FULL LENGTH ARTICLE

Study the effect of synthesized graft copolymer on the inhibitive water based drilling fluid system

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Graft copolymer; Drilling fluid; Rheological properties; Shale recovery

Abstract This research paper consists of the synthesis of carboxymethyl-graft-polyacrylamide copolymer by free radical polymerization technique and its characterization using Fourier transform infrared spectroscopy (FTIR), field-emission scanning electron microscopy (FESEM) and thermogravimetric analysis. This graft copolymer was used as a drilling fluid additive and its effect on the Indian reactive shale sample was analyzed. The characterization of the shale sample used in this study was done by X-ray diffraction technique (XRD), FTIR, FESEM, and energy-dispersive X-ray spectroscopy (EDX) to determine the presence of various clay minerals. Experimental investigations revealed that the synthesized graft copolymer has a significant effect on the rheological and filtration properties of the inhibitive drilling fluid system and has high shale recovery performance. Hence, inhibitive drilling fluid system using synthesized graft copolymer may be used for the drilling of water sensitive shale formations.

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1. Introduction

The drilling fluid which slows or stops the hydration, swelling and disintegration of shales is called inhibitive drilling fluid. In the inhibitive water based drilling fluid system the salt concentration is kept above 10,000 ppm. It consists of salt, synthetic and natural compounds to serve the purpose of drilling the oil/gas wells safely and efficiently. This type of aqueous drilling fluid system is used to inhibit the swelling tendency of the water sensitive geological formations like shale, mudstones, claystones etc, thus, providing minimal reactivity of the drilling fluid with the borehole. Apart from inhibitive function of this mud system, it has to perform other functions such as the circulation of cuttings, bore hole cleaning, pressure maintenance inside the wellbore, cool and lubricate the drill bit and to smoothen the functioning of drilling strings [1–5].

Several types of organic polymers are used to control the rheological and filtration properties of drilling fluid systems [6–8]. Among these, natural polymers are biodegradable, fairly shear stable, and cause drag reduction at relatively higher concentration. The synthetic polymers are much more effective than the natural polymers due to their versatile flexibility. But, these polymers are not shear resistant and hence, natural
polymers like xanthan gum are generally preferred to control rheological properties of water based drilling fluid system. To control the filtration characteristics of drilling fluid, semi-synthetic and biopolymers like carboxy methyl cellulose (CMC), polyanionic cellulose (PAC), starch etc are widely used [9–11].

Many industries use polyacrylamide to mitigate wellbore instability problems arising due to swelling & dispersion of shale and the cuttings inside the wellbore because of its unique property of shale encapsulation. The probable mechanism is based on the adsorption of the polymer on the reactive clay sites present in the shale. The adsorption of the polymer is increased in the presence of potassium chloride (KCl) salt, which also aids in the inhibition of swelling tendency of shale [12–15].

Sodium carboxymethyl cellulose (CMC) is a linear, long chain, water-soluble, anionic polysaccharide produced by reacting alkali cellulose with sodium monochloracetate which is used generally in the development of different drilling fluid systems. The various properties of the CMC depend on the distribution of carboxyl substituents along the polymer chain, molecular weight of the polymer, and average number of carboxyl content per anhydroglucose unit [16,17]. The rheological and filtration properties of the CMC solutions depend on many factors like concentration of polymer, temperature, pH, salt content, total degree of substitution of the polymer, molar mass, and presence of the surfactant [18,19].

To overcome the difficulties faced during the application of synthetic polymers, the grafting of synthetic polymers on the natural polymers is suggested by many researchers [20–22]. The graft copolymers have relatively high shear stability as grafting improves the shear stability during the turbulent flow [23]. Grafting of natural polysaccharides and synthetic polymers such as polyacrylamide has been reported earlier for different applications [24,25].

