



DOI: 10.5604/01.3001.0053.6920

Rheological studies of high-density fracturing fluid using high-density brine and carboxymethyl hydroxypropyl guar (CMHPG) for high-temperature well condition

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ABSTRACT

Purpose: This work investigated carboxymethyl hydroxypropyl guar (CMHPG) as a potential base polymer for a fracturing fluid used in deep tight gas wells, synonym to high-temperature reservoirs. Fracturing is a common method to improve oil production by creating fractures in potentially productive reservoirs using fracturing fluids. Fracturing fluids are typically composed of water, sand, proppant, and many different additives to alter the capabilities of the fluid.

Design/methodology/approach: A stable high-density fracturing fluid (HDF) was developed to cope with scorching good temperatures up to 350°F (177°C) as well as to reduce the surface treating pressure. The HDF consists of NaBr heavy brine, CMHPG, clay stabilizers, and zirconium and borate crosslinkers. A linear gel fluid was prepared by combining NaBr brine with CMHPG before adding the additives. The resulting fluid was then tested using a high-pressure, high-temperature (HPHT) rheometer.

Findings: The results showed that the performance of the HDF was optimum in an alkaline environment between pH 10 to 12. The efficiency of zirconium and borate crosslinkers were optimum at 0.7 ppm and 1.5 ppt, respectively. Concentrations higher than these values are not only uneconomical but will cause the fluid to be overly crosslinked, consequently reducing efficiency. In actual field operation, this is disastrous when the fluid does not flow to the fracturing column but instead swirls around the drill string as the drill string rotates. On the other hand, a reduction of concentration below the optimum values can cause fluid instability at high temperature reservoirs.

Research limitations/implications: The study was limited to using two different kinds of crosslinkers and various concentrations. Future studies can be conducted using other kinds of crosslinkers, as well as an investigation into the effects of varying temperatures, pressures, and pH on the HDF using CMHPG.

Practical implications: It can be concluded that HDF using CMHPG as the base polymer can be a potential use in formulating fracturing fluids.



Originality/value: Information on the rheological behaviour of HDFF using NaBr and CMHPG can provide a reference point for future scientists in developing a new formulation of fracturing fluid.

Keywords: Fracturing fluid, CMHPG, Rheology, Crosslinker, NaBr

Reference to this paper should be given in the following way:

R.F.W. Raja Idris, N.R. Rosli, E. Mohammadian, N. Hasan, Rheological studies of high-density fracturing fluid using high-density brine and carboxymethyl hydroxypropyl guar (CMHPG) for high-temperature well condition, Archives of Materials Science and Engineering 120/2 (2023) 49-59. DOI: <https://doi.org/10.5604/01.3001.0053.6920>

PROPERTIES

1. Introduction

High-temperature deep tight gas-producing zones, such as oil fields in the Middle East, require a stable high-density fracturing fluid (HDFF) to cope with their scorching well temperature that ranges from 275 to 400°F [1,2]. A stable HDFF depends upon parameters such as the viscosity of the fluids, the concentration of each of the additives (cross-linker, breaker, etc.), shear rate and temperature. Keeping the viscosity low during the pumping procedure is vital to minimize the pumping power. In contrast, high-viscosity fracturing fluid is needed at the bottom hole to delay the degradation of the fluid when high latent heat acts on the reservoir [3].

The formation temperature and pressure are two vital parameters that affect many aspects of the fracturing process. High pressure and high-temperature conditions are expected in deep-depth well situations, posing issues such as incompatible fracturing fluids [4,5]. Fracturing fluids are typically composed of water, sand, proppant and many different additives to alter the capabilities of the fluid [6,7]. Furthermore, certain formation exhibits permeability lower than 0.01 mD, further aggravating the situation [8]. Therefore, an HDFF needs to be formulated to manage this complicated situation. This issue can be solved using high-density brines.

