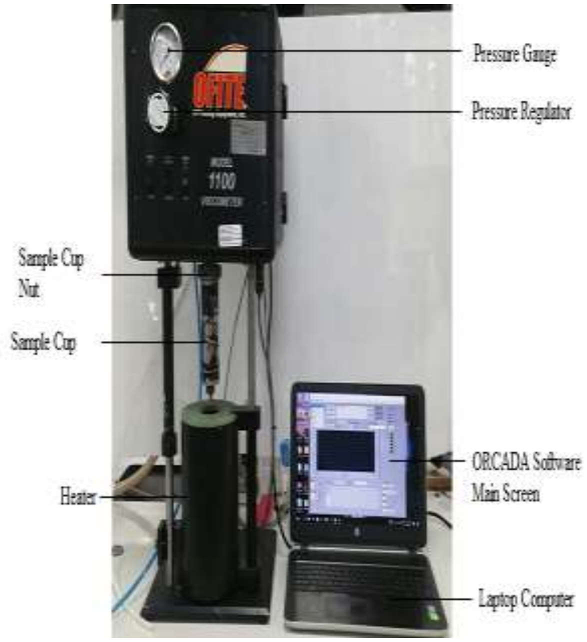


Fig. 2. Model 1100 Viscometer and laptop

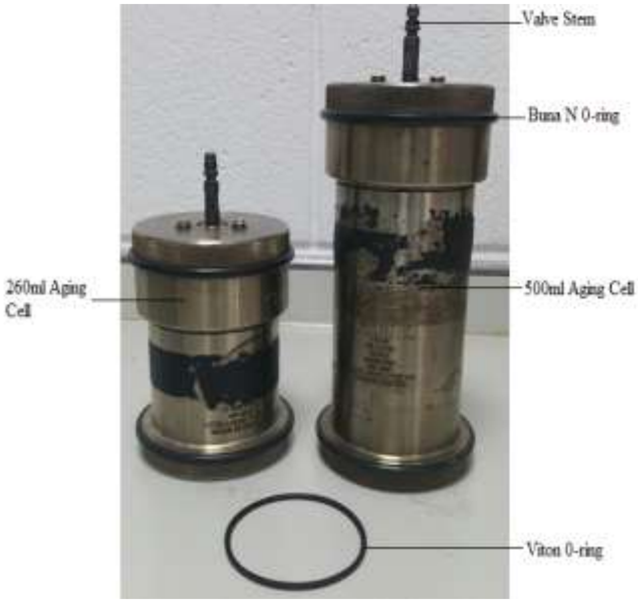


Fig. 3. Aging cells

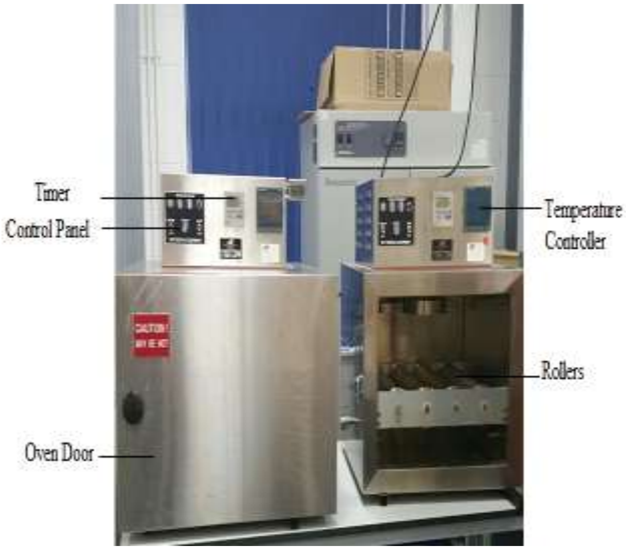


Fig. 4. The roller oven

Table 2 Mud formulations with diutan gum and the additives

Mud Name	Mud 1	Mud 2	Mud 3	Mud 4	Mud 5	Mud 6	Mud 7	Mud 8	Mud 9	Mud 10	Mud 11
Additives											
Water(bbl)	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Bentonite(Pre-hydrated)(Ibm)	10.5	10.5	10.5	10.5	10.50	10.5	10.5	10.5	10.5	10.5	10.5
Diutan gum (Ibm)	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
K-Formate (Ibm)	-	-	-	-	3.0	3.0	3.0	3.0	3.0	3.0	3.0
Sodium Erythorbate (Ibm)	-	-	-	-	-	-	1.0	1.5	1.5	3.0	1.5
Na carbonate (Ibm)	-	-	-	-	0.5	0.5	1.0	1.5	1.5	1.5	1.5
Na Bicarbonate (Ibm)	-	-	-	-	-	-	0.8	1.2	1.2	1.2	1.2
DispelairSE 574(bbl)	-	-	-	-	-	-	0.01	0.01	0.01	0.01	0.03
PEG(8000) (Ibm)	-	-	-	-	-	-	1.0	1.0	1.0	1.0	2.0

2.2.2. Shale Characterization

The Marcellus shale sample was characterised using a scanning electron microscope (SEM) photos and X-ray diffraction to determine its mineral and elemental contents. XRD analysis was carried out at Salford Analytical Services, University of Salford, United Kingdom. SEM photos of dry samples and samples immersed in fluids were taken. XRD was recorded at 15KV.

Table 3

XRD of Marcellus shale rock [32]

Mineral	Average mineral content %
Quartz silt	20
Clay	50
Pyrite	5
Calcite	25
Clay mineral Content %	
Chlorite	15
Illite	70
Illite-smectite	15 (mixed layered clay)
Illite-chlorite	tr
Kaolinite	-

2.2.3. Dispersion Tests

The test was intended to simulate the transportation of drilled cuttings in drilling fluids to the surface from a wellbore annulus: The shale cuttings were hot rolled dynamically in a roller oven. The shale samples were broken to sizes less than 8mm with a hammer. A sieve shaker, and 8 mm and 599-micron sieve sizes were used to prepare the shale cuttings for the dispersion tests. 6.0 g of shale cuttings and the inhibitive mud formulation were added to an aging cell. Another 6.0 g of shale cuttings and fresh water were added to another ageing cell. The aging cells were pressurised to prevent the drilling fluids from boiling at 100°C and above. After that, the aging cells were hot rolled dynamically in a roller oven at 120°C for 16 hours. After 16 hours, the aging cells were cooled, and their contents were poured into a 599-micron sieve. The sieve contents were washed with running water to remove all the shale cuttings less than 599 microns. The retained shale cutting in the drilling mud and fresh water were placed in an oven at 105ºC for 24hrs. After being dried, the shale cuttings were weighed.