In the present study, CMC-g-PAA graft copolymer is synthesized in an aqueous medium using potassium persulfate (KPS), an inorganic compound with the formula K2S2O8 as initiator. The free radical polymerization process took place in nitrogen gas (N2) atmosphere. Then, the synthesized graft copolymer is used to develop an inhibitive water based drilling fluid system and calcium carbonate was used as a bridging agent to minimize the API fluid loss volume in this drilling fluid system. In addition, mineralogical studies are carried out on the shale sample to determine the presence of reactive clay minerals in the shale. Then, the shale recovery tests are done to analyze the performance of the graft copolymer on the shale recovery.

2. Experimental

2.1. Materials

Acrylamide and potassium chloride salts were procured from the Merck Pvt. Ltd., Mumbai, India. Low viscosity grade CMC and calcium carbonate were obtained from the CDH, chemicals Ltd., New Delhi, India. Low viscosity grade polyanionic cellulose and xanthan gum were procured from the ONGC, India. Potassium persulfate was purchased from Qualigens, Mumbai, India. The shale sample was collected from Damodar valley basin, India.

2.2. Synthesis of the graft copolymer (CMC-g-PAA)

The graft copolymer of CMC and PAA has been synthesized in an inert atmosphere of nitrogen using potassium persulfate (KPS) as initiator. The procedure for the synthesis of the graft copolymer is as follows: 2 g of CMC was slowly dissolved in 200 ml of double distilled water in 500 ml 3-neck round bottom flask. The flask was kept in an oil (silicon) bath maintained at a temperature of 68 ± 1 °C fitted with an electrically operated magnetic stirrer (Tarsons, Model-Spinot Digital), for constant stirring. At this stage, 20 ml acrylamide solution of the desired concentration (14.28 M) was poured into the flask, acrylamide was allowed to mix with CMC solution and continued to heat at the same temperature for 3 h at 550 rpm. After proper mixing of the acrylamide with CMC, 5 ml of potassium persulfate solution (9.25 × 10⁻³ M) was added slowly and the reaction was continued for another 1.5 h at same stirring speed and temperature. The nitrogen gas was purged continuously during the reaction. The CMC is water soluble and it has imparted viscosity to the solution. Hence, sufficient time for mixing of acrylamide with CMC solution was given so that the resulted aqueous solution would be a homogenous solution. The reaction mixture was cooled at room temperature (25 °C), it was separated by precipitation using acetone. The solid product was filtered and washed with ethanol and then extracted using acetone as solvent in a Soxhlet apparatus for the removal of impurities [26,27]. Afterward, the graft copolymer was dried in a hot air oven at 50 °C for 24 h, pulverized by mortar–pestle. Then, the synthesized graft copolymer was used to develop inhibitive water based drilling fluid system.

2.3. Characterization of graft copolymer

2.3.1. FTIR analysis

FTIR spectra of various samples were recorded in PerkinElmer model Spectrum Two (USA) and range of the above measurements was 450–4000 cm⁻¹. 1 mg of the sample was mixed with a very small amount of KBr and KBr pellet was prepared using a hydraulic press by applying a pressure of 100 psi for 60 s. The IR spectrum of the KBr pellet is recorded and 100 scans were collected.

2.3.2. FESEM analysis

The morphology of the various samples used in this study was analyzed by FE-SEM Supra 55 model, Carl Zeiss (Germany) with Air Lock chamber to examine the morphology of different samples. Samples were charged with platinum coating to get clearer images.

2.3.3. Thermal analysis

Thermal stability of the graft copolymer, and CMC was examined using thermogravimetric analysis (TGA) in Netzsch-STA 449 Jupiter (Germany). TGA analysis was performed up to 370 °C in a nitrogen gas atmosphere with a heating rate of 10 °C/min.

2.4. Characterization of the shale sample

2.4.1. XRD analysis

The shale sample was characterized by X-ray diffraction (XRD) analysis to determine the qualitative mineral content
in the sample. XRD analysis was carried out in PANalytical X'Pert PROcrib Tellus X'Pert PRO (Spectris plc, UK) diffractometer. XRD patterns were recorded at 45 kV and 200 mA.