As wells are drilled deeper, the need and demand for fracturing fluid from high-density brine derivatives increase exponentially due to its unique adaptability in high-pressure conditions. Two types of pressures, bottom hole pressure (BHP) and hydrostatic pressure, need to be taken into consideration in the fracturing process. These two pressures will be the parameters that will affect the characteristics of the brine. Brine density should be selected accordingly so that the pressure of the brine will fall in the range provided by the parameters.

Since pressure is directly proportional to depth, high-density brine is needed in deep, high-pressure wells. If the brine density exceeds hydrostatic pressure, the formation

will not crack. On the other hand, if it exceeds the bottom hole pressure, it might cause serious damage to the formation obtained [9-11]. The selection of the types of salts is vital to the targeted density. Although zinc bromide exhibits high maximum density, it is not economically attractive for large-scale applications [12]. Calcium bromide can also be a candidate salt; however, it causes the precipitation of calcite scales, which then causes formation issues. Therefore, sodium bromide (NaBr), with a maximum density of 12.6 ppg, was selected due to its acceptable density and stability under harsh conditions compared to calcium bromide.

Guar gum, a naturally occurring polymer, is typically used to increase the viscosity of the fluid. Guar gum powder swells when it is in contact with water, increasing the fluid viscosity due to polymer hydration [13]. Two examples of guar gums that are typically added in fracturing fluids are hydroxypropyl guar (HPG) and carboxymethyl hydroxypropyl guar (CMHPG). HPG is more stable than normal gum and has been proven operative in elevated well-temperature conditions. It is a guar derivative, containing at most 2 to 4% of insoluble residue. CMHPG contains additional impurities that enhance the viscosity of the HDFF. The impurities add extra stability to this double derivatives guar under high-temperature conditions due to the presence of zirconate crosslinkers [14-16].

The concept of crosslinkers is to create links between the bonds, which increases the molecular weight of a particular polymer and consequently increases the viscosity of the fluid. The crosslinkers used in this work are zirconate and borate. These crosslinkers are governed by parameters that affect their performance, such as fluid temperature, pH readings, shear rate and concentration of reactants/polymer loading. The borate crosslinker acts as a fast-reacting crosslinker, while the zirconate crosslinker, which is a d-block element, works as a delayed crosslinker. Both types of crosslinkers are crucial in the stimulation operation such as fracturing; however, their activation temperatures must be determined and confirmed experimentally [17-19].

2. Methodology

The methodology is divided into several steps, as shown in Figure 1. The specifications of the HDFF were selected prior to the formulation of the fluid. The steps involved brine and linear gel preparation and mixing of additives such as crosslinkers, buffer solutions, and stabilizers. The resulting fluid was tested using a High-Pressure High Temperature (HPHT) rheometer and analysed in terms of its viscosity.

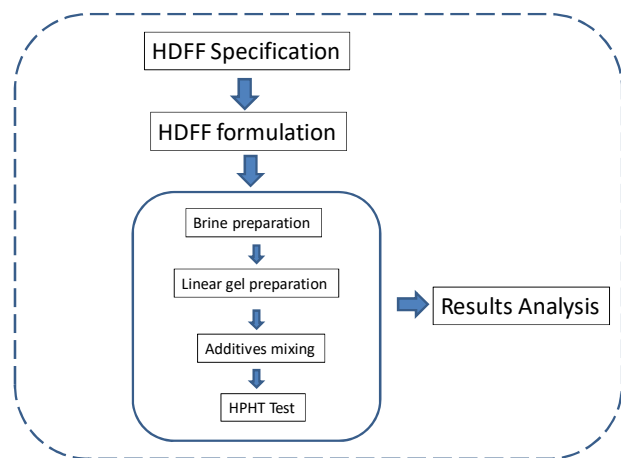


Fig. 1. Flow diagram of the overall experimental process

2.1. Materials preparation

The chemicals involved in this work are listed in Table 1, consisting of salt, polymer, and additives typical of the fracturing fluid formulation [20].

A high-density brine was prepared with NaBr in deionized water at room temperature. The density of the brine solution was tested using a densitometer in units of lb/gal (ppg).