The same procedure above was followed for the dispersion test on shale plugs. However, 14.99g of shale plug and 14.77g of another shale plug were immersed in freshwater and drilling fluid formulation respectively and aged dynamically in a roller oven at 120°C for 16 hours. The shale recovery rate from water (%RW) and shale recovery rate from muds (%RM) were calculated on a dry mass basis as follows:

$$\%RW = \frac{W_W}{W_{IC}} \times 100\ \% \qquad 1$$

$$\%RM = \frac{W_M}{W_{IC}} \times 100\ \% \qquad 2$$

Where W_W is the weight of dried shale cuttings recovered from freshwater (g); WM is the weight of dried shale cuttings recovered from the drilling fluid formulation (g), and W_{IC} is the initial weight of dried shale cuttings (g).

3. Results and discussion

3.1. Water-based mud formulations

Tables 3,4, and 5 presents the measured rheological properties – PV, YP, AV and 10-sec and 10-min gel strengths – of the mud formulations. Mud 1 was adopted as the control mud sample; Muds 2, 3 and 4 had the same composition and concentration as Mud 1 but were aged dynamically at different temperatures for 16 hours to determine the stability temperature of diutan gum in bentonite-water suspension.

Table 3

Properties of Muds 1-7 before and after aging

Muds	1	2	3	4	5	6	7
	(BHR @25ºC)	(AHR @115 ºC)	(AHR @120 ºC)	(AHR @130 ºC)	(AHR @150 ºC)	(AHR @170 ºC)	(AHR @170 ºC)
Properties							
Shear stress @511s^{-1}	32.0	16.0	12.0	6.0	27.0	12.0	`16.0
Shear stress 1021 s^{-1}	48.0	24.0	19.0	10.0	34.0	16.0	23.0
Viscosity (cP)@511s^{-1}	30.0	15.0	14.0	6.0	25.0	12.0	15.0
10-sec Gel	18.0	10.0	5.0	1.0	23.0	11.0	9.0
10-min	21.0	11.0	5.0	1.0	24.0	17.0	10.0
Plastic viscosity (PV)(cp)	10.0	5.0	4.0	2.0	5.0	3.5	4.0
Yield Point (YP) (Ib/100ft^2)	24.0	11.0	8.0	4.0	22.0	7.0	12.0

Table 4

Properties of Muds 8-11 after aging

Muds	8	9	10	11
	(AHR@200 ºC)	(AHR@210 ºC)	(AHR@232 ºC)	(AHR@232 ºC)
Properties				
Shear stress @511s^{-1}	24.0	17.0	5.0	10.0
Shear stress 1021 s^{-1}	25.0	20.0	7.0	13.0
Viscosity (cP)@ @511s^{-1}	18.0	15.0	10.0	19.0
10-sec Gel	11.0	5.0	4.0	5.0
10-min	15.0	6.0	4.0	6.0
Plastic viscosity (PV)(cp)	3.0	3.0	2.0	4.0
Yield Point (YP) (Ib/100ft^2)	17.0	7.0	4.0	8.0

Table 5

Experimental measurement of viscosities of mud formulations

Shear Rate (s^{-1})		1021	511	341	170	102	51	10
Viscosity (cP)	Mud 1 BHR @ 25°C	23	30	28	68	109	212	990
	Mud 2 AHR @ 115°C	12	19	25	40	58	96	300
	Mud 3 AHR@120°C	9	12	14	20	27	40	80
	Mud 4 AHR @ 130°C	4.5	6	7	10	12	17	70
	Mud 5 AHR@ 150°C	17	25	36	70	112	234	1110
	Mud 6 AHR@ 170 °C	8	12	14	25	42	94	442
	Mud 7 AHR@170 °C	11	15	20	36	60	100	570
	Mud 8 AHR@200 °C	12	19	28	54	90	200	700
	Mud 9 AHR@210 °C	17	27	38	74	123	243	800
	Mud 10 AHR@232 °C	6	10	12	22	40	98	400
	Mud 11 AHR@232 °C	12	19	27	51	86	174	642

Fig. 5 compares the viscosities of the control mud samples at different shear rates. It can be seen that the control mud formulations exhibited shear thinning behaviour: The viscosities of the muds decreased as the shear rate increases. Fig. 9 shows what happens to the viscosities of the mud formulations with increasing aging temperatures. As can be seen, the viscosities of the control mud formulations decrease as the aging temperature increases, meaning that diutan gum underwent thermal thining. **Figs. 6 (a)** and **(b)** show a biopolymer's behaviour at high temperatures and the loss of a biopolymer's viscosity in a solution at melting (transition) temperature respectively[15]. At the transition temperature (T_m), a biopolymer can undergo an order-disorder conformational change; the order-disorder conformational change is followed by a loss in viscosity and by an increase in the rate of hydrolytic degradation by two orders of magnitude [14]. As shown in Fig. 6 (b), the effects of high temperatures on diutan gum were irreversible after aging at 120°C for 16 hours, regarding the loss in its viscosity. Analysis of Fig. 9 shows that the control mud formulation lost more than half of its viscosity after dynamic aging at 120°C for 16 hours. A stable drilling fluid retains at least half of its viscosity after aging at a temperature for 16 hours [12]. These results, therefore, indicate thermal degradation of diutan gum in bentonite-water suspension after aging at 120°C for 16 hours.