2.4.2. FTIR
The FTIR spectrum of the sample was recorded at the PerkinElmer, model Spectrum Two (USA) and range of the above measurements was 450–4000 cm\(^{-1}\).

2.4.3. FESEM analysis
Field emission scanning electron microscope (FESEM) analysis was carried out in FE-SEM Supra 55 model Carl Zeiss, (Germany) with Air Lock chamber to examine the morphology of different samples. Samples were charged with platinum coating to get clearer images.

2.5. Development of inhibitive water based drilling fluid system

To develop the inhibitive water based drilling fluid system, the desired amount of graft copolymer was added to the base fluid system prepared with potassium chloride (KCl) salt, xanthan gum and polyanionic cellulose. All the constituents were mixed thoroughly using Hamilton Beach Mixer. The rheological & filtration properties of the developed homogenous drilling fluid were measured as per American Petroleum Institute (API) recommended procedures. The rheological properties were analyzed by using Fann VG meter, model 35, Fann Instrument Company (Houston, Texas). Instrument constants have been adjusted so that plastic viscosity, apparent viscosity and yield point are obtained by using readings from rotor sleeve speeds of 300 rpm and 600 rpm. The filtration properties were obtained by using Fann API filter press. Afterward, same drilling fluids were again prepared with calcium carbonate and rheological & filtration properties were again analyzed. The following formulas were used to obtain the rheological parameters as per API recommended practice of standard procedure for field testing of drilling fluids:

- Apparent viscosity \(\mu_a = \frac{\Theta_{600}}{2}\) (mPa-s)
- Plastic Viscosity \(\mu_p = \Theta_{600} - \Theta_{300}\) (mPa-s)
- Yield Point \(\mu_y = (\Theta_{500} - \mu_p) \times 0.5\) (Pa)

Thermal stability of the developed drilling fluid system was determined by aging it in a hot air roller oven for 16 h at 95 °C temperature. The rheological and filtration properties of the inhibitive water based drilling fluid system after hot rolling were again measured using API, recommended procedures.

2.6. Shale recovery test with the synthesized graft copolymer

To analyze the effect of various concentrations of the synthesized graft copolymer on the shale recovery performance, the shale sample was grounded and sieved to retain a suitable mesh fraction after screening through 20 and 30 mesh size screen. The 50 gm \((W_1)\) of the sample was added to the developed inhibitive water based drilling fluid in a stainless steel aging cell and the system was hot rolled at 95 °C for 16 h. The retained shale was washed with clear water and dried in the oven at 60 °C. After drying in oven, the weight \((W_1)\) of the sample was recorded. Recovered shale was added to the fresh water in the aging cell for hot rolling at 95 °C for 2 h. After aging, the shale was recovered and dried in the oven at 60 °C. Then, the weight \((W_2)\) of the recovered shale was recorded again. The first shale recovery rate \((\% R_1)\) and the second shale recovery rate \((\% R_2)\) were calculated on a dry mass basis as follows:

\[
\% R_1 = \frac{W_1}{W_0} \times 100\%
\%
\]

\[
\% R_2 = \frac{W_2}{W_0} \times 100\%
\%
\]

3. Results and discussions

3.1. Synthesis of graft copolymer
Various concentrations of acrylamide, initiator and CMC were used to synthesize the graft copolymer. Different ratios (w/w) of the monomer and CMC (5:1, 6:1, and 8:1) were used to carry out the synthesis in this analysis. The higher concentration of the monomer may result in the formation of undesirable homopolymer and the higher amount of the CMC has resulted in higher viscosity of the solution which affects the grafting efficiency. The increase in the viscosity of the solution at higher concentrations of the CMC, causes hindrance in normal graft copolymerization [28,29]. The optimized concentration of the KPS was kept at 9.25 × 10^{-3} M which is quite suitable for the synthesis of the graft copolymer [30]. The probable mechanism in the synthesis of the graft copolymer is based on the generation of free radical sites on the backbone of CMC by KPS, which in turn react with the monomer (acrylamide) to form graft copolymer through initiation, propagation and termination. Finally, the graft copolymer with monomer to CMC ratio of 8:1 w/w was selected for the development of the drilling fluid system.