Linear gel viscosity is an important criterion of a fracturing fluid. It is the initial viscosity of the fluid before it is mixed with cross-linkers and other additives. A linear

gel consists of a pure polymer solution in water or brine. The ionic effect of the added salt only showed an insignificant change in the linear gel behaviour [21,22].

The linear gel, also known as the base gel, was prepared by placing 250 mL of the prepared 11.5 ppg NaBr brine into a clean blender. The required amount of polymer (CMHPG) and acid buffer (10 wt.% acetic acids) was added to the brine solution. The mixture was then sheared at a constant low speed for about 5 minutes to hydrate the linear gel. After the linear gel was hydrated, the viscosity of the base gel was measured using a rheometer at 100 rpm and 300 rpm rotational speeds. The pH of the linear gel was also recorded.

A required amount of tetramethyl ammonium chloride was added to the linear gel as the clay stabilizer, oxygen scavenger, and high-temperature stabilizer. The crosslinkers (zirconate or borate) were then added to the linear gel. The borate crosslinker was prepared by dissolving the boric acid powder in 6 ppm of deionized water. Finally, both crosslinkers were added simultaneously into the mixture. The pH, vortex closure, and hang lip time were recorded. Vortex closure is the time when the vortex inside the blender starts to close down as soon as these two crosslinkers are added. Hang lip time corresponds to the amount of time the fracturing fluid is transferred from one beaker to another [23].

2.2. HPHT tests and experimental matrix

The fracturing fluid was tested using an HPHT Rheometer. The tests started at room temperature condition at 78°F (25°C). The temperature was then increased to the required test temperature at 350°F (177°C). A pressure of about 400 psi was applied, and shear ramps were conducted as per the test requirement and API RP397. A B5 bob was used for the tests at the following shear ramping: 118 rpm (100 s⁻¹), 88.5 rpm (75 s⁻¹), 59 rpm (50 s⁻¹), 29.5 rpm (25 s⁻¹), 59 rpm (50 s⁻¹), 88.5 rpm (75 s⁻¹), 118 rpm (100 s⁻¹). The stir rate interval was set at 118 rpm (100 s⁻¹) for 23.5 minutes between each ramping sequence.

Table 1.

List of chemicals

Type of chemical	Functions / Descriptions	Amount
NaBr	High-density brine	11.5 lb/gal
CMHPG	Guar gum (polymer)	60 ppt
10 wt.% acetic acid	Buffer	1, 2 ppm
NaOH-based stabilizer	High-temperature stabilizer	1, 4, 4.5 ppm
Gel stabilizer	Oxygen scavenger	18 ppm
Tetramethyl ammonium chloride	Clay stabilizer	2 ppm
Zirconium powder	Zirconate crosslinker	0.7, 0.6, 2.0 ppm
Boric acid powder	Borate crosslinker	33.39, 1.623, 1.5, 1.2, 1.0 ppt

The minimal criteria for an HDFF to be implemented on the field operation are to sustain above or on the targeted temperature of about 350°F for more than 60 minutes above 100 cp [24]. The value of 100 cp is the lower viscosity limit that an HDFF should have because any viscosity subpar to 100 cp will lose its ability to sustain the proppant inside and leave it scattered all over the wellbore. HDFF should have moderate initial viscosity and degrade gradually as temperature rises while maintaining stability above the lower limit. Too high initial viscosity does possess a high chance for the Weissenberg effect to take place. Weissenberg effect will cause the fluid to swirl up and cut the efficiency of fracturing the well [25,26].

Tests were conducted for eight different formulations, as shown in Table 2. The amount of clay stabilizer, CMHPG, and oxygen scavenger was kept constant for all formulations. Formulations A and B were conducted to compare the effect of borate crosslinker concentrations on the performance of the fracturing fluid. Formulations C and D compare the effect of pH (acidity) on the fluid's performance. Zirconium crosslinker concentrations were varied in formulations F, G, and H. The NaOH additive acted as a high-temperature stabilizer.