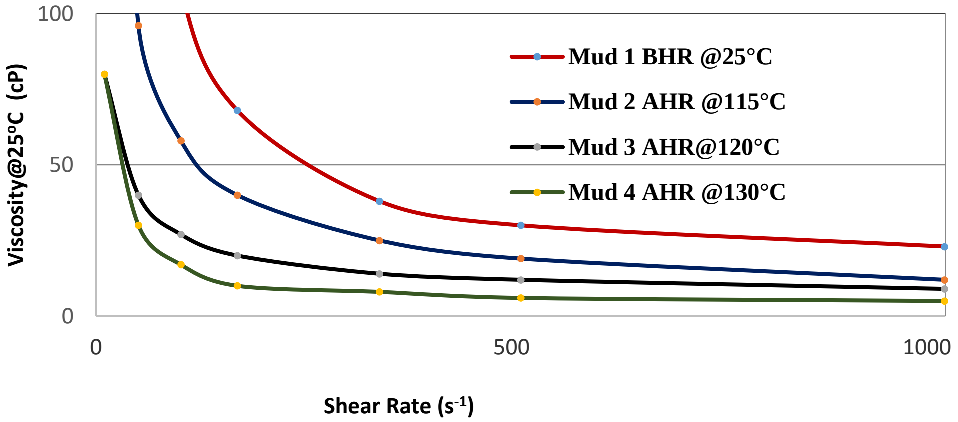
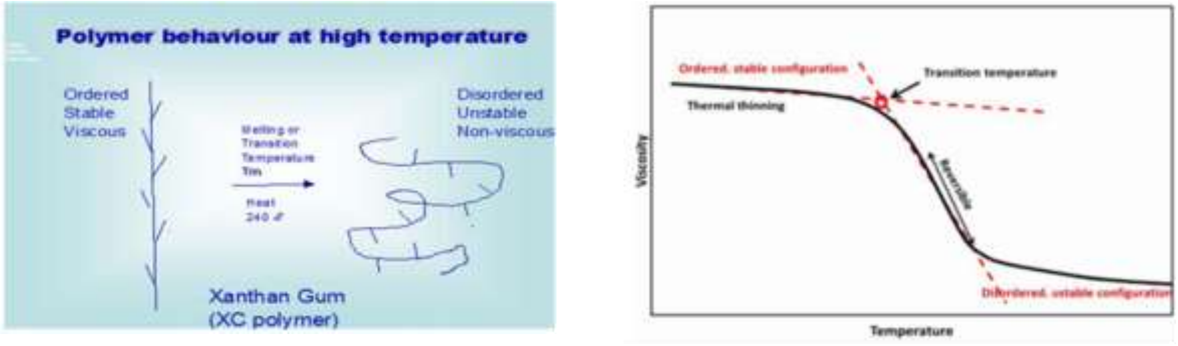


Fig. 5. The viscosities of the control drilling fluid formulations at different shear rates.



(a)

(b)

Fig. 6: Effects of temperature on biopolymer[15]: (a) xanthan gum's behaviour when heated to its transition(melting temperature, T_m) ; (b) the effects of temperature on the viscosity of a typical biopolymer.

As shown in Fig.7, after aging at 115°C for 16 hours, Mud 2 lost 50% of its viscosity when compared with the control mud sample, Mud 1; its viscosity measured at 25°C was 16 cP at 511s^{-1} while the viscosity of the control mud sample (Mud 1) measured at 25°C was 32cP at 511s^{-1}. These experimental results, therefore, show that diutan gum stability temperature in bentonite water-suspension after aging for 16 hours is 115°C. Fig.8 shows the low shear rate viscosities of the control mud formulations. The low shear rate viscosities of the control mud formulation after aging at 120°C and 130°C for 16 hours were very low. A drilling mud with high viscosity at low shear rate is required for a successful drilling operation [33]. The low viscosities of the control mud formulation - after aging at 120°C and 130°C for 16 hours and measured at low shear rates - indicate its poor cuttings suspension capacity. In addition, the values of the YP and Gel strengths of the control mud formulation measured after aging at 120°C and 130°C were very low (**Figs.7&8**), thus showing the control fluid instabilities. Consequently, the control mud formulation is not suitable for use in formation with temperatures above 115°C. In order words, when exposed to a temperature above115°C for 16 hours, diutan gum in a bentonite-water suspension will lose more than 50% of its viscosity through biopolymer degradation processes. To design a water-based drilling fluid containing diutan gum for formations with temperatures above 115°C, we deploy additives that could prevent, counteract, and retard the biopolymer degradation processes as shown in Table 1.

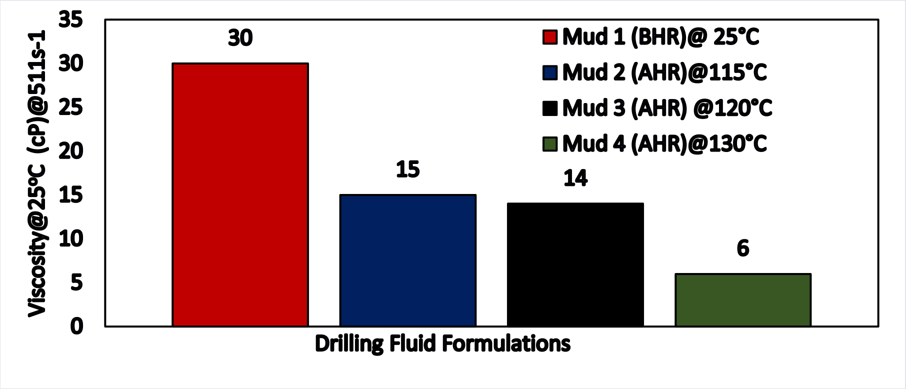


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Fig. 7. Viscosities of the control mud formulation aged at different temperatures

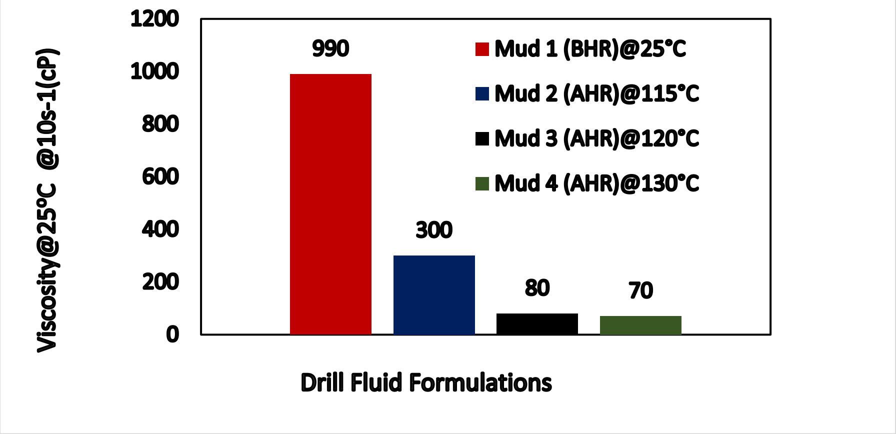


Fig. 8: Low shear rate viscosities of the control mud formulation aged at different temperatures.

