Dicationic Surfactants as an Additive in Fracturing Fluids to Mitigate Clay Swelling: A Petrophysical and Rock Mechanical Assessment

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ABSTRACT: The interactions of clays with freshwater in unconventional tight sandstones can affect the mechanical properties of the rock. The hydraulic fracturing technique is the most successful technique to produce hydrocarbons from unconventional tight sandstone formations. Knowledge of clay minerals and their chemical interactions with fracturing fluids is extremely vital in the optimal design of fracturing fluids. In this study, quaternary ammonium-based dicationic surfactants are proposed as clay swelling inhibitors in fracturing fluids to reduce the fractured face skin. For this purpose, several core flooding and breakdown pressure experiments were conducted on the Scioto sandstone samples, and the rock mechanical properties of the flooded samples after drying were assessed. Coreflooding experiments proceeded in a way that the samples were flooded with the investigated fluid and then postflooded with deionized water (DW). Rock mechanical parameters, such as compressive strength, tensile strength, and linear elastic properties, were evaluated using unconfined compressive strength test, scratch test, indirect Brazilian disc test, and breakdown pressure test. The performance of novel synthesized surfactants was compared with commercially used clay stabilizing additives such as sodium chloride (NaCl) and potassium chloride (KCl). For comparison, base case experiments were performed with untreated samples and samples treated with DW. Scioto sandstone samples with high illite contents were used in this study. Results showed that the samples treated with conventional electrolyte solutions lost permeability up to 65% when postflooded with DW. In contrast, fracturing fluid containing surfactant solutions retained the original permeability even after being postflooded with DW. Conventional clay stabilizing additives led to the swelling of clays caused by high compression and tensile strength of the rock when tested at dry conditions. Consequently, the rock fractures at a higher breakdown pressure. However, novel dicationic surfactants do not cause any swelling, and therefore, the rock fractures at the original breakdown pressure.

1. INTRODUCTION

Fracturing fluids are commonly prepared in a water-based fluid that requires several chemical additives to achieve several purposes such as corrosion inhibition, proppant-carrying capacity, pumpable rheology, friction reduction, and so on. Improperly designed fracturing fluid when penetrating the geological formation could reduce the permeability because of the swelling of clay around the fractured area, which ultimately affects the rate of oil and gas production. Therefore, it is essential to investigate the swelling inhibition mechanism of fracturing fluids to prevent fracture face skin problems. In the past, many researchers studied the swelling of clays in the sandstone formation. It was reported that sandstone formations swell and weaken upon interactions with water. The cracks in sandstone formation expanded, which often caused severe geological problems. The clay-rich formations lose their strengths upon interaction with water as Erguler and Ulusay reported that mudstone lost more than 40% of its dry strength upon soaking in water.

The presence of clay minerals in rocks can alter the rock’s mechanical properties when interacted with fresh water. Rock mechanical parameters of rocks, such as compressive strength, tensile strength, and linear elastic parameters, are greatly affected. The amount of saturation directly impacts the unconfined compressive strength (UCS) and Young’s modulus. Vasarhelyi has found that the saturated UCS is three-fourth of dry UCS. If the rock is tested at the wet state after

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saturation, then the UCS and Young’s modulus tend to decrease while Poisson’s ratios tend to increase.3

The clay stabilization phenomenon is categorized into two classes: (i) stabilization by physically sealing the micropores and small cracks and (ii) chemical stabilization, in which stabilizer molecules encapsulate the clay surface to prevent the interactions of clay with water. The stabilizers that follow the chemical inhibition are considered superior when compared to those that follow the physical sealing mechanism. In the oil field industry, potassium chloride (KCl) is the most commonly used clay stabilizer to prevent wellbore instability caused by swelling in sandstone upon its hydration.4 However, KCl has many limitations. For instance, it stimulates the dispersion of kaolinite clay and can cause fine migration.5−7 Polymers and their composites as clay stabilizers are not useful at high-temperature conditions encountered in the deep wells.8,9 The amine-based stabilizers have limited applications because of some associated disadvantages including toxicity, pH-dependent inhibition, high dosage, and low-temperature tolerance.10−12 For nanoparticles, their homogenous distribution in fluids and high impact on rheological properties are the primary concern in their application.13,14 Considering the limitations of current solutions, developing a novel clay stabilizer with a strong inhibition capacity is still a research hotspot.