Table 2.
Fracturing fluids formulations

HDDF Formulation	Zirconium crosslinker, ppm	Borate Crosslinker, ppt	NaOH, ppm	10 wt.% acetic acid, ppm
A	2	33.39	1	2
B	2	1.623	1	2
C	0.7	1.5	4	2
D	0.7	1.5	4	1
E	0.7	1.2	4.5	2
F	1.5	1.5	2	2
G	1	1.5	3	2
H	0.6	1.5	4	2

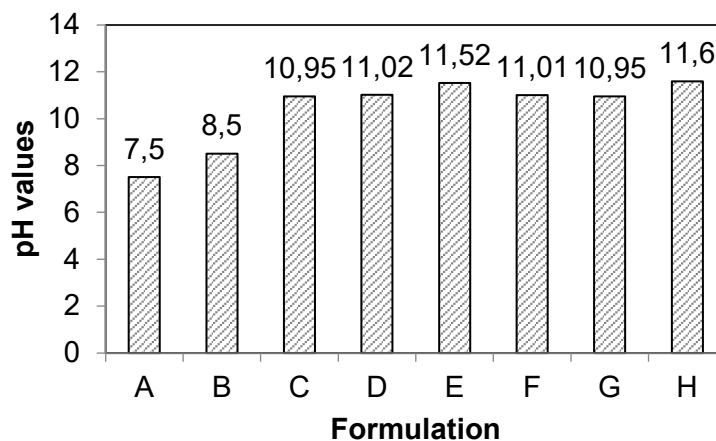


Fig. 2. pH of formulations after crosslinked

3. Results and discussions

3.1. General mechanics of HDFF under high-temperature tests

The pH values of the crosslinked fluids are tabulated in Figure 2. Formulation A exhibited the lowest pH value of 7.5, while Formulation H was the most alkaline fluid with a pH of 11.6. Formulation A has the highest borate crosslinker concentration. Borate crosslinkers react with hydroxyl groups on the CMHPG polymer backbone to create a viscoelastic fluid which is shear thinning and has good thermal stability, as shown in Figure 3 [27]. This is probably the factor that causes the low pH reading found in Formulation A.

The viscosity behaviours versus the time of five selected fluid formulations A, B, C, D, and E are shown in Figure 4, while the viscosity trend for formulations F, G, and H will be discussed separately. The temperature change is also illustrated in this figure. Details for each of the respective formulations displayed on the figure are enlightened in the following paragraphs.

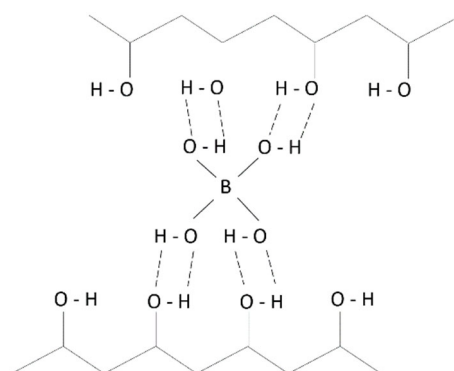


Fig. 3. Borate ion crosslinking with the hydroxyl group of the CMHPH polymer chains