3.2. Effects of potassium formate, sodium erythorbate, and polyethylene glycol on mud formulations at high temperatures

3.2.1. The viscosities of the mud formulations

The stability temperatures of the mud formulations were investigated through aging tests. Tables 3, 4 and 5 summarise the measured rheological properties of the drilling muds formulated with the additives**.**

Figs. 9 and 10 compare the viscosities of drilling muds prepared with the additives and with those of the control mud sample, Mud 1. As shown in Fig. 9, the mud formulations exhibited shear thinning behaviour: The viscosities of the mud formulations decrease as the shear rate increases. At low shear rates, the slopes of the curves were very high and became very small at high shear rates, suggesting that not very high pump pressure will be required to initiate flow with the mud formulations. In addition, the low solids in the mud formulations and the sharp reduction in viscosity as the shear rate is increased show that the mud formulations will flow at high speed through the bit nozzle, thereby cleaning the bit face and carrying drilled solid particles effectively in the wellbore. As shown in Fig. 10, after aging dynamically for 16 hours at temperatures above 120°C, the viscosities of the mud formulations – Mud 5, Mud 7, Mud 8, Mud 9, and Mud 11-were more than 50% of the viscosity of the control mud formulation, Mud 1. Howard et al [12] showed that a stable mud retained 50% of its viscosity after aging at a given temperature for 16 hours. These experimental results, therefore, show that the additives used in this work were very effective in protecting diutan gum in the mud formulations from thermal degradation at high temperatures. During measurement, the drilling fluid formulations exhibited excellent thixotropic behaviour at low shear rates.

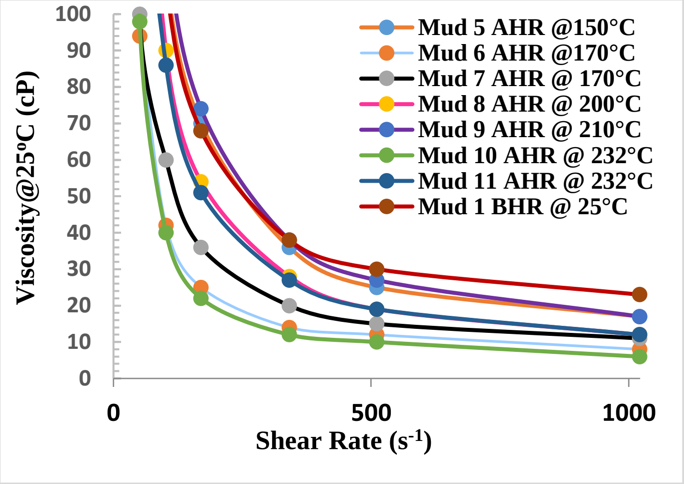


Fig. 9. The viscosities of drilling fluid formulations and that of the control mud formulation, Mud 1 at different shear rates.

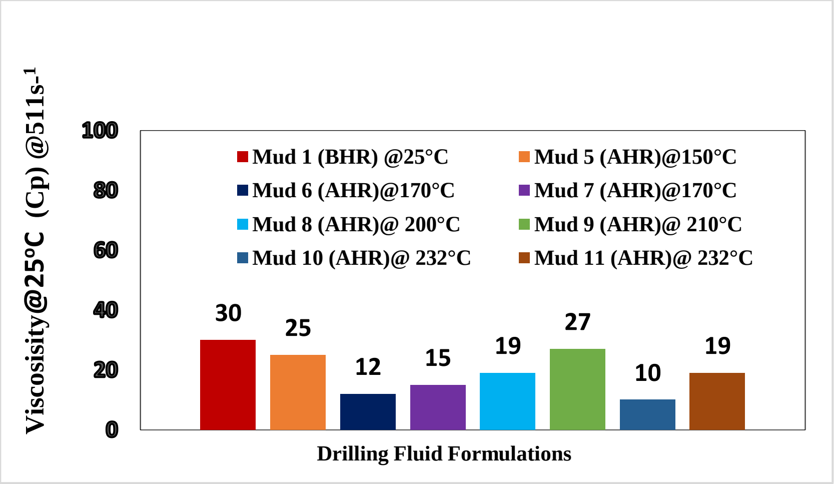


Fig. 10. Comparison of the viscosities of the drilling fluid formulations with the viscosity of the control mud formulation, Mud 1.

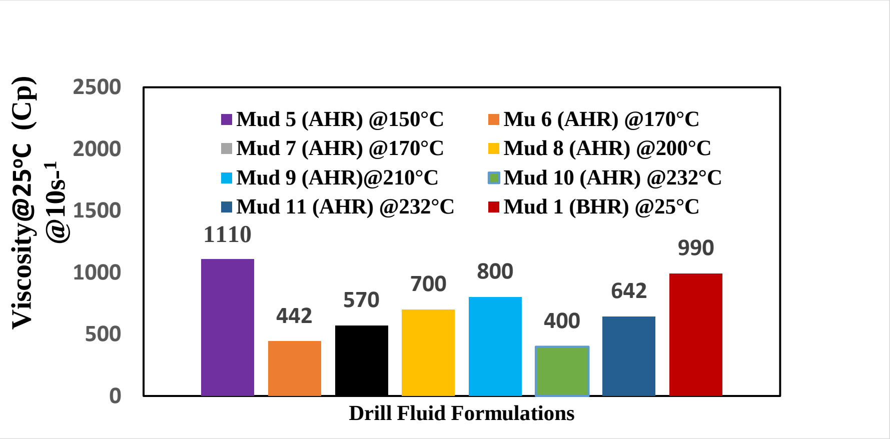


Fig. 11. Comparison of the viscosities of drilling mud formulations and that of the control mud formulation at a low shear rate.

As shown in Fig. 9, the low viscosities at high shear rates indicate that not high pump pressure will be needed to pump the mud formulations, thereby preventing formation fracture. In addition, the viscosities of the mud formulations were very high at low shear rates, thus indicating drilled cuttings and weighting material suspension potentials (Fig. 11).

As shown in Fig. 10, Mud 5 formulated with potassium formate without the antioxidant and polyethylene glycol remained stable up to 150°C when compared with Mud 1. When aged at 170°C for 16 hours as Mud 6, Mud 5 lost more than 50 % of its viscosity. It can, therefore, be seen that the potassium formate could only protect the biopolymer up to 150°C. This finding confirmed the suggestion that formate salt could stabilise biopolymer up to 150°C [7, 15]. In addition, Mud 9 formulated with a low concentration of polyethene glycol and a high concentration of the antioxidant (sodium erythorbate) remain stable up to 210°C, when compared with Mud 1. Mud 10 formulated with the same composition and concentration as Mud 9 degrade when aged for 16 hours at 232°C. Therefore, the combination of potassium formate and the antioxidant could only protect diutan gum in the mud formulations up to 210°C. Potassium formate raises the transition temperature(Tm) of biopolymers and provides free-radical properties that retarded oxidative processes, thereby stabilising them at high temperatures; sodium erythorbate, the antioxidant, prevented oxidative processes and reacted with hydroxyl free radicals, thereby stabilising biopolymers at high temperatures [15]. Mud 11 formulated with a higher concentration of polyethylene glycol remain stable up to 232°C when compared with Mud 1. It can be seen that the combination of polyglycol, a formate salt, and sodium erythorbate protected diutan gum in the mud formulations from thermal degradation at higher temperatures. Polygycol acted as a sacrificial agent, thereby protecting the biopolymer from thermal degradation [15]. As the maximum operating temperature of the roller oven was 232°C, the experiments were not carried out beyond 232°C. The best additive package that stabilised the mud formulations at high temperatures was the combination of potassium formate, sodium erythorbate, and polyethylene glycol.