3.2. Characterization of the graft copolymer

3.2.1. FTIR
The infrared spectra of CMC, acrylamide, PAA and graft copolymer are shown in Fig. 1. The spectrum of CMC displays a broad absorption band at 3413 cm\(^{-1}\), a typical stretch for OH group. The band at 2924 cm\(^{-1}\) is due to stretching of CH group. The bands at 1633 cm\(^{-1}\) and 1456 cm\(^{-1}\) are for the stretching of the NH bond of NH\(_2\) group and C\(=\)O which confirms the presence of COO\(^{-}\) group. The bands around 1416 cm\(^{-1}\) and 1024 cm\(^{-1}\) are assigned to angular deformation of \(\equiv\)CH\(_2\) groups and stretching of the CH=O–CH\(_2\) group, respectively. In the case of polyacrylamide the bands at 3436 cm\(^{-1}\) and 2924 cm\(^{-1}\) are for the stretching of the NH bond of NH\(_2\) group and C–H bond stretching vibrations. The bands at 1656 cm\(^{-1}\) and 1456 cm\(^{-1}\) contribute to stretching of the C=O group of amide and CH\(_2\) scissoring, respectively. Finally, in the spectrum of CMC-g-PAA graft copolymer the peak at 3433 cm\(^{-1}\) is the due to overlapping of the hydroxyl group of CMC and amine group of PAA. A peak attributing to the stretching of CH bond is seen at 2925 cm\(^{-1}\), while an intense peak associated with the superposition of the amide I band of polyacrylamide and asymmetric stretching of C=O appears at 1665 cm\(^{-1}\). The band at 1723 is attributed to the presence of free acid groups [27].
3.2.2. FESEM

Field emission scanning electron microscopy was used to investigate the morphological features of synthesized CMC-g-PAA graft copolymer (Fig. 2). The morphological feature of the CMC reveals about its granular structure. After grafting with polyacrylamide, the granular appearance of CMC was distorted and changed to fibrillar. Also, morphology of PAA has changed after grafting with CMC. This observation suggests that grafting of polyacrylamide affects the morphological arrangement of CMC. The micrograph of CMC-g-PAA showed a number of long chains of PAA which got agglomerated and changed in fibrillar structure.

3.2.3. Thermal analysis

The TGA curves of the CMC and graft copolymer are shown in Fig. 3. In the case of graft copolymer the initial weight loss may be attributed to the presence of moisture in the sample. The first weight loss is due to the degradation of the CMC main chain in the temperature range of 245 °C–270 °C with a weight loss of 11 wt%. It should be noted that the weight loss percent of CMC is about 28 wt% which is higher than the graft copolymer in the same temperature range. It was found that the thermal stability of the polymer is increased due to grafting [28].

3.3. Characterization of the shale sample

3.3.1. XRD analysis

XRD analysis was used for the characterization of the shale sample. The labeled XRD pattern of representative sample is shown in Fig. 4. The crystalline components present in the shale are quartz (SiO$_2$) along with kaolinite, and montmorillonite mineral. The XRD spectrum of shale indicates the presence of montmorillonite, a clay mineral of smectite group at various 2$h$ positions (8.5, 12.5). Quartz low and kaolinite are also present in the sample. Goldfieldite and zinc acetate hydrate in very low quantity are also present in the sample [31].