Several studies had been conducted to investigate the potential of surfactants as clay stabilizers.14,15 It was noticed that surfactants showed promising potential to modify the surface properties of the clay. Dicationic surfactants are a class of surfactant that contains more than one head and tail groups connected with a spacer.16−24 Dicationic or gemini surfactants have recently been employed for many applications in the oil and gas industry because of their unique properties such as excellent solubility, higher interface/surface properties, lower critical micelle concentration, and high thermal stability as compared to their monomeric counterparts.25−28 The application of dicationic surfactants is very vast because they contain multiple adsorption positions, unlike conventional surfactants. As a result, dicationic surfactants can enhance their adsorption on clay surfaces because of their higher hydrophobicity nature and cation exchange capacity.29

This study investigates the performance of novel synthesized dicationic surfactants as an additive in fracturing fluids to mitigate clay swelling. The study presented rock mechanical and petrophysical assessment. The performance of the novel synthesized surfactants was also compared with the commercially used clay stabilizing additives, such as KCl and NaCl. The rock mechanical parameters tested were UCS, scratch test, tensile strength, and breakdown pressure test. In this study, the formation mechanical damage caused by fracturing fluid was assessed on oven-dried rock samples. The petrophysical parameters evaluated were permeability and porosity. Permeability was evaluated through coreflooding experiments, and porosity was evaluated using helium gas porosimeter and nuclear magnetic resonance (NMR). Previous studies30−33 were conducted on the rock mechanical assessment of samples in a saturated state. It was found that the compressive strength of the sedimentary rock can be decreased by 20% because of the saturation effect.1,5,34 Thus, to eliminate the effect of water saturation on the rock mechanical properties, all the tests conducted in this study are in a dry state of rock.

2. MATERIAL AND METHODS

2.1. Materials. 2.1.1. Core Sample Preparation. Ten core plugs of 2 in. in length were taken out from a single whole core (20 in.). The core plugs were cleaned, polished, and enfaced before performing subsequent experiments. The samples were later used in coreflooding and rock mechanical tests. The core plugs were vacuum dried at 100 °C for 24 h, followed by saturation with the fracturing fluids’ clay stabilizing additive. The saturation was performed under a pressure of 1000 psi and a temperature of 26 °C for 24 h.

2.1.2. Core Sample Characterization. The bulk density of the cores was 2.651 g/cm³, the mean porosity of the plugs was 15.7%, and the mean absolute permeability was 0.27 mD. The initial liquid permeability was calculated by applying the Klinkenberg effect, which is as follows:

\[ k_l = k_0 \left(1 + \frac{b}{P_m}\right) \]

where \( k_l \) is absolute liquid permeability in mD, \( k_0 \) is the gas permeability in mD, \( P_m \) is the average pressure of the upstream and downstream in the gas permeameter, and \( b \) is the Klinkenberg constant. The mean pore volume (PV) was determined by the saturation weight method. Table 1 presents some properties of the used core plugs.

| Table 1. Petrophysical Properties of the Core Plug Used in This Study |
|---|---|---|---|---|---|
| sample | length (cm) | diameter (cm) | bulk volume (cc) | porosity (%) | absolute liquid permeability (mD) |
| SS-1 | 5.0 | 3.81 | 57.13 | 15.26 | 0.45 |
| SS-2 | 5.0 | 3.81 | 57.13 | 15.18 | 0.39 |
| SS-6 | 5.1 | 3.81 | 58.27 | 14.77 | 0.49 |
| SS-9 | 5.0 | 3.81 | 57.13 | 15.41 | 0.55 |
| SA-2 | 4.8 | 3.81 | 54.85 | 15.82 | 0.31 |
| SA-3 | 4.9 | 3.81 | 55.99 | 15.31 | 0.65 |
| SA-4 | 5.0 | 3.81 | 57.13 | 14.98 | 0.60 |
| SA-5 | 5.0 | 3.81 | 57.13 | 15.45 | 0.59 |
| SA-6 | 5.0 | 3.81 | 57.13 | 15.26 | 0.56 |