3.2.2. The plastic viscosities of the mud formulations

Fig. 12 shows the effects of temperature on the plastic viscosities of mud samples formulated using diutan gum with the mud additives before and after aging for 16 hours at different temperatures. Plastic viscosity is a combination of the viscosities of the liquid and solids in a drilling fluid. The experimental data showed a decrease in the plastic viscosities of the mud formulations (Muds 2-11) at high temperatures when compared with that of the control mud sample, Mud 1. This decrease in plastic viscosities could be due to the deflocculation and dispersion of the mud particles after aging at high temperatures. High plastic viscosity is not desirable in drilling fluid as it causes the following: (1) increase in torque and drag, (2) low bit penetration rate, (3) increase surge and swab pressures, and (4) the possibility of pipe sticking. As high plastic viscosity is associated with wellbore problems, the low plastic viscosities at high temperatures indicate that the mud formulations are lubricious and are capable of a fast rate of penetration (ROP). It is clear that the mud formulations - those that retain their rheological properties and have low plastic viscosities at high temperatures- are suitable for use as drilling fluids.

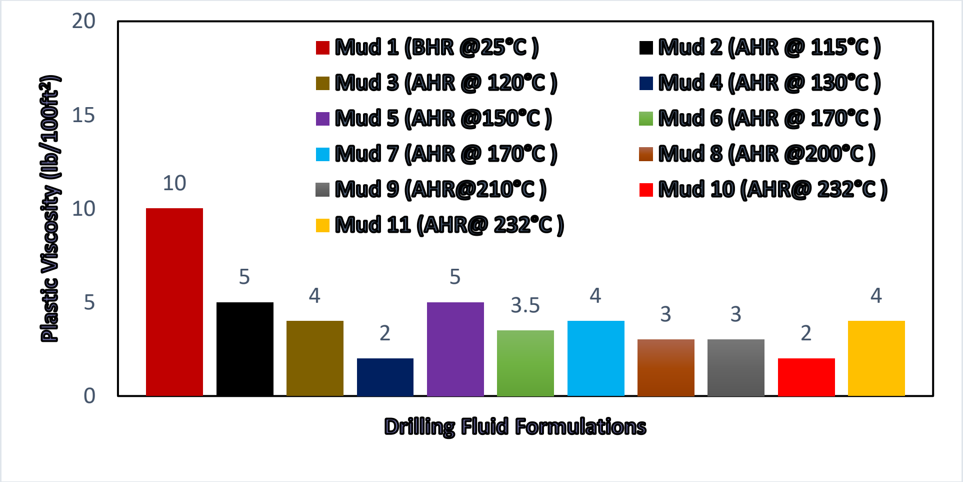


Fig. 12. Plastic viscosities of mud formulations at different temperatures.

3.2.3. Gel strength of the mud formulations

Fig. 13 compares the gel strengths (10 seconds and 10 minutes) of Muds 2 – 11 with the gel strength (10 seconds and 10 minutes) of Mud 1, the control mud sample. The measure of the attractive forces presents in a drilling fluid when it is static is gel strength. Very high gel strength is not desirable as it can cause swabbing, surging, difficulty in running logging tools, entrapping of gas in mud, and difficulty in removal of cuttings at the surface [31]. Drilling muds with low gel strength cannot suspend drilled cutting and weighting material even if they have high viscosity. High viscosity can only slow down the settling rate of solid particles to the bottom of a drilled hole. The settling of drilled cutting and weighting materials to the bottom of a drilled hole can cause pipe sticking and deposition of cutting beds. The additive package shown in Table 1 was used to formulate drilling Muds 5-11 to protect diutan gum at high temperatures.

As shown in Fig. 13, Mud 5 formulated with potassium formate and sodium carbonate without the antioxidant and polyethylene glycol has a higher gel strength than the control mud, Mud 1 even after aging for 16 hours at 150°C. The same mud composition and concentration were used to formulate Mud 6. Aged at 170°C for 16 hours, Mud 6 gel strength was high when compared with Mud 1. The potassium formate promoted the linking of solid particles to form gel strength, but the presence of carbonate and bicarbonate caused flocculation in the drilling fluid formulations [34]. Flocculation in water-based fluids increased gel strength while deflocculation decreases the gel strength. The carbonate and bicarbonate in Muds 5 and 6 caused flocculation, which led to high gel strength. The high gel strength can be treated with the addition of dispersant, the base content (pH) of which must be adequate to activate it. The problem of high gel strength could equally be solved temporarily by dilution with water [35].

When the difference between the10-seconds and the 10-minutes gel is small, the gel strength is said to be flat. Flat gel strength is desirable. Gel strength is progressive when the difference between the10-seconds and 10-minute gel readings is wide. Progressive gel is not desirable. As shown in Fig. 13, Muds 7, 8, 9, 10 and 11 have flat gel strengths. The additives used in this work, therefore, create favourable conditions for the deflocculation and linking of solid particles in the fluid formulations at high temperatures, hence the flat gel strengths.

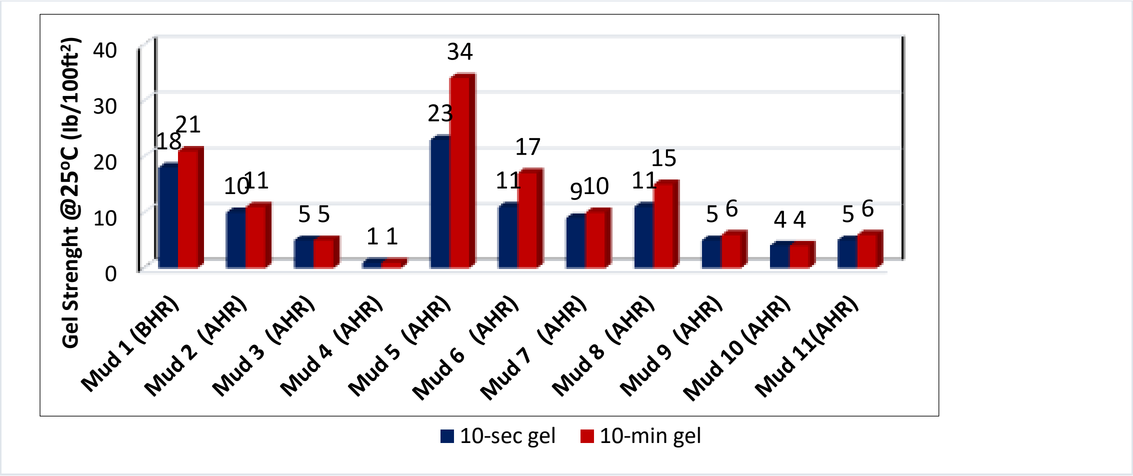


Figure 13: Gel strengths of mud formulations at different temperatures.