3.3.2. FTIR analysis

In the FTIR spectrum for the shale sample (Fig. 5), the bands at 3620 cm$^{-1}$ and 3696 cm$^{-1}$ are due to the OH stretching vibrations of inner-surface hydroxyl groups, indicating about the hydrous nature of the sample and the presence of smectite, respectively. The band at 2924 cm$^{-1}$ is associated with aliphatic hydrocarbons present in the sample. The peaks at 1031 cm$^{-1}$, 912 cm$^{-1}$, 778 cm$^{-1}$, 694 cm$^{-1}$, 536 cm$^{-1}$, and 469 cm$^{-1}$ show the presence of quartz, montmorillonite and kaolinite clay minerals (Al$_2$O$_3$·2SiO$_2$·2H$_2$O) in the shale [32].

3.3.3. FESEM analysis

The FESEM image of shale indicates the presence of patches (Fig. 6), the shale is rich in quartz and clay platelets as confirmed by XRD, FTIR. Quartz and clay platelets are stacked together. In the shale sample there is irregularity in the surface with few undulations, indicating a crystalline-type order and stacking of the sheets of the material due to the presence of montmorillonite and sodium phosphate hydrate. Energy-dispersive X-ray spectroscopy (EDX) analysis shows the presence of high amount of Al, Si and oxygen in the samples. The higher amount of Al, Si, oxygen and potassium may be due to the presence of kaolinite-montmorillonite (Na$_{0.3}$Al$_4$Si$_6$O$_{18}$ (OH)$_4$·4H$_2$O) clay minerals as revealed by FTIR and XRD analysis [33].

3.4. Development of inhibitive water based drilling fluid system

3.4.1. Effect of graft copolymer on the rheological and filtration properties of inhibitive water based drilling fluid system and comparison with its substrate (CMC)

The developed inhibitive drilling fluids using different concentrations of graft copolymer are non-Newtonian in nature. The fixed concentration of potassium chloride salt (5.0 wt/v%), xanthan gum (0.3 wt/v%) and polyanionic cellulose (0.8 wt%) polymers was used as base fluid to impart initial rheological and filtration properties. Further, graft copolymer was
added to enhance rheological and filtration properties of the base fluid desired for drilling of oil & gas wells. The presence of KCl aids in the inhibitive property of the drilling fluid while drilling clay formations. The composition and various properties of base fluid are shown in Table 1. The influence of graft copolymer on the rheological and filtration parameters of the base fluid can be analyzed from Table 2. The viscosity of the base fluid has increased significantly at higher...
concentration of the graft copolymer. The effect of graft copolymer on the shear thinning characteristics of the developed fluid system can be depicted from the consistency index ‘$K$’ and flow behavior ‘$n$’ as shown in Table 3. To explain this, it is generally supposed that the large molecular chains tumble at random and affect large volumes of fluid under low shear, but at higher shear rates these gradually align themselves in the direction of increasing shear and produce less resistance. The increase in the value of ‘$K$’ is the indication of the increase in the overall hole cleaning effectiveness of the fluid. The consistency index increases with the increase in the concentration of the graft polymer. The value of ‘$n$’ is less than unity which indicates its deviation from the Newtonian behavior. The rheological properties of the base fluid increased with the increase in the concentration of the graft copolymer. The plastic viscosity which affects the rate of penetration is in low range. Apparent viscosity is also in the optimum range as it helps in the circulation of the cuttings at low shear rates in the annulus. The filtration properties of the base fluid improved with the addition of graft copolymer. The presence of the graft copolymer aids in the flocculation phenomenon which has resulted in the formation of low permeability filter cake resulting in better filtration properties. The $pH$ value of the developed inhibitive water based drilling fluid system is in the range of 9.5–10 which is favorable for various drilling operations [34].