2.1.3. Surfactant Preparation. The surfactants investigated in this study were designed for swelling inhibition and wettability alteration. Several screening experiments were conducted before coreflooding was reported in this work. All synthesized surfactants have the same functionalities (head group and tail) except the spacer group. Based on the spacer type, these surfactants were characterized in three different classes. The first category of surfactants contains spacer with varying lengths of the tail. The spacer of the surfactant GS1 consists of four carbon atoms, while the spacer of the surfactant GS2 consists of 12 carbon atoms. The second category of surfactants consists of surfactants having unsaturation in the spacer. The spacer of the surfactant GS1 consists of a single bond, while that of the GS3 and GS4 consists of a double and triple bond, respectively. The third class of surfactants includes those surfactants that contain aromatic groups in the spacer. The spacer of the GS5 surfactant comprises one phenyl ring, while two phenyl rings are present in the GS6 surfactant. A detailed discussion about these surfactants is reported in our previous publications.35
2.1.4. Fracturing Fluid Additive Preparation. All fracturing fluids’ clay swelling additives were prepared using deionized water (DW). Surfactant concentration (0.5 wt %) in DW was implemented in all subsequent experiments. The viscosity of the prepared solution was approximately 1 cP, and the density was 1.0 g/cc.

2.2. Methods. 2.2.1. Unconfined Compressive Strength. The UCS test was performed on a cylindrical sample (length 2” and diameter 2”) using the Matest compression machine. UCS was calculated by eq 2:

$$\sigma_c = \frac{F_c}{A}$$

where $F_c$ is the maximum axial force at failure and $A$ is the cross-sectional area.

2.2.2. Scratch Test. A scratch test determines the indirect compressive strength of the rock sample called scratch strength. Scratch test has several advantages over conventional UCS tests that include but not limited to minimum sample preparation, nondestructive nature, and higher repeatability. The detailed discussion about scratch test is reported in our previous publications. In this study, a scratch test was conducted on different Scioto sandstone samples saturated with different solutions of clay stabilizers. After flooding with surfactants and commercially available clay stabilizers, the samples were dried before performing a scratch test. In scratch tests, a groove on the surface of samples was created using polycrystalline diamond cutter. The values of these parameters are given in Table 2.

Table 2. Parameters Associated with the Scratch Test

| parameters       | values | unit    |
|------------------|--------|---------|
| cutter velocity  | 10     | mm/s    |
| cutter width     | 10     | mm      |
| rake angle       | 15     | degrees |
| depth of cut     | 0.5−0.7 | mm     |

calculating splitting tensile strength based on the Brazilian disc test is given by eq 3:

$$\sigma_t = \frac{2P}{\pi dl}$$

where $P$ is the maximum axial stress acting on the cylinder, $d$ is the diameter of the cylindrical sample, and $l$ is the length of the cylindrical sample.

2.2.3. Breakdown Pressure Measurement. The breakdown pressure of the rock was measured using conventional and synthesized clay stabilizing fracturing fluid additives. The experimental system is comprised of several components. Figure 1 shows the breakdown pressure setup.

The breakdown pressure test was conducted on a core sample having dimensions of 2 in. diameter and 2 in. length. A borehole of 3.5 mm diameter and 19 mm depth was drilled in the middle of the core sample, as shown in Figure 2. A 1/4” stainless steel tubing was inserted in the borehole and cemented in place with HPHT epoxy. After applying the epoxy, the core sample was aged for 24 h to provide enough time for the setting of epoxy. After 24 h of aging, the core samples were loaded in a modified core holder and connected with a breakdown system. The fluid injection rate was 5 cc/min. The clay stabilizing fluid was injected through a drilled hole inside a core sample. The pressure increased gradually, and upon reaching the breakdown pressure, the pressure was suddenly dropped indicating the breakdown pressure of a core sample. All the experiments were carried out without any confining pressure.

2.2.4. Brazilian Disc Test. The length of the core plugs for this test was 0.75 in. The sample was diametrically compressed using Matest compression machine. The formula for calculating splitting tensile strength based on the Brazilian disc test is given by eq 3:

$$\sigma_t = \frac{2P}{\pi dl}$$

where $P$ is the maximum axial stress acting on the cylinder, $d$ is the diameter of the cylindrical sample, and $l$ is the length of the cylindrical sample.