3.2.4. Yield points of the mud formulations

Fig.14 shows the YP of the drilling fluid formulations with the additives. It can be seen that the mud samples retained their YP values after aging dynamically at high temperatures. The retained YP values indicate that the mud formulations can carry drilled cuttings and weighting materials successfully.

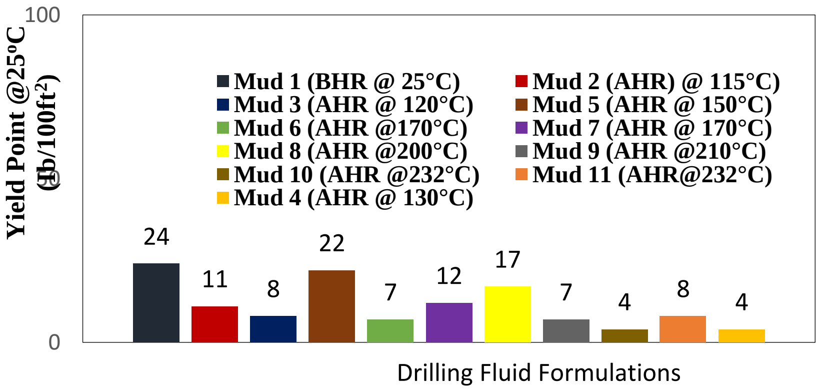


Fig. 14. Yield points of mud formulations at different temperatures.

3.3. Shale characterisation results

The shale samples were characterised using energy-dispersive X-ray spectroscopy (EDX) and SEM photos. The SEM photos showed the presence of illite (Fig. 15). The presence of illite and smectite in the shale sample was confirmed as the elements, Si, K, Mg, Fe, Ca, Na and Al were found in the shale samples, and other components were calcite, pyrite, and quartz (Fig 16). The dry SEM - before aging in freshwater and drilling fluid formulation - showed the clay distribution and pore structure (Figs.17 (a) and (c)). Illite has low exchange capacities (CEC), indicating that it is not swelling clay as swelling clays have high CEC values [35], and it is suitable for hardness testing as it is less swelling and dispersible. The most common instability in shale formation is shale swelling and dispersion.