The grafting of the polyacrylamide into the main chain of carboxymethyl cellulose polymer has resulted in better performance of the graft copolymer than its substrate. The drilling fluid system developed with synthesized graft copolymer, is compared with conventional water based drilling fluid system formulated with carboxy methyl cellulose, xanthan gum (0.3 wt/v%), polyanionic cellulose (0.8 wt/v%) and KCl (5 wt/v%). The effect of CMC on the rheological and filtration properties of the base fluid can be analyzed from Table 4. CMC has a lower impact on rheological properties as compared to graft copolymer. The values of plastic viscosity, apparent viscosity, yield point and gel strength are found to be relatively lower. Moreover, the effect of CMC on the filtrat-
The effect of synthesized graft copolymer

3.4.2. Effect of graft copolymer on the rheological and filtration properties of the inhibitive water based drilling fluid system in presence of calcium carbonate

The wellbore instability causes many problems which affect the overall drilling and completion program. The filtration properties of the drilling fluid system need to be controlled as filtrate invasion into the sensitive formations may accelerate physiochemical reactions inside the sensitive formations [35]. The presence of calcium carbonate in the system helps in preventing pressure increase near wellbore by showing its bridging effect on the face of geological formation inside the wellbore. The effect of graft copolymer in presence of calcium carbonate (3 wt/v%) on the rheological and filtration properties of the base fluid is reported in Table 5. Filtration properties of the developed drilling fluid system are controlled drastically in the presence of calcium carbonate. The calcium carbonate particles cause more colloidal particle formation by relating with other particles present in the drilling fluid when a hydrostatic head of 100 psi is applied as per API recommended procedure on the system. The quick formation of impermeable cake takes place due to the presence of calcium carbonate particles which reduces the net API fluid loss volume. Another advantage of adding calcium carbonate in the present drilling fluid system is the formation of external filter cake on the walls of pay zone inside the wellbore which can be removed easily when the well is put on production phase by various well stimulation jobs [36]. However, the rheological properties of the drilling fluid system remain unaffected in the presence of calcium carbonate.

3.4.3. Thermal stability of the inhibitive water based drilling fluid system

The various constituents of mud degrade slowly at elevated temperatures. The degradation of the constituents becomes

<table>
<thead>
<tr>
<th>Concentration of graft copolymer (wt/v%)</th>
<th>Plastic viscosity (mPa-s)</th>
<th>Apparent viscosity (mPa-s)</th>
<th>Yield point (Pa)</th>
<th>Initial gel strength (Pa)</th>
<th>10 min gel strength (Pa)</th>
<th>API fluid loss (ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.3</td>
<td>12</td>
<td>21.0</td>
<td>9.0</td>
<td>1.0</td>
<td>1.5</td>
<td>10.5</td>
</tr>
<tr>
<td>0.4</td>
<td>13</td>
<td>24.0</td>
<td>10.0</td>
<td>1.5</td>
<td>2.0</td>
<td>9.0</td>
</tr>
<tr>
<td>0.5</td>
<td>14</td>
<td>29.5</td>
<td>15.5</td>
<td>2.0</td>
<td>2.5</td>
<td>8.5</td>
</tr>
<tr>
<td>0.6</td>
<td>16</td>
<td>32.0</td>
<td>16.0</td>
<td>3.0</td>
<td>4.0</td>
<td>7.5</td>
</tr>
<tr>
<td>0.7</td>
<td>17</td>
<td>34.0</td>
<td>17.0</td>
<td>3.5</td>
<td>4.5</td>
<td>7.0</td>
</tr>
<tr>
<td>0.8</td>
<td>19</td>
<td>39.0</td>
<td>20.0</td>
<td>3.5</td>
<td>5.5</td>
<td>6.0</td>
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</table>

Table 2 Effect of graft copolymer on the rheological and filtration properties of the base fluid system.

Figure 6 FESEM image and EDX spectrum of the shale sample.

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3.4.1. The importance of carboxy methyl cellulose properties is found to be lower than the graft copolymer based drilling fluid system as the API fluid loss volume of the CMC based drilling fluid system is high which is not desirable while drilling water sensitive formations. The probable reason for this is the inability of the carboxy methyl cellulose to form a thin impermeable filter cake when a differential pressure is applied to the drilling fluid.

Table 1 Rheological & filtration properties of developed base fluid system (5% KCl, 0.3% xanthan gum, 0.8% polyanionic cellulose).