2.2.5. Coreflooding Experimental Setup and Procedure. Figure 3 shows the coreflooding experimental setup used to measure the initial and final permeabilities with different investigated fluids. All coreflooding experiments were conducted at room temperature with a back pressure of 1000 psi and a constant overburden pressure of 1500 psi. The initial permeability was determined with different investigated fluids such as conventional clay stabilizing electrolytes and surfactants. The DW was used to evaluate the final permeability. The permeability was calculated using the Darcy law (eq 4):

$$k_l = \frac{q \mu L}{A \Delta P}$$

where $k_l$ is the liquid permeability (mD), $q$ is the liquid flow rate in cc/min, $L$ is the length (cm), $A$ is the area of the core plug in cm², and $\Delta P$ is the pressure difference between upstream and downstream of the core plug in psi.

2.3. Experimental Plan. Two experimental schemes, namely, the coreflooding scheme and breakdown pressure, were adopted in this study. Under the coreflooding scheme, the core plugs of dimension 1.5 in. diameter and 2 in. length
Figure 3. Process Flow diagram of the Coreflooding Apparatus.

Figure 4. Pressure drop profiles were obtained from the coreflooding experiment using conventional clay stabilizing fracturing fluids and surfactants.
were taken out from the 12 in. whole core. The samples were saturated with the subjected fluid for 24 h. The samples were then used for the coreflooding experiment. After conducting the coreflooding experiments, the flooded samples were oven-dried and then proceeded for the rock mechanical assessment. Similar steps were adopted in the breakdown pressure measurement scheme except for saturation and different dimension.

3. RESULTS AND DISCUSSIONS

3.1. Mineral Analysis. The mineral contents were evaluated in the laboratory by X-ray diffraction technique. The results showed that the mineral content in the sample was 70% quartz, 18% illite, 5% plagioclase, 4% chlorite, 1% kaolinite, and 2% potassium feldspar. Illite was the main clay mineral in the investigated sample. Illite is a 2:1 layer of clay mineral and has a density between 2.6 and 2.9 g/cc.45 Illites are weaker than kaolinite and have a specific surface area of 100 m²/g.43 Specific surface area is responsible for the mineral’s ability to hydrate.44 Illites, when interacting with freshwater, can leach potassium ions and become expandable clay, causing both swelling and fine migration.45

3.2. Coreflooding Experiments. A total of eight coreflooding experiments were conducted: two experiments with conventional clay stabilizing fluids such as sodium chloride (NaCl) and potassium chloride (KCl), and six experiments with different spacer types of novel synthesized dicationic surfactants (GS1–GS6). Formation damage was evaluated by comparing the initial and final permeabilities. These surfactants were selected to investigate the change in permeability when the spacer of the surfactant was changed. Figure 4 shows the pressure drop profile obtained in the coreflooding experiment on Scioto sandstone with conventional clay stabilizing electrolytes and dicationic surfactants. The samples were preflushed with conventional clay stabilizing fluids such as 3 wt % KCl and 3 wt % NaCl solutions and dicationic surfactants. Postflooding was done using DW. The permeability measured during preflushing and postflooding periods of coreflooding experiments is listed in Table 3.

3.3. Nuclear Magnetic Resonance. To verify that the change in porosity and plugging of different pore systems is very minimal with novel synthesized dicationic surfactant, NMR measurements were conducted on one of the samples flooded with surfactant. For this purpose, the sample treated with GS4 was used, a benchtop low-field NMR equipment from Oxford Instrument Core Analyzer Geospec2 was utilized for measuring T2 relaxation time. The low-field NMR spectrometer was operated at 0.05 T (Larmor frequency of 2 MHz). The NMR system is equipped with Green Imaging Technologies system. The NMR experiments were performed at three different intervals during coreflooding experiments, such as after saturation of the sample with GS4, called conditioning; after preflushing with GS4; and after postflooding. Figure 5 shows the incremental and cumulative porosity distribution curves. During conditioning with GS4, the cumulative porosity was found to be 17.2 porosity units (p.u); when preflushed with GS4, the cumulative porosity was found to be 16.8 p.u. After postflushing with GS4, the incremental porosity distribution indicated that the sample had a dual-porosity system such as micropores and macropores. A slight reduction in cumulative porosity was observed after water injection in the postflooding period. On the T2 time scale, the incremental porosity distribution still showed the dual-porosity behavior. Hence, DW did not invade any of the pores’ systems, which proved that no swelling had occurred. Moreover, the decrease in p.u after flooding with DW was negligible.