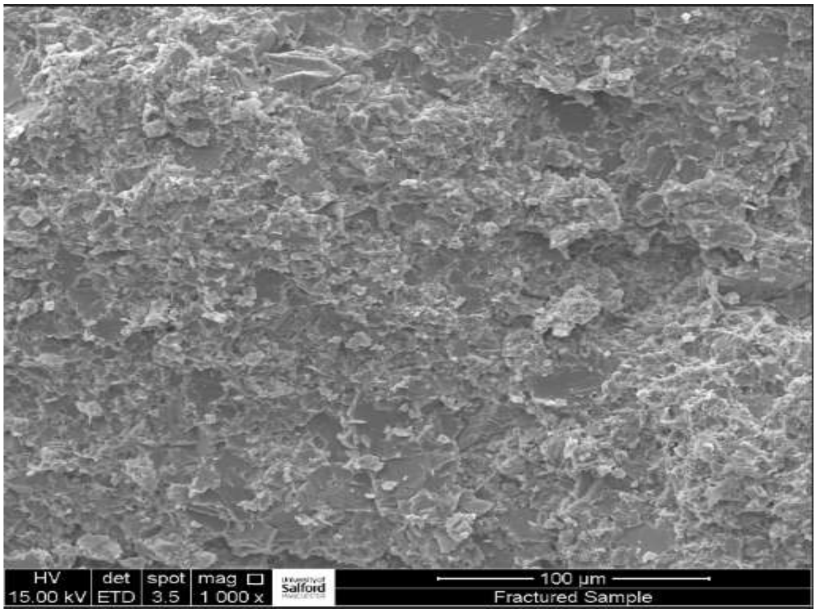


Fig.15. SEM Image of shale cutting

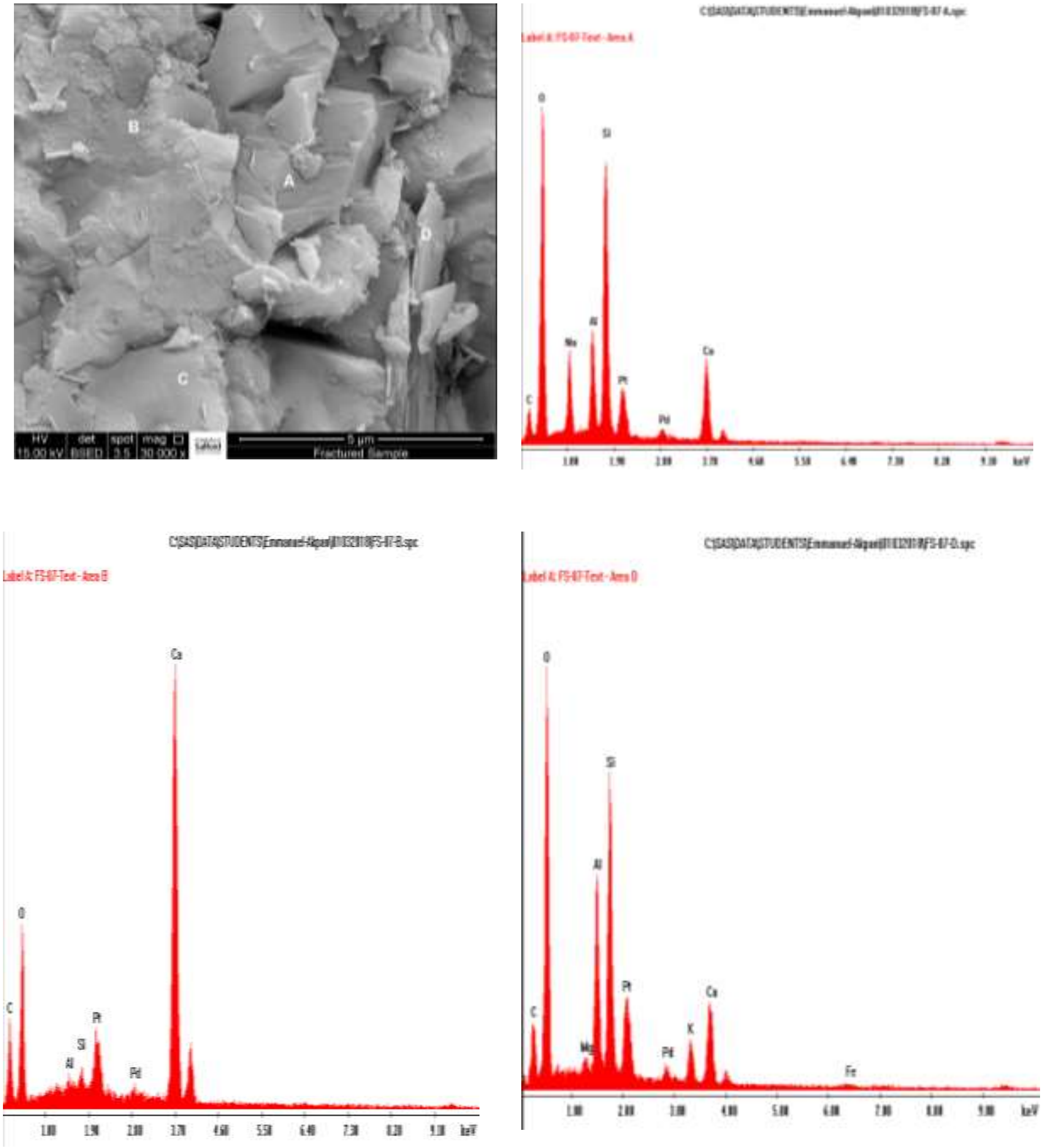
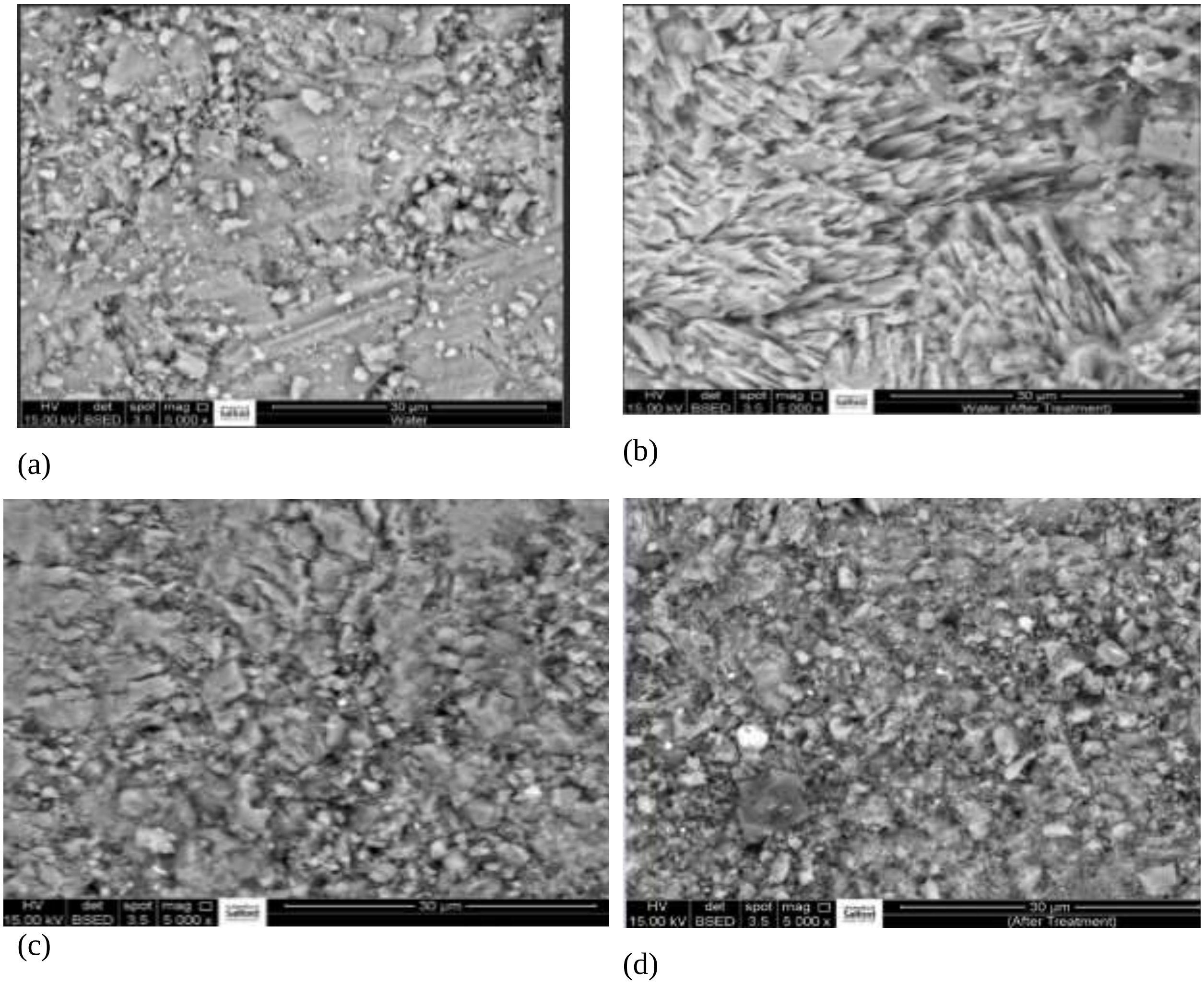


Fig. 16. EDX of shale sample showing the presence of illite, smectite, calcite, and quartz.