<table>
<thead>
<tr>
<th>Properties</th>
<th>Units</th>
<th>Observed values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plastic viscosity</td>
<td>(mPa-s)</td>
<td>10.0</td>
</tr>
<tr>
<td>Apparent viscosity</td>
<td>(mPa-s)</td>
<td>16.5</td>
</tr>
<tr>
<td>Yield point</td>
<td>(Pa)</td>
<td>6.5</td>
</tr>
<tr>
<td>Initial gel strength</td>
<td>(Pa)</td>
<td>1.0</td>
</tr>
<tr>
<td>10 min gel strength</td>
<td>(Pa)</td>
<td>1.5</td>
</tr>
<tr>
<td>API fluid loss</td>
<td>(ml)</td>
<td>13.2</td>
</tr>
</tbody>
</table>
more severe during tripping operations when mud is left in the hole at higher temperature. The polymers used in the present inhibitive water based drilling fluid system have good thermal stability and do not degrade at elevated temperatures. PAC and CMC have got temperature stability up to 148°C (300 F) and 121°C (250 F), respectively. Graft copolymer has a temperature stability up to 230 each. The thermal stability of the developed drilling fluid system is well checked at 95°C (203 F) in roller oven as per the recommended procedure. The filtration and rheological parameters before and after aging are reported in Table 6. These properties were not affected significantly after aging.

### 3.4.4. Shale recovery rate in the presence of graft copolymer

The troublesome shale formations cause many wellbore instability problems sometimes leading to complete wellbore failure [37]. The effect of graft copolymer on the shale recovery rate is shown in Table 7. The %R value for the inhibitive drilling fluid system developed with graft copolymer is greater than the %R of the drilling fluid developed without graft copolymer. Also, the %R and %R have increased with the increase in the concentration of the graft copolymer thus, indicating the positive effect of the graft copolymer in preventing disintegration or dispersion of shale. The probable mechanism for this effect is the adsorption of the graft copolymer on the surface of the shale and the formation of the film on its surface thus, restricting the entrance of the water molecules into the shale.

### Table 3 Effect of graft copolymer on the flow behavior (n) and consistency index (K) of base fluid system.

<table>
<thead>
<tr>
<th>Graft copolymer (wt/v%)</th>
<th>n</th>
<th>K</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.3</td>
<td>0.541</td>
<td>0.988</td>
</tr>
<tr>
<td>0.4</td>
<td>0.531</td>
<td>1.211</td>
</tr>
<tr>
<td>0.5</td>
<td>0.529</td>
<td>1.515</td>
</tr>
<tr>
<td>0.6</td>
<td>0.521</td>
<td>1.731</td>
</tr>
<tr>
<td>0.7</td>
<td>0.514</td>
<td>1.930</td>
</tr>
<tr>
<td>0.8</td>
<td>0.495</td>
<td>2.526</td>
</tr>
</tbody>
</table>

### Table 4 Effect of carboxymethyl cellulose on the rheological properties and API fluid loss volume of the base fluid system.

<table>
<thead>
<tr>
<th>CMC concentration (wt/v%)</th>
<th>Plastic viscosity (mPa-s)</th>
<th>Apparent viscosity (mPa-s)</th>
<th>Yield point (Pa)</th>
<th>Initial gel strength (Pa)</th>
<th>10 min gel strength (Pa)</th>
<th>API fluid loss (ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.3</td>
<td>11</td>
<td>19</td>
<td>8.0</td>
<td>1.0</td>
<td>1.5</td>
<td>16.0</td>
</tr>
<tr>
<td>0.4</td>
<td>12</td>
<td>20.5</td>
<td>8.5</td>
<td>1.0</td>
<td>2.0</td>
<td>15.5</td>
</tr>
<tr>
<td>0.5</td>
<td>13</td>
<td>22</td>
<td>9.0</td>
<td>1.5</td>
<td>3.0</td>
<td>15.2</td>
</tr>
<tr>
<td>0.6</td>
<td>14</td>
<td>23.5</td>
<td>9.5</td>
<td>2.0</td>
<td>3.5</td>
<td>14.0</td>
</tr>
<tr>
<td>0.7</td>
<td>15.5</td>
<td>25.5</td>
<td>10</td>
<td>2.5</td>
<td>4.0</td>
<td>13.0</td>
</tr>
<tr>
<td>0.8</td>
<td>17</td>
<td>28.5</td>
<td>11.5</td>
<td>3.0</td>
<td>5.0</td>
<td>11.8</td>
</tr>
</tbody>
</table>