3.4. Scratch Test Analysis. After the coreflooding experiments, all the samples tested for the scratch test were oven-dried under a temperature of 100 °C for 2 h to eliminate the effect of wetting on rock mechanical properties. After oven drying, the samples were taken out for the scratch test analysis.

Figure 6 shows the average scratch strength of the original rock sample and the samples flooded with commercial clay stabilizing additives such as DW, KCl, and NaCl and novel synthesized surfactants (GS1–GS6). The average scratch strength of the original, untreated rock sample was 39.77 MPa. When the sample was treated with DW, the strength was increased to 43.65 MPa. Muqtadir et al.30 found the change in the strength of the Scioto sandstone sample after conditioning with brine and oil. They carried out the strength test on the wet sample. Because of rock saturation, the strength was decreased. Similarly, Chen et al.31 observed the effect of KCl on the strength of the mudstones; they have found the increase in the concentration of KCl increases the strength of the rock. The average scratch strengths of the KCl and NaCl were found to be 40.98 and 41.65 MPa. The scratch strength for the surfactant treated samples lay in the range of 38.02–40.66 MPa, which was close to the original, untreated rock sample.

| surfactant  | permeability during preflushing (mD) | permeability during postflooding (mD) | percentage reduction in permeability (%) |
|------------|-------------------------------------|--------------------------------------|----------------------------------------|
| surfactant 1 | 0.26                                | 0.25                                 | 3.85                                   |
| surfactant 2 | 0.22                                | 0.21                                 | 4.55                                   |
| surfactant 3 | 0.30                                | 0.27                                 | 10.00                                  |
| surfactant 4 | 0.286                               | 0.285                                | 0.34                                   |
| surfactant 5 | 0.28                                | 0.28                                 | 0.00                                   |
| surfactant 6 | 0.376                               | 0.371                                | 1.33                                   |
| KCl         | 0.19                                | 0.12                                 | 38.00                                  |
| NaCl        | 0.20                                | 0.07                                 | 64.47                                  |
that means the dicationic surfactant does not change the internal structure of the rock.

3.5. Acoustic Wave Velocity Measurement. Acoustic waves such as compressional-wave (P) and shear-wave (S) velocities of all the treated rock samples were measured using scratch test equipment. The values are reported in Figure 7. P- and S-waves are elastic body waves that depend on the density and elastic moduli of the material. P-waves are primary waves that can travel faster than S-waves. For the original untreated rock sample, the value of the P-wave velocity recorded was 3000 m/s. This value was increased to 3356 m/s for a sample that was treated with base case DW. P-waves can travel faster in solids compared to liquids and gases. The increase in P-wave velocity indicates that the empty pore space in the rock sample was occupied by the swelled clay when treated with DW. Similarly, for the same reason, the value of P-wave was increased in the cases of KCl and NaCl. The value of P-wave was not changed significantly and was within the range of 2949—3082 m/s in cases when samples were treated with dicationic surfactants. In all cases, there was no significant change in S-wave velocities. This is because S-wave velocity does not change with the change in travel medium.

Dynamic Young’s modulus (\(E_{\text{dyn}}\)) and dynamic Poisson’s ratio (\(\nu_{\text{dyn}}\)) were calculated from P- and S-wave velocities using eqs 5 and 6. Young’s modulus defines the stiffness of perfectly elastic material. Generally, the value of Poisson’s ratio for any rock remains between 0 and 0.5.

\[
E_{\text{dyn}} = \rho V_S^2 \left( \frac{3V_P^2 - 4V_S^2}{V_P^2 - V_S^2} \right)
\]
where $V_p$ represents the compressional-wave velocity, while $V_s$ represents shear-wave velocities in m/s, and $\rho$ is the bulk density in g/cc. For all Scioto sandstone samples, the value of density was taken to be 2.65 g/cc.