3.4. Dispersion test

The problems associated with shale drilling are swelling and dispersion. Swelling involves the expansion of shale when it comes in contact with water. Dispersion involves the disintegration of shale rock when it is exposed to water; the dispersion of shale is caused by stresses in the wellbore, the extent to which the shale is hydrated, the drilling fluids velocity in the wellbore annulus, and the degree of shale brittleness [36]. Reactive shale formations are associated with hole instability problems, which cause wellbore failure. Before testing, the shale samples show well-formed structures (Figs.17 (a) and (c)). The post-SEM test analyses showed that freshwater and the drilling fluid formulation interacted with the shale samples**.** Figs. 17 (a) and (b) show the shale samples before and after aging in freshwater dynamically for 16 hours at 120°C. It can be seen that illite/smectite in the shale sample responded negatively to the freshwater (Fig. 19 (b)). Figs. 19 (c) and (d) show a shale sample before and after aging dynamically for 16 hours at 120°C in the inhibitive fluid formulation. After testing, the illite had dispersed as it came in contact with freshwater (Fig. 17 (b)) while the additives in the mud formulation filled the pore spaces in the shale, thereby preventing mud filtrate penetration (Fig. 17 (d)). The shale sample aged in freshwater showed that the clay mineral had dispersed, thereby leaving a spike-like structure on the surface of the shale sample (Fig.17 (b)). Conversely, there was no significant change in the surface structure of the shale sample aged in the inhibitive mud formulation (Fig.17 (d)). It can be seen that the additives sealed the pores spaces and were absorbed on the shale samples surface, thereby preventing the interaction between water in the drilling fluid and the shale rock. In addition, the potassium formate and sodium erythorbate reduced the water activity of the inhibitive mud formulation, thereby preventing the movement of water into the shale rock. The stabilisation of the shale sample was also achieved through the cation exchange process as the smaller K^+ ions replaced the Na+ ions in the shale sample, thereby reducing the spaces between the shale layers [31]. The interaction among potassium ions, sodium ions, and polyethylene glycol on the clay surface was, therefore, essential in reducing the shale dispersion. The cations, K^+ and Na^+, were provided by non-chloride salts, potassium formate and sodium erythorbate. High concentrations of chloride salts in water-based fluids are environmental concerns [27]. It can be seen that the mud formulation was strongly shale-inhibitive. When a shale plug (14.99g) was exposed to freshwater, it was found that 79% of the shale plug was recovered. When another shale plug (13.77g) interacted with the inhibitive mud formulation, it was found that 100% of the shale rock was recovered. It is possible that the additives in the inhibitive mud formulation encapsulate the shale rock, thereby preventing water invasion.



(a)

(b)

(c)

(d)

Fig. 17. Shale samples aged in fluids: (a) Shale sample before being aged in freshwater; (b) Shale sample after being aged in freshwater at 120°C for 16 hours; (c) Shale sample before aging in an inhibitive mud formulation; (d) shale sample after aging in an inhibitive mud formulation at 120°C for 16 hours.

The shale sample investigated in this work has high clay contents. The weights of shale cuttings recovered from freshwater and inhibitive mud formulation are shown in Table 6; the effects of freshwater and the mud additives on the shale dispersion is shown in Fig. 18. The %RW value for shale cuttings recovered from freshwater was 78%. The %RM value of cuttings recovered from mud formulated with the additives was 100%. The additives show a positive effect in retarding the dispersion and disintegration of the shale samples. The interaction among potassium ions, sodium ions and polyethylene glycol at the clay surface was vital in preventing the disintegration and dispersion of the shale sample. The shale sample was, therefore, not susceptible to dispersion as the additives restricted the movement of water into the shale.

Table 6

The initial and recovered shale cuttings

Fluid	Initial Dried Weight (Shale cuttings)	Dried Weight after Dispersion test (Shale cuttings)
Freshwater	6.0	4.69
Mud 11	6.0	6.00

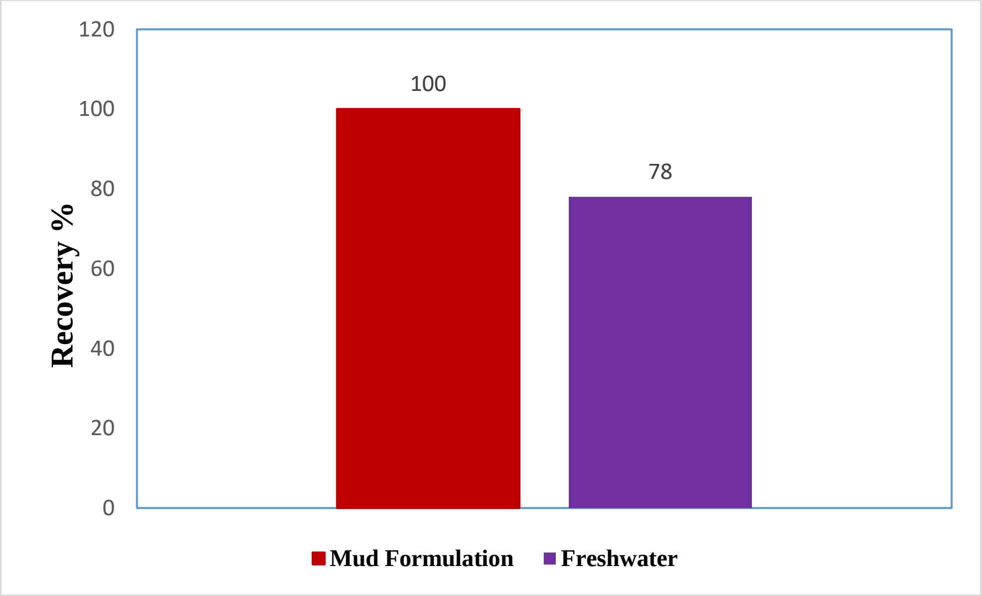


Fig. 18. Effects of freshwater and inhibitive water-based mud on shale recovery performance.

4. Conclusion`

The objective of this work was to investigate whether the combination of anti-oxidants, thermal stabilisers, and pH control agents could stabilise biopolymer (diutan gum) in water-based drilling fluids and inhibit shale dispersion at high temperatures. The additives were screened to ascertain if they could raise the 16-hour stability temperature of the biopolymer in the presence of buffering agents. The following conclusions were made:

1. All the mud formulations exhibited pseudo-plastic shear thinning and thixotropic characteristics.
2. Diutan gum stability temperature in bentonite water-suspension after dynamic aging for 16 hours was 115°C.
3. The stability temperature of diutan gum increased in the presence of the mud additives from 115°C up to 232°C. The additives were, therefore, able to counteract oxidative and hydrolytic degradation processes in the mud formulations at high temperatures.
4. From this work, water-based fluids containing clay and biopolymers can be designed for high-temperature operations in the 120-232°C range without using synthetic polymers, which are expensive and cause formation damage.
5. The shale rock aged in freshwater disintegrated and dispersed. In contrast, the shale rock aged in the drilling fluid formulated with the additive package remained virtually the same, thus showing that the additives used in this work prevented shale-fluid interaction.
6. In the shale dispersion test, the recovery rate of shale cuttings from the inhibitive fluid formulation was 100%, meaning that the additives used in this work can inhibit shale dispersion very effectively.

Given the above, the additives used in this work may be used to stabilise water-based muds at high temperatures and to formulate water-based fluids for use in reactive shale formations.

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