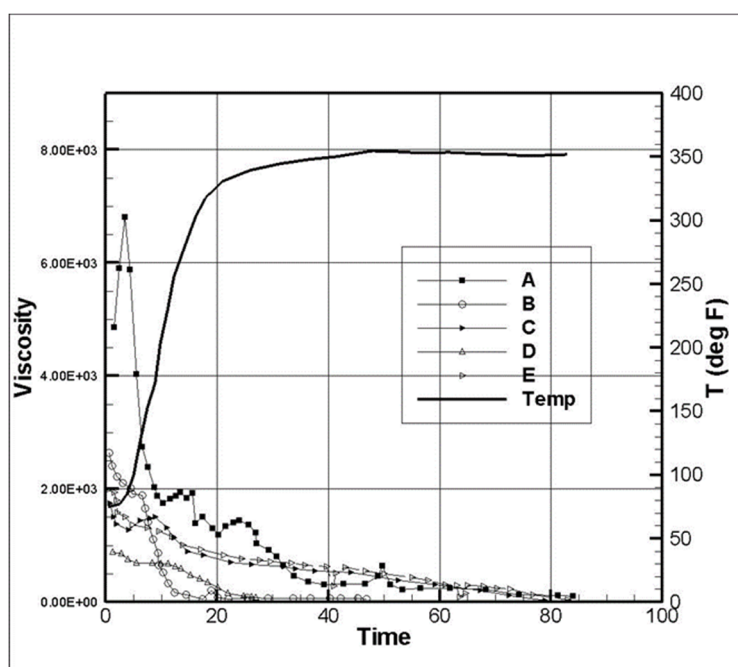


Fig. 4. Viscosity, in centipoise, of five different HDFF formulations

Formulation A displayed the highest viscosity spike up to 6872 cp during the first 5 minutes of the experiment. The viscosity immediately dipped to approximately 1900 cp around 18 minutes, gradually decreasing to zero after 55.8 minutes. The viscosity spike in this formulation indicated fast activation of the crosslinker, but it went down rapidly due to an unsuitable pH value. The measured pH was 7.5, below the optimum range for crosslinking activities, from pH 10 to 12.

The prime problem for this formulation is the excessive amount of borate crosslinker added to the recipe. Borate crosslinker activated in medium range temperature, between 150 to 200°F, as shown in the initial surge of viscosity spike

in this run. Excessive concentration of crosslinker caused the occurrence of the Weissenberg phenomenon, which made the fluid climb up the bob, leaving no fluid at the bottom of the metal tube in the HPHT rheometer. This consequently resulted in the declination of its viscosity.

The initial viscosity surge behaviour in Formulation A was not observed in Formulation B. This is due to the huge decrease in the amount of borate crosslinker from 33.39 ppt to 1.623 ppt. However, the viscosity life of Formulation B was the shortest among the rest of the formulations due to the unsuitable pH, leading to an insufficient crosslinking mechanism. The concentration of borate crosslinker is directly proportional to the initial viscosity of the solution.

Therefore, the reduced borate concentration resulted in lower initial viscosity. As a fast-reacting crosslinker, the borate crosslinker kicked in at the early stage of the run, observed at low-temperature conditions (between 80 to 110°F). This proves that a high amount of borate crosslinker is unnecessary because high initial viscosity is unsuitable for actual field operations. This is because pumping high-viscosity fluid into the wellbore is inefficient due to the high horsepower consumption pushing the fluid downward.

Formulation C exhibited the best overall result. It did not have a tremendous surge of viscosity in the early stage, but it constantly supplied enough strength to keep the fluid together for more than 60 minutes above 100 cp. The Borate crosslinker was initiated as early as the 14th minute, and the zirconium crosslinker tapped in approximately at the 32nd minute. The concentrations of both crosslinkers are regarded as the most favourable concentration because problems of the Weissenberg effect were eliminated, exhibited longer fluid stability, and the fluid was able to be unbroken for a longer period. Weissenberg effect was not observed in this formulation, as shown in the absence of a viscosity drop to 0 cp.

Formulation D was carried out to determine the optimum amount of acetic acid and the effect of pH higher than 11. In this run, the amount of acid buffer solution (10% acetic acid) was reduced from 2 ppm to 1 ppm, therefore creating a more alkaline condition. The fluid in Formulation D was visibly watery even before it was brought to the HPHT rheometer for high-temperature testing. Even though the recipe for Formulation D is almost similar to the best formulation, Formulation C, the results produced were contradictory. The fluid tested in this formulation did not show any stability even at the initial stage of the run, and the time taken for the curve to dip below the lower limit was at the 26th minute, which was the fastest after Formulation B. Weissenberg phenomenon was not observed because the overall viscosity of this formulation is too low for the fluid to climb up the bob. The reduction of acetic acid caused the fluid to approach the point of neutralization. The fluid without any acetic acid added to it was basically a weak acid in the pH range of 6 to 6.5. Any increment of addition of acetic acid will steer the pH towards the stronger acidic level and vice versa. Approaching the neutralization point, there is a possibility that the amount of COOH⁻ ions and Br⁻ ions swarm the binding molecules causing an increase in the repulsion of the fluid molecules. This repulsion is greater than the Van der Waals forces that keep the molecules together, leading to breaking-off of the molecular binds and chains. CMHPG was used as the polymer, along with crosslinkers, to bind the molecules. As the CMHPG was brought upon contact with water/brine, each micelle in the