Figure 8 shows the $\nu_{\text{dyn}}$ values calculated using eq 6 for all oven-dried flooded core samples. The $\nu_{\text{dyn}}$ of the untreated original dry rock was found to be 0.204. After flooding with DW and conventional electrolytes such as KCl and NaCl, the $\nu_{\text{dyn}}$ increased to 0.296, 0.279, and 0.291, respectively. The increase in $\nu_{\text{dyn}}$ shows the reduction in rock compressibility. The results are aligned with the findings of Yu et al. The value of $\nu_{\text{dyn}}$ was not changed significantly in cases when samples were treated with dicationic surfactants and lay within the range of 0.1987—0.2498.

Figure 8. Dynamic Poisson’s ratio values of oven-dried Scioto sandstone samples after coreflooding with novel synthesized dicationic surfactants (GS1–GS6) and commercial clay stabilizing electrolytes (NaCl and KCl).

Figure 9 shows the $E_{\text{dyn}}$ calculated on the rock samples after coreflooding experiments. The $E_{\text{dyn}}$ of the untreated, dry rock was found to be 21.3. After flooding with DW and conventional electrolytes such as KCl and NaCl, the $E_{\text{dyn}}$ increased to 22.4, 21.4, and 21.6. The increase in $E_{\text{dyn}}$ in rock shows the increase in rock stiffness. The value of $E_{\text{dyn}}$ was not changed significantly in cases when samples were treated with dicationic surfactants and remain within the range of 20.2—21.2.

In the field when rock is subjected to in situ stresses, then an increase in Poisson’s ratio can increase the breakdown pressure of the rock. Breakdown pressure is the pressure at which rock fails. The breakdown pressure is determined by eq 7.
\[ P_{bu} = 3\sigma_h - \sigma_H + T_o - P_o \]  
where \( P_{bu} \) is the rock breakdown pressure, \( P_o \) is the pore pressure of the rock, \( T_o \) is the tensile strength of the rock, and \( \sigma_H \) and \( \sigma_h \) are the maximum and minimum horizontal stresses acting on the subjected rock, respectively. \( \sigma_H \) and \( \sigma_h \) are directly proportional to Poisson’s ratio and Young's modulus. The equation to find \( \sigma_h \) is given as follows:

\[ \sigma_h = \frac{\nu}{1-\nu} \sigma_v \]  
where \( \sigma_v \) is the vertical stress and \( \nu \) is Poisson’s ratio of the rock.

### 3.6. UCS Measurement
An UCS test was performed on Scioto sandstone samples after flooding with conventional electrolytes such as DW, KCl, and NaCl and dicationic surfactants (GS1–GS6). All the samples tested for UCS were oven-dried under a temperature of 100 °C for 2 h to eliminate the effect of wetting on rock mechanical properties. Figure 10 shows the view of the dried rock samples after conducting the UCS test.

![Figure 10. Scioto sandstone samples after uniaxial compressive strength test.](image)

The UCS value of the original Scioto sandstone sample without any treatment was 34.97 MPa; when the sample was flooded with DW, the strength increased to 44.70 MPa. The increase in strength was attributed to the fact that flooding with DW resulted in the swelling of the rock that ultimately resulted in the rise of the strength. Similarly, commercial electrolytes such as KCl and NaCl were also resulted in higher strength as compared to the original, untreated rock sample. The increase in strength is directly related to the ability of the electrolytes to inhibit the swelling. Three groups of novel synthesized dicationic surfactants were tested. The strengths of the samples flooded with dicationic surfactant with linear spacers (GS1 and GS2) were 38.65 and 40.26. The strengths of the samples flooded with dicationic surfactant with saturated spacers (GS3 and GS4) were 39.44 and 38.26, while the strengths of the samples flooded with dicationic surfactants with aromatic spacers (GS5 and GS6) were 35.86, 35.65, and 34.97. The average strength of the samples flooded with the aromatic spacer group was close to the original dry rock sample because the clay inhibition capacity of the aromatic spacer group was superior to the linear and nonlinear spacer groups, though all dicationic surfactants resulted in the inhibition of the clay. Figure 11 shows the UCS values of the samples after the coreflooding experiment.

As shown in Figure 11 that the value of UCS was almost constant for the cases of novel synthesized dicationic surfactants (GS1–GS6), while in the case of DW, KCl, and NaCl, the value of UCS varied a lot. Therefore, the samples with different surfactants are grouped as a linear spacer, saturated spacer, and aromatic spacer, and their average values were taken in each group. When illite clay swelled, it made new grain to grain connections, which lead to the increased surface area that ultimately became the reason for the high strength.