### Table 5 Effect of graft copolymer on the rheological and filtration properties of the base fluid system in presence of 3 wt/v% calcium carbonate.

<table>
<thead>
<tr>
<th>Concentration of graft copolymer (wt/v%)</th>
<th>Plastic viscosity (mPa-s)</th>
<th>Apparent viscosity (mPa-s)</th>
<th>Yield point (Pa)</th>
<th>Initial gel strength (Pa)</th>
<th>10 min gel strength (Pa)</th>
<th>API fluid loss (ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.3</td>
<td>12</td>
<td>21.0</td>
<td>9.0</td>
<td>1.0</td>
<td>1.5</td>
<td>5.2</td>
</tr>
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<td>0.4</td>
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<td>39.0</td>
<td>20.0</td>
<td>3.5</td>
<td>5.5</td>
<td>4.0</td>
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</table>

### Table 6 Rheological and filtration properties of the developed drilling fluid formulation (base fluid + 0.8% graft copolymer) after the thermal aging test at 95°C for 16 h.

<table>
<thead>
<tr>
<th>Properties</th>
<th>Units</th>
<th>Before aging</th>
<th>After aging</th>
</tr>
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<tbody>
<tr>
<td>Plastic viscosity</td>
<td>mPa-s</td>
<td>19</td>
<td>20</td>
</tr>
<tr>
<td>Apparent viscosity</td>
<td>mPa-s</td>
<td>39.0</td>
<td>37.0</td>
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<tr>
<td>Yield point</td>
<td>(Pa)</td>
<td>20.0</td>
<td>17.0</td>
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<tr>
<td>Initial gel strength</td>
<td>(Pa)</td>
<td>3.5</td>
<td>3.5</td>
</tr>
<tr>
<td>10 min gel strength</td>
<td>(Pa)</td>
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<td>5.5</td>
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<tr>
<td>API fluid loss</td>
<td>(ml)</td>
<td>6.0</td>
<td>5.9</td>
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</table>

### Table 7 Effect of graft copolymer on the shale recovery performance tests.

<table>
<thead>
<tr>
<th>Base fluid + graft copolymer (wt/v%)</th>
<th>W1 (gm)</th>
<th>W2 (gm)</th>
<th>%R1</th>
<th>%R2</th>
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<td>44.12</td>
<td>89.4</td>
<td>88.24</td>
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</table>
4. Conclusion

The inhibitive water based drilling fluid system developed with synthesized graft copolymer had shown favorable rheological and filtration properties desired for the optimum performance in oil/gas well drilling. Thermogravimetric analysis had revealed that the graft copolymer has better thermal stability than the carboxymethyl cellulose. The rheological properties were increased at higher concentrations of the graft copolymer. The fluid loss control property of the inhibitive water based drilling fluid system was found to be better with the increase in the concentration of graft copolymer. Further, the filtration properties of the developed inhibitive system were controlled by adding calcium carbonate. This may aid in the quick formation of external filter cake while drilling geological formations and it will be easily removed during the exploitation of hydrocarbons from the reservoir. The shale recovery performance tests had revealed that the developed graft copolymer has a high shale encapsulating efficiency. Thus, the drilling fluid system developed with graft copolymer may be suitable for the optimum performance of the drilling fluid while drilling the shale formations.

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References