component swells. Incompatibility developed from the unstable concentration between the mixture of the weak acid and guar causes the anions to accumulate in the fluids, thus reversing the swelling of the micelles and subsequently reducing the viscosity of the tested fluid [28]. Besides that, kinetic parameters such as cyclization kinetic, crosslinking kinetic and crosslinking size could play a certain role at this point. Watery and low-viscosity fluid is closely associated with the cyclization process because of the inability of the fluid to be fully crosslinked at a cyclic state [29].

The amount of borate crosslinker was reduced in Formulation E compared to Formulation C. This formulation exhibited better behaviour as compared to Formulation D because it was able to maintain its viscosity for a longer period. Several dips were witnessed in the graph, which corresponds to the concentration of the insufficient crosslinker. Borate crosslinkers obviously helped promote the viscosity, as shown in the early stage of the graph, while zirconium crosslinkers kicked in at the later part of the test. The high initial viscosity of a fracturing fluid will stay longer than the lower limit compared to the low initial viscosity fracturing fluid. Initial viscosity is closely related to borate crosslinkers, and the relationship between both parameters is directly proportional to each other. Increasing temperature possibly degrades the fracturing fluid by supplying enough latent heat in the form of kinetic energy to increase the molecular vibrations between the molecules resulting in the breaking of the bonds between particles. Apparently, the excellent pH value in this run activated the zirconium crosslinkers, and the spike of viscosity is clearly displayed on the graph at the 40th minute. The dip tended to show up in this curve again, as it appeared at the 63rd minute. The dips at the 63rd and 85th minutes were not due to Weissenberg nor crosslinker activation but because of very low viscosity.

3.2. Effect of zirconium crosslinker in HDFF

Several runs were selected to portray the effect of zirconium on the high-density fracturing fluid formulation. Based on Table 3, Formulations C, F, G, and H were chosen to compare and contrast the experimenting parameters at different zirconate crosslinkers concentrations. Both borate crosslinker concentration and 10% acetic acid concentration were kept constant to emphasize the effect of zirconium on fracturing fluid purely.

The pH values for each of these runs were in the range of 10.3 to 11.6, the optimum pH range for a decent crosslinking mechanism to take place, as precisely showcased in Figure 5.

The results of these four formulations are shown in Figure 6. The highest amount of zirconium crosslinker was

Table 3.

Effects of zirconium crosslinker on HDFF pH and rheology

HDFF Formulation	Zirconium crosslinker, ppm	Borate crosslinker, ppt	10wt% acetic acid, ppm	pH after cross-linked
C	0.7	1.5	2	10.30
F	1.5	1.5	2	11.01
G	1.0	1.5	2	10.95
H	0.6	1.5	2	11.60