### 3.7. Indirect Tensile Strength Measurement
Figure 12 shows the bar chart of the indirect tensile strength test values with different clay stabilizing fluids. Like the results obtained in the compressive strength tests, the Brazilian disc test also showed a similar trend and revealed that the DW and 3 wt % KCl solution caused the swelling of the clays in the tested samples. These fluids resulted in higher tensile strength values, while the novel surfactant solution does not cause any noticeable swelling of clays and, therefore, resulted in the same tensile strength as that obtained in the case of the dry rock sample.

### 3.8. Breakdown Pressure Measurement
The fracturing experiments were conducted with conventional clay stabilizing electrolytes and dicationic surfactants. These experiments were conducted to determine the breakdown pressure of the samples. Breakdown pressure is the pressure at which the fracture is generated in the subjected rock sample. The breakdown pressure of the rock depends on many factors, such as fracturing fluid viscosity, injection rate, in situ stresses, rock strength, and density of fracturing fluids.48

Figure 13 shows the fracture created on one of the samples after fracturing with a dicationic surfactant. Figure 14 shows the bar chart of the breakdown pressure values with different clay stabilizing fluids. It can be seen from the chart that the breakdown pressure recorded in the samples fractured with the novel dicationic surfactants was lower than the conventional clay stabilizing electrolytes such as NaCl (breakdown pressure, 5.82 MPa), KCl (breakdown pressure, 5.49 MPa), and DW (breakdown pressure, 6.24). Dicationic surfactant with linear spacer, saturated spacer, and aromatic spacer resulted in breakdown pressures of 5.028, 5.22, and 5.145 MPa, respectively. All three categories of dicationic surfactant result in almost similar breakdown pressure. From NaCl, the breakdown pressure is 16% more than the breakdown pressure with the linear spacer surfactants.

### 3.9. Analysis of Mechanism
The clay itself is a negatively charged mineral, but it contains several interlayers of cations such as sodium ions (Na⁺) or calcium ions (Ca²⁺). The clay swelling happens due to the hydration of cations that may lead to the expansion of clay. The introduction of dicationic surfactants results in the cation exchange phenomenon and surfactant adsorb and intercalate between the layers. These surfactants contain a positively charged ammonium head group that can balance the negatively charged clay minerals. Once the significant molecules of surfactant adsorb, the lipophilic tails inhibit the adsorption of water, which results in the reduction in clay swelling. The reduction in clay swelling retains the original permeability and strength of the sample, whereas in the case of commercial electrolytes due to the clay swelling, the petrophysical and rock mechanical properties are altered.
4. CONCLUSIONS

The inhouse-developed cationic surfactants were treated with Scioto sandstone that has very high illite content. The rock mechanical properties, such as compressive strength, tensile strength, and breakdown pressure, were evaluated. Following are some major conclusions:

1. Commercially used clay stabilizing electrolytes such as KCl and NaCl in a fracturing fluid cannot provide long-term clay stabilization and reduce the permeability up to 65% of the original value.

2. The proposed dicationic surfactants can maintain the permeability of the tight sandstones to their original values.

3. Dicationic gemini surfactants are also capable of maintaining the pores system as confirmed by NMR measurements.

4. Compressive and tensile strengths of the samples treated with commercially used clay stabilizing electrolytes such as KCl and NaCl were found to be higher than the original untreated sample, while the strength measurements of the rock sample flooded with novel synthesized dicationic surfactants were equivalent to the original untreated sample.

5. Linear elastic parameters such as dynamic Poisson’s ratio and dynamic Young’s modulus of the samples flooded with commercially used clay stabilizing electrolytes such as KCl and NaCl were found to be higher than the
original untreated sample, while with novel synthesized dicaticonic surfactants, Poisson’s ratio and Young’s modulus were equivalent to the untreated rock sample. (6) Higher breakdown pressures up to 16% more than the surfactants were recorded with commercially used clay stabilizing electrolytes. (7) The use of novel dicaticonic surfactants is economically feasible because they are used in 0.5 wt %, while commercially used clay stabilizing electrolytes such as KCl and NaCl are used as much as 3 wt %.

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Notes

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