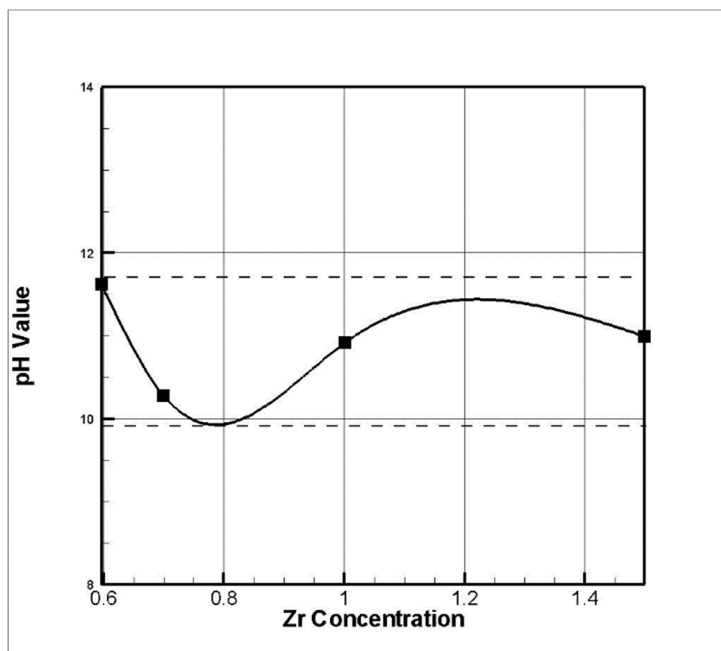


Fig. 5. HDFF pH after crosslinked versus concentration of zirconium crosslinker

added in Formulation F, which was 1.5 ppm. The figure illustrates a presentable curve which lasted more than 60 minutes above the lower limit. The prime problem with formulation F was that the drop hit the floor of the lower limit before it went back up again due to the activation of crosslinkers. Weissenberg effect is the major suspect because a bob climbing event tends to occur if the crosslinker concentration exceeds what it requires. This formulation could be verified as an overly crosslinked fluid because huge leftovers of the fluid remained hanging on the top of the bob after the testing was finished.

As the zirconium crosslinkers were reduced from 1.5 to 1.0 ppm in formulation G, the graph still showed instability in improvising the fracturing fluid's viscosity. The fluid was presumed to degrade and sink below the lower limit due to the unfavourable ratio between crosslinkers. The Weissenberg effect is the main cause in this formulation because the initial viscosity of the fluid was too high in the beginning causing the fluid to climb up the bob and lead to a severe viscosity drop that hit the lower limit (100 cp) of an

HDFF as mentioned in the criteria section. The fluid viscosity tended to rise back up after the fall due to the increasing test temperature in HPHT because the fluid which was on top of the bob was experiencing degradation as the result of the breaking of chains and bonds of the fluid and hence reduced the viscosity flowing downward back into the metal cup. As soon as the fluid travelled downward, the zirconium crosslinker kicked in, and a small surge of viscosity could be witnessed from the graph. This surge was ample to make the fluid last more than 60 minutes however, this formulation was still a failure because the Weissenberg effect did take place, and the viscosity dropped to zero.

Further decrement of zirconium crosslinkers was made in Formulation C, 0.7 ppm was added, and the result showed a superb viscosity curve. The curve viscosity life was more than 60 minutes; therefore, it was predicted to activate approximately at 280°F. The blend between the two crosslinkers was perfect since there was no dip in the viscosity and the crosslinker added was enough to preserve the curve above the lower limit.

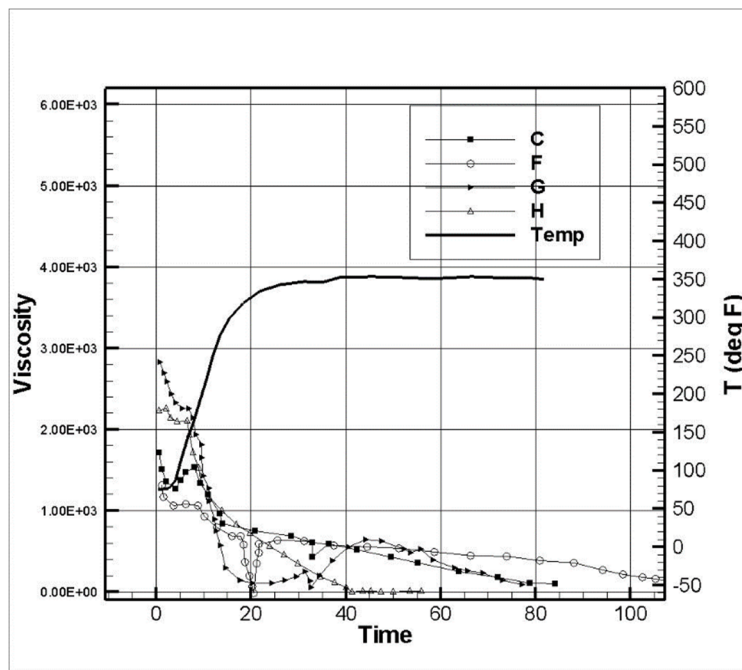


Fig. 6. Viscosity behaviour of formulations C, F, G, and H versus time

Table 4.

Comparison of high resistance fracturing fluids after at least 60 s

Works	Temperature, °C	Apparent viscosity, cp	Base fluids and crosslinkers	Shear rate, sec ⁻¹
Formulation C	177	100	NaBr, CMHPG, Zr, Br	25 to 100
Saini & Mezei, 2017 [30]	85	10000	Produced water, guar gum	23 to 1000
Yang Shao et al., 2020 [31]	130	100	AANM	170
Al-Driweesh et al., 2014 [32]	168	100	KCl, CMHPG, Zr, Br	40
Hang Xu et al., 2022 [33]	150	50	Guar gum, Br	170
Yang Zhen Zhou et al., 2018 [34]	230	130	GAHT, Zr	100
Liang Feng, et al., 2020 [35]	204	500	Field water, Zr	40

Formulation C was compared with Formulation H, which had 0.6 ppm zirconium crosslinker. Although the difference between these two formulations was just 0.1 ppm of zirconium crosslinker, it brought huge changes to the fluid's behaviour. Since this experiment used 250 mL of fracturing fluid, the difference could be considered great, and 0.6 ppm of zirconium crosslinker was absolutely insufficient to bind the molecules in the breaking-up phase, therefore, slowing the process of degradation under high-temperature conditions.

In a nutshell, zirconium plays an important role in preserving the stability of the fluid under high-temperature conditions. As a delayed crosslinker, it must be added in an optimum amount to deliver the best result to the fracturing fluid. The optimum amount of zirconium crosslinker in this experiment was observed at 0.7 ppm. A concentration higher than 0.7 ppm will cause the fluid to be overly crosslinked,

consequently allowing it to climb up the bob and reducing the fracturing fluid's efficiency. An excessive amount added during the actual operation on the fluid is not only uneconomical but disastrous because the fluid will not flow to the fracturing column but instead swirl around the drill string as the drill string rotates. On the other hand, a reduction of concentration below 0.7 ppm can cause fluid instability under high temperature conditions. Under high-temperature conditions, HDFF needs aid from the crosslinkers to bind the molecules as the scorching temperature breaks the molecular binds and chains. An insufficient amount of crosslinker is not beneficial as it cannot slow down the degradation process. 0.7 ppm of zirconium crosslinker is the optimum concentration to negate any field-related problems and thus creates the best possible HDFF to meet the objectives of this experiment.

3.3. Comparison of similar HDFF formulations

Many other researchers share a similar interest in developing fracturing fluids suited for the ever-challenging oil fields as they venture into harsher environments. Table 4 presents a summary of similar works.

4. Conclusions

The HDFF formulation C, which consisted of 0.7 ppm zirconium crosslinker, 1.5 ppt borate crosslinker, and 2 ppm of 10 wt.% acetic acids, was considered the most favourable formulation in this study. This formulation illustrated good balance in terms of overall stability towards increasing temperature over time. The HDFF was able to sustain its apparent velocity above the targeted temperature of about 350°F for more than 60 minutes above 100 cp [22]. The formulation possessed moderate viscosity and degraded gradually as temperature rose and maintaining to be stable. Weissenberg effect was not detected, and the fluid was unbroken for longer due to good mixing between the fast-reacting (borate) and delayed (zirconate) crosslinkers. Zirconate and borate crosslinkers were needed together, and one could not work at the optimum conditions without the other. Both crosslinkers worked best in medium and high-temperature ranges. However, the focus should be emphasized on the high temperature range. Certain polymer starts to degrade at high temperature and immediately reduces the viscosity of the fluid, which will lead to failure in sustaining the proppants.

Acknowledgements

Special thanks to staff from Schlumberger WTA Malaysia for the facilities, help and assistance given to complete this study. This research was partially funded by the Fundamental Research Grant Scheme (FRGS) 600-IRMI/FRGS 5/3 (085/2019) provided by the Ministry of Higher Education Malaysia.

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